

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JAN. 3 and 10, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Improving the performance of rectifying columns and evaporators. E. MISHKIN (Azerbeid. Neft. Choz., 1929, No. 3, 72—74).—The lower part of the column carries, immersed in the distillate, an open vertical cylinder, at the bottom of which steam is injected. The condensate discharge tube is attached at the upper level of the condensate and has a U-bend to ensure a constant level and prevent the escape of gases and vapours.

CHEMICAL ABSTRACTS.

New methods of gas washing. VI. Viscosity of the absorbent. L. PIATTI (Z. angew. Chem., 1929, 42, 1035—1036; cf. Weissenberger and co-workers, B., 1926, 111).—In continuation of the experiments on the use of tetralin as a "protector" of cresol for preventing the formation of molecular complexes, the viscosity-temperature relationships over the range 50° to —15° have been determined. With tetralin there is a small increase in viscosity as temperature falls, whereas with cresol (mixture of three isomerides) the increase is relatively small from 50° to 15°, but changes rapidly from 15° to 5°, and below 5° the viscosity-temperature curve becomes a straight line with a very steep slope. Experiments with mixtures of cresol and tetralin yielded curves intermediate between those of the pure constituents. By plotting viscosity against composition at the fixed temperatures of —5°, 0°, 20°, and 50°, it is shown that the curves do not follow the law of mixtures. At —5° the curve falls considerably below the line joining the values for the pure components; thus with a 4:1 mixture of cresol and tetralin the measured viscosity is only about one half the calculated value. The calculated and observed values approach more closely at the higher temperatures, and at 50° the two curves are coincident. It appears, therefore, that tetralin is efficacious in inhibiting the association of cresol molecules only at comparatively low temperatures. The washing is more efficient if a low temperature is used and the internal friction of the absorbent reduced by addition of a material such as tetralin, than when the temperature is raised by blowing steam into the wash-tower.

H. J. DOWDEN.

Vacuum measurements in chemical works. I. MÖLLER (Chem. Fabr., 1929, 503—504).—The disadvantages of the usual types of manometer for works' purposes are recounted. An improved form of vacuum gauge is described in which the usual U-shaped tube is replaced by one of nearly circular form, fixed in a frame which turns on a central pivot. On connecting one limb to a vacuum pump the tube turns on its axis, and the degree of vacuum is indicated directly by an attached pointer. A self-recording type of the above apparatus is also described.

H. F. HARWOOD.

See also A., Dec., 1377, **Lyosorption** (OSTWALD and HALLER). 1399, **Particle formation and particle action** (VOLMER). 1409, **Segregation of analysed samples** (SMITH and others). 1415, **Circulation of liquids and vapours** (HUGHESDON and others). 1416, **Device for constant leaching** (SULLIVAN).

PATENTS.

Furnace. W. E. RYNIKER (U.S.P. 1,730,298, 1.10.29. Appl., 5.5.28).—A furnace for air heating is constructed with a complicated series of passages for the products of combustion, which latter do not mix with the heated air.

B. M. VENABLES.

Apparatus for drying materials. CARRIER ENG. Co., LTD., A. FOWLER, and K. J. R. ROBERTSON (B.P. 320,644, 18.6.28).—An oven is heated by a furnace below, but the heat is also transferred to the oven by means of the drying air, which before entering the oven passes through passages adjacent to the flue from the furnace, countercurrent to the gases of combustion. Recirculation of part of the air used for drying is provided for. One end of the oven is preferably permanently closed; the other is open but protected by a hood.

B. M. VENABLES.

Electrical heating and mixing apparatus. DRYSDALE & Co., LTD., and J. YOUNG (B.P. 321,006, 10.9.28).—An annular stator with vertical axis is supplied with polyphase current, thus producing a rotating magnetic field within the stator. The material is contained in a pot within the stator and within the pot is a stirrer of magnetic material which is dragged round by the field. The heating is effected by hysteresis in the walls of the pot, if it is made of magnetic material, and by eddy currents in the pot and/or contents if either are conducting.

B. M. VENABLES.

Preheater. A. R. MCARTHUR, Assr. to AMER. SHEET & TIN PLATE Co. (U.S.P. 1,730,739, 8.10.29. Appl., 14.1.28).—A furnace has an outlet port, which is preferably wide and shallow, that serves for exit of both goods and hot gases. Immediately above the port outside the furnace is a preheater for fluid comprising a number of cross-tubes extending horizontally between inlet and outlet manifolds on either side (when facing the port) through which the hot gases rise to a flue and stack. Since cold air is also inevitably drawn in, a space (nearer the spectator) without tubes is partitioned off inside the preheater casing, which is intended to pass the cold air and a small portion of the hot gases.

B. M. VENABLES.

Heat exchangers. HEENAN & FROUDE, LTD., and G. H. WALKER (B.P. 320,279, 11.12.28).—A heat exchanger of which the heat-transmitting member is formed from deeply-corrugated sheet metal is arranged

* The remainder of this set of Abstracts will appear in next week's issue.

so that the fluids, which are each confined to one side of the metal sheet, pass through the corrugations lengthways in series. B. M. VENABLES.

Heat exchanger. H. BERGQUIST and P. T. KEEBLER, Assrs. to ELLIOTT Co. (U.S.P. 1,726,943, 3.9.29. Appl., 16.2.28).—In a multi-tubular heat exchanger the tube plates are secured to a stiff skeleton, obviating the use of stay bolts. B. M. VENABLES.

Heat-exchange device. A. B. MODINE (U.S.P. 1,726,361, 27.8.29. Appl., 26.3.26).—The apparatus comprises a fan in front of a steam-heated radiator. B. M. VENABLES.

Heat-exchange apparatus. E. N. SIEDER, Assr. to FOSTER WHEELER CORP. (U.S.P. 1,726,995, 3.9.29. Appl., 26.10.28).—In a multi-tubular heat exchanger the smaller tube-plate is divided into two to permit removal of the bundle of tubes and large tube-plate without disturbing the longitudinal baffle attached to the shell. B. M. VENABLES.

Heat-transfer means. F. W. GAY (U.S.P. 1,725,906, 27.8.29. Appl., 5.7.27).—Heat is transferred from a lower conduit carrying hot products of combustion to an adjacent upper conduit carrying air for combustion, by means of a number of vertical, hermetically-sealed tubes containing a volatile liquid. The tubes are inserted about half-way through a plate which forms the horizontal dividing wall of the two gas conduits. B. M. VENABLES.

Heat exchanger. [Jet condenser.] J. P. RATHBUN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,730,242, 1.10.29. Appl., 28.10.27).—For every pair or small group of nozzles of a jet condenser or similar apparatus is provided an auxiliary movable jet supplied with high-pressure fluid adapted to clean the main jets. B. M. VENABLES.

Cooling apparatus. G. E. COX, Assr. to AMER. CYANAMID Co. (U.S.P. 1,733,582, 29.10.29. Appl., 25.4.25).—A joist is arranged with the web horizontal, and a plate is welded to the lower edges of the flanges, forming a closed passage for the cooling agent; a pair of plates form extensions of the upper flanges and constitute an open trough through which the substance to be cooled is conveyed. B. M. VENABLES.

Cooling apparatus for pulverulent or granular material. VICKERS-ARMSTRONG, LTD., and L. D. PARKER (B.P. 321,036, 3.10.28).—A tube mill, particularly for the fine grinding of rapid-hardening cement, is surrounded by a number of water-jacketed tubular bodies (rotating with it) through which the cement is caused to travel either by helical blades or by making the tubes slightly conical. Part of the cooling water may be sprayed on to the shell of the grinding mill. B. M. VENABLES.

Drying of materials. W. J. HARSHAW and C. S. PARKE, Assrs. to HARSHAW CHEM. Co. (U.S.P. 1,729,424, 24.9.29. Appl., 9.2.24).—There are two grinding-drying circuits each comprising an air heater, grinding mill, cyclone separator, and fan in a closed circuit, with a feeder for material leading into the grinding mill and an outlet for ground dry material from the cyclone. The air current is maintained at a pressure sufficient

to remove dried particles from the mill, and the air is used mainly over and over again. The partly-ground dry material from circuit A is the sole feed to circuit B, raw material being fed only to A and finished material withdrawn only from separator B. The air in circuit B is gradually replaced by fresh air, but the air in circuit A is replenished only by air taken from B. B. M. VENABLES.

Cooling and condensing tower. E. BURHORN (U.S.P. 1,732,963, 22.10.29. Appl., 20.6.27).—In the upper part of the tower are means for distributing the water in descending streams through a natural air current; then follow deflectors which guide the water in a few transversely elongated streams upon a corresponding number of rows of superposed condenser pipes from which the water falls to a collecting vessel below. B. M. VENABLES.

Refrigerant. J. G. TANDBERG, Assr. to ELECTROLUX SERVEL (U.S.P. 1,734,278, 5.11.29. Appl., 1.10.25. Swed., 7.7.25).—A solution of a methylamine and a salt, e.g., a chromate, in an alcohol is used. F. G. CLARKE.

Grinding mill. W. A. SEYMOUR, Assr. to HESSE MANUF. Co. (U.S.P. 1,730,300, 1.10.29. Appl., 3.11.27).—A mill of the disintegrator type has a screen (in the lower part) curved to a greater radius than that of the rotor, the lowest point being closest to the rotor. The screen is continued upwards each way by grinding plates, one being tangential to, the other concentric with, the rotor. The inlet for material is above the grinding plates. B. M. VENABLES.

Pulveriser. G. E. BEAR (U.S.P. 1,729,471, 24.9.29. Appl., 10.5.26).—A grinding cone with helicoidal screw on the small end rotates inside a conical grinding die; a fan chamber is situated at the base of the cones, the feed for material being at the apex. Means for adjustment are described. B. M. VENABLES.

Dual-feed pulverising apparatus. F. H. DANIELS, Assr. to RILEY STOKER CORP. (U.S.P. 1,731,649, 15.10.29. Appl., 6.11.26).—Air is caused to flow through a pulveriser from an air inlet at one side to an outlet at the other side. The machine is provided with two independently regulable feeding devices for different materials which are mixed while being ground. B. M. VENABLES.

Separation of intermixed divided materials. R. PEALE, W. S. DAVIES, and W. S. WALLACE (B.P. 290,276 and 292,495, 12.4.28. U.S., [A] 12.5.27, [B] 20.6.27).—Forms of pneumatic shaking tables for the separation of materials varying greatly in size but not much in sp. gr. are described. B. M. VENABLES.

Mixer. O. O. HOWARD (U.S.P. 1,728,411, 17.9.29. Appl., 6.9.27).—A tank is formed with a cylindrical upper and long conical lower part, and a screw conveyor extends from the bottom (apex) of the tank to a point above the base of the cone; the material lifted by the screw is spread by a deflector plate extending downwards and outwards to the level of the base (top) of the cone. B. M. VENABLES.

Mixer. G. S. P. DE BÉTHUNE (U.S.P. 1,727,753, 10.9.29. Appl., 24.6.27).—The material passes through a number of chambers in succession; the chambers are

symmetrical about the axis of the stirrer shaft, and each chamber is provided with radial stirrers.

B. M. VENABLES.

Mixing of powders with liquids in flow. W. EVANS (B.P. 320,710, 10.8.28).—The liquid is admitted to the interior of a bowl, over which it spreads in a thin sheet. The solid matter is fed (*e.g.*, by a table feeder) into the whirling sheet of liquid, and the mixture leaves the bowl by a bottom outlet.

B. M. VENABLES.

Apparatus for emulsifying, homogenising, and mixing liquids with fats, soluble and insoluble powders, etc. R. J. ELLINGHOUSE (B.P. 321,130, 20.2.29).—In an apparatus comprising a propeller rotating inside a perforated screen, the screen is made dome-shaped or ribbed-conical, and more than one may be provided with a common axis.

B. M. VENABLES.

Defecation of liquid. A. U. WETHERBEE, Assr. to GILCHRIST & Co. (U.S.P. 1,733,324, 29.10.29. Appl., 16.2.25).—The liquid is supplied through an upper horn-shaped portion to a cylindrical portion which is continued downwards as a conical portion with bottom outlet for the heavier constituent. The cylindrical portion is provided with annular baffles, and from each annulus is an outlet for clear liquid.

B. M. VENABLES.

Rotary filter. S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,730,159, 1.10.29. Appl., 25.2.24).—A rotary filter has the internal filtering surface built up of porous blocks which are provided with ribs projecting from three edges of the blocks. The ribs serve the double purpose of forming passages for filtrate and of acting as keys so that the blocks hold themselves in position.

B. M. VENABLES.

Centrifuges for separation of solid matter from liquids. BERGEDORFER EISENWERK A.-G. (B.P. 307,065, 20.2.29. Ger., 2.3.28).—The solid matter is deposited on the wall (preferably coned or vaulted) of the bowl and is moved by helical scrapers to an enlarged zone which is also provided with scrapers and with outlets which expand outwardly. The viscosity of the solid matter being discharged may be reduced by the supply of an additional liquid in close proximity to the outlets.

B. M. VENABLES.

Press for the extraction of liquids. E. T. MEAKIN (U.S.P. 1,733,381, 29.10.29. Appl., 12.7.21).—An Archimedean conveyer press has the casing constructed of longitudinal grooved bars held by clamping rings. The annular outlet (for solids) is obstructed by a spring-controlled plug ring.

B. M. VENABLES.

Apparatus for separating liquids of different densities. F. PINK (B.P. 320,986, 17.8.28).—In an apparatus such as a gravity oil-water separator, the separating vessel has a top which is closed except for a series of outlets for lighter liquid, but the normal liquid level is well below the top. A float is provided which will float in the heavier but sink in the lighter liquid, so that when lighter liquid accumulates the float falls and eventually closes the normal outlet, allowing pressure to generate within the vessel, with the result that lighter liquid will be expelled through the top outlets, which are individually guarded by valves which will float (and close the orifices) when the heavier liquid arrives.

B. M. VENABLES.

Apparatus for vaporising liquids by means of accumulated heat. A. BREISIG (U.S.P. 1,733,292, 29.10.29. Appl., 21.4.24. Ger., 22.5.23).—A number of A-shaped metal bars are arranged horizontally in staggered rows within a heatproof chamber which is subjected alternately to hot gases (upwards) and liquid to be vaporised (downwards).

B. M. VENABLES.

Evaporation, distillation, or the like apparatus. W. VOGELBUSCH (U.S.P. 1,733,476, 29.10.29. Appl., 17.2.27. Holl., 14.4.26).—The vertical part of the apparatus comprises only a vapour space above and liquor space below. From the latter one or more tubes of large diameter extend sideways to the bottom of an inclined bundle of smaller tubes, the upper ends of which discharge into the vapour space. Both sets of tubes are jacketed for the heating medium, which enters at the top of the bundle and flows counter-current to the circulating liquor and vapour.

B. M. VENABLES.

Film evaporators. K. W. PFEFFERKORN (B.P. 300,655, 16.11.28. Switz., 17.11.27).—The liquid is supplied in a thin film to the outer surface of the steeply inclined upward run of a belt conveyer travelling in a triangular course. Heat is applied to the interior of the belt and air is drawn over the outer surface. The dried material is scraped off at the bottom of the downward vertical run of the conveyer. The warm, moist air leaves through a pipe concentric with one in which fresh air arrives, thus preheating the latter.

B. M. VENABLES.

Treatment of gases with liquids. CARRIER ENG. Co., LTD. (B.P. 309,403, 11.9.28. U.S., 9.4.28).—Liquid is ejected rearwardly from the ends of the arms of a rotating sprinkler, the velocity of issue being about equal to the rotative velocity of the arms, so that the issuing liquid has no substantial momentum. The jets are so shaped as to cause the spray to whirl about axes tangential to the circle described by the sprayer. The gas current is drawn through the mist produced.

B. M. VENABLES.

Separation of gas mixtures. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 294,611, 26.7.28. Ger., 27.7.27).—An impure gaseous remainder of low b.p. is scrubbed at a low temperature with liquid obtained earlier in the series of condensations (which are effected in heat interchangers) with the object of removing impurities from the residual gas by dissolution, rather than by difference of b.p. In the preparation of, *e.g.*, hydrogen from coke-oven gas, the hydrogen containing carbon monoxide and nitrogen is scrubbed with liquid rich in methane while being cooled by nitrogen boiling in a pipe coil: this cooling, besides reducing the impurities in the hydrogen to a low figure, is utilised to make up the cold losses of the system.

B. M. VENABLES.

Fume-treating device. W. ZITELLA (U.S.P. 1,733,710, 29.10.29. Appl., 30.12.25).—Air is blown from a fan through one horizontal pipe and is distributed at right angles (by means of baffles), through a series of hoods over the furnace from which fume is to be collected, to another horizontal pipe provided with water sprays. The hoods are elongated and their undersides

are open but provided with inclined baffles so that the currents of air suck in the fumes. B. M. VENABLES.

Means for separating solid particles from combustion gases. A. R. V. KEARSLEY (B.P. 321,050, 22.10.28).—The delivery part of the casing of a centrifugal induced-draught fan is shaped so as to collect the solid particles which are flung out of the gases by centrifugal force. B. M. VENABLES.

Air cleaner. A. C. BENNETT (U.S.P. 1,734,030, 5.11.29. Appl., 6.11.26).—Air enters one end of a tubular casing and assumes a whirling motion due to the action of deflecting blades arranged round an inner axial tube, which forms an outlet, the other end of the casing being closed and provided with a dust chamber. A second set of deflecting blades are arranged round the inner end of the outlet tube, which is perforated between the two sets of blades to enable some of the air to enter the outlet directly after passing the first set.

F. G. CLARKE.

Apparatus for determining the sp. gr. of solid substances or bodies. W. & T. AVERY, LTD., and W. A. BENTON (B.P. 320,416, 14.7.28).—The apparatus comprises a stand supporting a spring balance, or other device, of which the deflection is proportional to the load. Three vertical, adjustable sliders which can be set by hand to the positions of zero, weight in air, and weight in water, carry knife-edges or pivots that determine the position of a parallel-rule linkwork and of an inclined computing bar, upon the last of which the sp. gr. is read off without any calculation, for any weight of substance within the range of the spring balance. B. M. VENABLES.

[Recuperators for] heating furnaces. W. H. FITCH (U.S.P. 1,735,605—8, 12.11.29. Appl., 18.12.26).—See B.P. 282,443; B., 1929, 495.

Production of flame or smoke for signalling. C. W. BONNIKEN and S. BARRATT (U.S.P. 1,735,373, 12.11.29. Appl., 22.11.26. U.K., 30.11.25).—See B.P. 268,004; B., 1927, 719.

Crushing and grinding machine. S. BRAMLEY-MOORE (B.P. 309,159, 11.10.28. U.S., 6.4.28).—See U.S.P. 1,692,884; B., 1929, 154.

[Supplying air to marine-type boiler] furnaces. J. REID (B.P. 321,207, 1.8.28).

Refrigerating apparatus. M. H. ACKERMANN and L. C. SICKEL (B.P. 321,590, 21.12.28).

[Absorption] refrigerating apparatus. H. D. FITZPATRICK (B.P. 321,488, 31.8.28).

II.—FUEL; GAS; TAR; MINERAL OILS.

Combustible matter in brown coal. W. EISENSCHMIDT and H. KOOP (Chem.-Ztg., 1929, 53, 858—859).—A simple analysis of a coal is no absolute indication as to its calorific value, since the character of the carbon compounds is an important factor. Hence two coals of the same elementary analysis may have different calorific values. G. E. WENTWORTH.

Colloidal and chemical properties of peat. I. I. SHUKOV, E. A. KREMLEVA, and A. V. TIKHOMIROV (J. Appl. Chem., Russia, 1929, 2, 257—266).—When

examined by Ostwald's method of filtration analysis, peat of a high degree of decomposition shows rapid filtration. The rate increases with rise of temperature and with increase of valency of the coagulating agent; it also increases, and then falls, with increase in concentration of the agent. CHEMICAL ABSTRACTS.

Fuels yielding power gas. P. LEBEAU (Ann. Off. Nat. Combust. liq., 1929, 4, 19—64; Chem. Zentr., 1929, ii, 114).—The thermal fractionation of pyroligneous gases and its relation to the degree of carbonisation of the product are shown graphically. A. A. ELDRIDGE.

Low-temperature carbonisation of fuel, and its combination with the production of electricity. E. H. SMYTHE and E. G. WEEKS (Inst. Elect. Eng., 1929. Advance copy. 10 pp.).—The Babcock coal distillation plant, as operated at the Dunstan power station of the Newcastle-upon-Tyne Electric Supply Co., consists of two retorts, working in conjunction with a Babcock boiler of 30,000 lb. of steam per hour evaporative capacity. The coal used is screened, the fines being utilised in a pulverised fuel boiler, whilst the larger coal is dried and preheated (to 120°) by passing through a chamber in contact with waste flue gases. The coal is then fed into the retort, in which the fuel bed is 3 ft. deep, and is carried on six slowly-rotating rollers which act as a supporting grate and coke extractor; the lower portion of the retort forms a chamber for the reception of the heating gases and serves as a coke hopper. The distillation of the coal is effected by passing a mixture of superheated steam and combustion products at 600—700° through the fuel bed. The distilling medium is obtained by burning suitable fuel such as producer or coke-oven gas in a small combustion chamber located below the retort, and when combustion is complete sufficient steam is added to produce distilling gases of the required composition and temperature; part of the steam required is obtained from the quenching of the coke, and the additional amount necessary is supplied by bleeding the turbine casings in the main power station. The coke produced is crushed and used under an adjacent boiler equipped with a chain-grate stoker. The rich retort gas and oil vapours from the coal, mixed with the distilling medium, are drawn off from the retort and passed through dust extractors, condensers, tar extractors, and a rotary gas washer; the remaining gas is burned in the combustion chambers of a boiler. The by-products are worked up by usual distillation and purification methods. By this process the following yields per ton of coal are obtained: coke 15 cwt., motor spirit 1 gal., steam-distilled creosote 6 gals., heavy creosote 2.4 gals., pitch 5 gals., and air-blown low-boiling tar acids 0.4 gal. C. B. MARSON.

Economics of coke-oven gas utilisation in industry. E. C. EVANS (J. Inst. Fuel, 1929, 3, 90—99).—Attention is drawn to the necessity for a re-organisation of the coking industry, which will be rapidly accelerated if a reasonable price could be obtained for the coke-oven gas that would be rendered available if existing coking plants were replaced by modern ovens. The chief uses of coke-oven gas are for steam raising, power production in gas engines, and for heating metallurgical furnaces in the iron and steel industry, of which the last-

named is the largest potential consumer. Examination of economic conditions shows that the price which the iron and steel industry can afford to pay for coke-oven gas, purified, delivered, and purchased in large quantities, is approx. 1.2—2d. per therm, based on existing costs for equivalent service. C. B. MARSON.

Determination of carbon monoxide [in gases]. P. SCHLÄPFER and E. HOFMANN (Monats. Bull. Schweiz. Ver. Gas-Wasserfachmännern, 1927, 7, 293—303, 349—372; Chem. Zentr., 1929, i, 3013—3014).—The iodine pentoxide, palladous chloride, silver nitrate, and the Damiens methods have been critically examined. (1) The iodine pentoxide must be dehydrated at 190—200°, and the reaction temperature should be 130—135°. The gas velocity, up to 0.5% CO, may be 50 c.c. per min. for 50 g. of reactive material, 200 c.c. of gas being used. The iodine is removed in 6—8 min. from the tube with 600—800 c.c. of air or nitrogen free from carbon monoxide. The gas under test must be dry and free from unsaturated hydrocarbons, aldehydes, hydrogen sulphide, etc.; pure hydrogen attacks iodine pentoxide only above 150°. Two forms of apparatus are described; the iodine may be determined colorimetrically. (2) The palladous chloride method is approximate only. (3) The silver nitrate method is approximate, but rapidly indicates the presence of small quantities of carbon monoxide. Quantities less than 0.05% are difficult to detect. For the determination of quantities greater than 0.2% in the usual gas-analysis apparatus, iodine pentoxide suspended in 10% oleum at the 'ordinary' temperature is preferable to Damiens' reagent; it is unattacked by hydrogen, oxygen, or methane, but heavy hydrocarbons must be removed. A. A. ELDRIDGE.

Installation and operation of gas producers for metallurgical furnaces. J. S. ATKINSON (J. Inst. Fuel, 1929, 3, 71—89).—The preparation and use of fuels such as bituminous coal, lignite, coke, anthracite, etc., and the factors governing choice of producers are discussed. Various types of producers, methods for their efficient operation, and the use of mechanical equipment are described. C. B. MARSON.

Anthracene from oil tar. I. BESPOLOV (Azerbeid. Neft. Choz., 1929, No. 5, 72—78).—The pitch (b.p. above 280°) was distilled and the crystalline distillate repeatedly refractionated; 0.6% of anthracene was obtained from a tar (*d* 1.062) of which 30.8% boils below 300° and 40.2% above 300°. The purity of the anthracene, recrystallised from the xylene fraction of a light oil, was 80%. CHEMICAL ABSTRACTS.

Hydrolysis of naphthenic acids at elevated temperatures and cracking under pressure of hydrogen. V. N. IPATIEV and A. D. PETROV (J. Appl. Chem., Russia, 1929, 2, 327—335).—The acids (100 g., b.p. 260—360°, acid value 233, average mol. wt. 240) were heated with water (50 c.c.) and alumina (10 g.) for 5 hrs. at 440—460° and 210 atm., affording an oil (80 c.c.) having acid value 131.4, the fractional composition of which is indicated. Tetralin, similarly treated, afforded an oil containing only a little benzene, but large amounts of aromatic hydrocarbons of high b.p. Experiments with hexahydrotholuene and paraffins are de-

scribed. The fatty acids of the paraffin series are stable. When cracked in presence of water (alumina, and hydrogen) naphthenic acids afford more oxygen compounds than in absence of water, and the amount of compounds of low b.p. is lower. CHEMICAL ABSTRACTS.

Kuban crude oils. M. A. BESTUZHEV (Neft. Choz., 1928, 15, 339—342).—The characteristics of light and heavy Iliski crude oils and their fractions are described. CHEMICAL ABSTRACTS.

Refractometric analysis of paraffin products. W. J. PIOTROWSKI and J. WINKLER (Przemysl Chem., 1929, 13, 381—387).—Diggs and Buchler's refractometric method for the analysis of paraffin wax (B., 1927, 625) is modified for application to Galician conditions. Blue oil, obtained by cooling the oil pressed out of paraffin wax to —6°, is mixed with infusorial earth, cooled to —21°, and filtered under reduced pressure. The mean refractive index (n_D) of a number of filtrates of Boryslaw oil is 1.4887, and this value is taken as being the refractive index of 100% oil present as impurity in the wax. Pure paraffin wax is obtained by filtering 10 g. of wax at 80° through 5 g. of Terran, an adsorptive powder which removes only the oily constituents, and the refractive index, n_D , of the first drop of filtrate is determined (1.4370). These indices, which are determined at 60°, are taken as being constant for paraffin wax derived from a given region, and the paraffin content p of a given specimen, the refractive index of which is n_x , is given by the formula $p = 100 \times (n_0 - n_x) / (n_0 - n_p)$. The values given by this method are 1.1—2.6% lower than those obtained by Holde's method, and are in very close agreement with the actual values in artificially prepared mixtures of pure paraffin and oil. The same method is applicable to the determination of the paraffin content of blue oil. R. TRUSZKOWSKI.

Determination of the ageing tendency of insulation and turbine oils. A. BAADER (Petroleum, 1929, 25, 235—243, 279—282).—Existing tests are vitiated because they are carried out under one set of conditions only, which may not correspond with those of actual use, and also the refining process that has been employed is not considered. The apparatus now used consists of an electrically heated thermostat in which the outer jacket contains water that is boiled and refluxed by condensers on either side; the inner vessel is filled with oil and in it are immersed the test vessels. These are four in number, have Liebig condensers, and contain stirrers which are constructed of different metals, glass, bakelite, etc., so that an oil can be treated simultaneously under four sets of conditions at the same temperature. After heating for a suitable time the saponification value of the sample is determined and gives a direct measure of the sensitiveness of the oil to any particular treatment. The useful limits which may be reached for this factor are defined for different types of oil, and results are given for a wide range of samples; these are compared with the figures provided by older methods of testing. It is found that temperature affects some oils much more than others; steel is usually inert, but iron often promotes change, whilst copper and lead are particularly active. The behaviour of individual oils is discussed in detail, and the general influence of temperature on their changes is described. R. H. GRIFFITH.

[Use of] sodium peroxide in ascertaining the condition of used insulation oils. G. SCHWITTAY (Petroleum, 1929, 25; Motorenbetrieb, 2, No. 10, 6—8).—Samples of oil after use in transformers etc. have been treated with aqueous sodium peroxide; two solutions were found to be equally useful: (i) a mixture of equal parts of 0.1*N*-sodium carbonate and 0.1*N*-sodium hydroxide treated with 2 g. of sodium peroxide for each 100 c.c., and (ii) a solution of 4 g. of peroxide in 100 c.c. of water. The oil (5 c.c.) is mixed with an equal volume of one of these solutions and of benzine, warmed to about 90°, and allowed to separate. A layer of a dark tarry substance generally settles out, the amount of which is a measure of the ageing of the oil. If more than 2% is thus changed, the sample is considered unfit for further use. The results given by this method are compared with those obtained by other tests.

R. H. GRIFFITH.

Making "viscosines" from lubricating oil bottoms. P. MONTAG (Azerbeid. Neft. Choz., 1929, No. 3, 52—55).—A mixture of Balakhany lubricating oil bottoms with a black gas oil was agitated by air with sulphuric acid. After settling, the oil was blown with air and steam at 300°. The properties of various fractions are recorded.

CHEMICAL ABSTRACTS.

Physical basis of lubrication, especially in the explosion motor. S. KYROPOULOS (Z. tech. Phys., 1929, 10, 2—18; Chem. Zentr., 1929, ii, 115—116).—True lubrication and lubrication under the working conditions of the explosion motor are differentiated. Various physical properties of oil were determined and discussed.

A. A. ELDRIDGE.

Recovery of paraffin wax absorbed by fuller's earth. L. SELSKI (Azerbeid. Neft. Choz., 1929, No. 3, 84—85).—The wax is extracted (thrice) with a naphtha, b.p. 90—130°, and the earth is heated to redness to regenerate its absorptive power. CHEMICAL ABSTRACTS.

Prevention of explosion danger in an oil tanker. Physical analysis of a very weak mixture of petroleum vapour. Z. TUZI and O. KADITA (Bull. Inst. Phys. Chem. Res., Tokyo, 1929, 8, 851—882).—A petroleum content of 10⁻⁴ pt. by vol. in air can be detected refractometrically.

C. W. GIBBY.

Apparatus for determining the degree of refining of petrol. M. R. CORELLI (Annali Chim. Appl., 1929, 19, 453—458).—A special burette is described which gives far more accurate results than the usual Kraemer and Böttcher graduated receiver in the determination of the degree of refining of various petrols by measuring the diminution in volume on shaking with concentrated sulphuric acid. The burette serves well also for determining the reciprocal solubility of two liquids, the extraction of one or more components from a liquid mixture, emulsification tests, etc.

T. H. POPE.

Benzinometer. W. VAUBEL (Chem.-Ztg., 1929, 53, 859—860).—An apparatus is described which permits comparative measurement of the vapour pressure of motor fuels and the effect thereon of anti-knocks and other adulterants. The apparatus consists simply of a thermostated flask fitted with a bung through which

passes an inverted burette tube. The method is to introduce 100 c.c. of the spirit into the flask, and place the bung in position so that the mouth of the burette is close to the bottom of the flask; by this means some of the liquid will be forced up into the burette, due to exertion of its partial pressure in the air space of the flask. Two readings are taken: (a) 1 hr., (b) 2 hrs. after closure, from which, using fuels of different composition, a comparison of the vapour pressures is made. Results are given for mixtures of, e.g., pure benzene and benzine, commercial benzene and benzine, benzine and water, benzine and carbon tetrachloride.

G. E. WENTWORTH.

Lubricant friction and flow orientation. S. KYROPOULOS (Z. tech. Phys., 1929, 10, 46—52; Chem. Zentr., 1929, ii, 116—117).

Mathematical determination of the calorific value of gaseous saturated hydrocarbons and their mixtures. J. HOŠEK (Chem. Obzor, 1929, 4, 105—107; Chem. Zentr., 1929, ii, 196).

Gas washing. PIATTI.—See I. Coke-oven refractories. RICHARDS.—See VIII.

See also A., Dec., 1376, Mixed absorbents (SCHILOV and others). 1397, Chemical equilibrium in autoxidation (GILLET and GUIRCHFELD). 1401, Gaseous combustion in electric discharges (FINCH and HODGE). 1423, New petroleum by-product (BALDESCHWIELDER and CASSAR). 1472, Lead diaryldialkyl compounds (GILMAN and BALASSA).

PATENTS.

Distillation of coal in vertical retorts. H. SHEW-RING (B.P. 320,788, 3.11.28).—In order to lessen the production of dust during the discharge of coke from vertical retorts, the coke chambers are flooded with water and drained again prior to discharging the coke. For this purpose the chambers are connected to a water tank which can be alternately raised and lowered; or two separate tanks may be provided, the upper one for flooding the chambers and the lower one for draining them.

A. B. MANNING.

Apparatus to control the behaviour of coal or other material while being carbonised in a retort, in order to facilitate the discharging of it. W. FRASER and W. DAVIDSON (B.P. 320,780, 31.10.28).—A tapered vertical retort is provided with a movable bottom, operated by hydraulic cylinders or similar device and capable of being lowered during the period of carbonisation so that additional space is provided to accommodate any swelling that may occur. A "dip ring" attached to the retort fits into a water compartment in the movable bottom, forming a seal which prevents air from entering the retort during the process.

A. B. MANNING.

Distillation of coal, lignite, etc. having high contents of volatiles. SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 294,568, 18.6.28. Ger., 26.7.27).—Finely-divided coal, lignite, etc. (volatile matter 26—38%) is agglomerated with a hydrocarbon binding medium, e.g., anthracene oil, and distilled rapidly in a current of superheated steam at 500°. The binding medium must be of such a character that it

forms no pitch at any stage of the process, but decomposes with the production of a substantial quantity of coke. The compact product obtained may, if desired, be converted into a metallurgical coke by further distillation at 700—1000°.

A. B. MANNING.

Treatment of carbonaceous materials to obtain distillation products such as hydrocarbons, metalised coke, etc. FABR. NAT. DE PROD. CHIM. ET D'EXPLOSIFS SOC. ANON. (B.P. 296,443, 29.8.28. Belg., 2.9.27).—Carbonaceous materials are mixed with about 15% of an inorganic oxidising agent, *e.g.*, iron or manganese oxide, alkali or alkaline-earth nitrates, and are then carbonised. A high yield of tar having qualities similar to those of crude petroleum, and a coherent coke suitable for metallurgical purposes, are produced.

A. B. MANNING.

Forming smokeless fuel briquettes. W. E. TRENT (B.P. 320,814, 22.12.28).—The process is carried out in two stages; in the first the agglomerated masses of finely-divided coal and oil (cf. B.P. 228,862; B., 1926, 263) are heated at 260—315° in an atmosphere almost free from oxygen until the water and lighter constituents of the oil are removed; and in the second the heating is continued in an oxidising atmosphere in order to harden and coke the oil residuum. The lighter oil constituents evolved in the first stage are condensed and recovered.

A. B. MANNING.

Manufacture of vegetable char. C. E. COATES, Assr. to O. L. BARNEBY (U.S.P. 1,729,162, 24.9.29. Appl., 22.4.21).—Vegetable matter is destructively distilled, the resultant char is heated above 550° in air, the amount of which is regulated to burn out most of the hydrogen and a limited amount of carbon, and the char is then cooled in the absence of air, treated with mineral acids, *e.g.*, hydrofluoric acid, to remove silica, and dried.

W. G. CAREY.

Distillation of tar and recovery of products therefrom. BARRETT CO., Assees. of S. P. MILLER (B.P. 289,023, 10.4.28. U.S., 20.4.27).—Tar or light pitch is distilled by bringing it into intimate contact with hot coal-distillation gases (cf. B.P. 288,270; B., 1929, 1006), and the enriched gases are scrubbed while still at a high temperature with tar or light pitch in order to give relatively clean oils on condensation, and to produce a heavier pitch residue in the scrubber.

A. B. MANNING.

Manufacture of pitch composition. BARRETT CO., Assees. of S. P. MILLER (B.P. 295,687, 10.8.28. U.S., 17.8.27).—Tar is distilled by being brought into intimate contact with hot coal-distillation gases in order to produce a pitch residue of high m.p. (150—200°) and low free carbon content (cf. B., 1929, 633, 803). This is blended while hot with tar to form a homogeneous mixture which can be used as a fuel, for road treating, etc. The oils condensed from the enriched gases are blended with other tarry oils to form a composition suitable for creosoting purposes.

A. B. MANNING.

Removing oil from mineral oil sulphonc bodies. C. FISCHER, JUN., and W. T. REDDISH, Assr. to TWITCHELL PROCESS CO. (U.S.P. 1,731,716, 15.10.29. Appl., 29.6.26).—By treating crude lubricating oil from petroleum refining with fuming sulphuric acid, separating the acid

layer, and treating it with sodium hydroxide and dilute alcohol, an aqueous solution of the sodium salts of various sulphonc acids is obtained. This is freed from oil by evaporation to dryness, dehydration at 110—120°, and treatment of the dry residue with superheated steam.

A. R. POWELL.

Sulphonc body [from mineral oil sludge]. C. FISCHER, JUN., and W. T. REDDISH, Assrs. to TWITCHELL PROCESS CO. (U.S.P. 1,734,369, 5.11.29. Appl., 2.9.26).—The sludge, comprising aluminium sulphonates, is neutralised with caustic soda, using phenolphthalein as an indicator.

F. G. CLARKE.

Manufacture of active carbons. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,735,096, 12.11.29. Appl., 24.4.25. Fr., 24.12.24).—See F.P. 603,806; B., 1926, 732.

Retort [for distillation of oil shale]. I. B. NEWBERRY, Assr. to LIGNITE PRODUCTS CORP. OF AMERICA (U.S.P. 1,734,774, 5.11.29. Appl., 12.6.22. Renewed 22.10.25).—See B.P. 261,575; B., 1927, 63.

Powdered-fuel burners. H. NIELSEN and B. LAING (B.P. 321,172, 1.5.28).

Plant for generating, storing, and supplying acetylene gas. O. SIMONIS, and LIQUID AIR, LTD. (B.P. 321,234, 7.8.28).

Separation of gas mixtures (B.P. 294,611).—See I. **Heating bituminous materials** (B.P. 319,298 and 319,391).—See XI.

III.—ORGANIC INTERMEDIATES.

Catalytic oxidation of alcohol. L. Y. KARPOV (J. Appl. Chem., Russia, 1928, 1, 121—126).—Ferric oxide, trimanganese tetroxide, cupric oxide, and chromic oxide are preferred as catalysts for the oxidation of ethyl alcohol to acetaldehyde; with nickel or cobalt monoxide carbon is formed. The use of twice the calculated quantity of air is recommended. Optimal temperatures are: nickel sesquioxide 170—190°, chromium dioxide 170—220°; further oxidation to acetic acid takes place.

CHEMICAL ABSTRACTS.

Continuously-operating benzene-washing plant for recovery of aniline from aqueous salt solutions. O. KREBS (Chem. Fabr., 1929, 407—408, 417—419).—In certain dye works large quantities of an alkaline solution of glycine containing about 30% of aniline are continuously produced. After cooling and settling, an aqueous layer containing 12% of aniline is obtained. Recovery of this aniline by steam-distillation is impractical owing to the large amount of steam required and the necessity of reconcentrating the glycine solution. A continuous extraction process by which the aniline liquor is passed downwards in thin streams through a rising column of benzene affords a means whereby the glycine solution may be freed almost completely from aniline at a small cost. The aqueous liquor (*d* 1.175) is allowed to flow downwards through a column divided by perforated plates into a number of superimposed shallow chambers up which the benzene is caused to flow by hydrostatic pressure from a container situated well above the column. The perforated plates ensure thorough mixing of the liquids

and prevent the almost saturated aniline-benzene solution in the upper part of the column from sinking through the less saturated benzene in the lower parts. The purified glycine solution passes to a heat interchanger, where it is warmed to 80° by hot glycine solution which has been steamed to remove benzene, and then passes into the steam-distillation vessel where the dissolved benzene is recovered. A similar procedure is used to separate the aniline and benzene, the former being finally purified by distillation under reduced pressure. A sketch of the necessary apparatus is included and the economics of the process are discussed.

A. R. POWELL.

Wolfenstein and Boeters' process for the preparation of picric acid from benzene. L. DESVERGNES (Chim. et Ind., 1929, 22, 451—461; cf. Wolfenstein and Boeters, B.P. 17,521 of 1907 and G.P. 214,045; B., 1907, 1194, and 1909, 1167. Also cf. A., 1913, 279).—The process consists in heating a mixture of benzene, mercuric nitrate, and nitric acid at 50—60° (cf. Vignon, B., 1920, 567 A; Brewster, U.S.P. 1,380,185; B., 1921, 538 A). Excess benzene is distilled off in steam, and the washings of the filtered product are added to subsequent batches. The acid filtrate may be brought up to strength with fresh acid and used again indefinitely, no loss of mercury occurring. A pure picric acid is obtainable by heating with a further quantity of nitric acid after removal of the benzene, but the yields are reduced. The mercuric nitrate cannot be replaced by nitrates of manganese, copper, aluminium, or zinc.

C. HOLLINS.

Anthracene from oil tar. BESPOLOV. **Hydrolysis of naphthenic acids.** IPATIEV and PETROV.—See II. **Glycerin from molasses.** GOLOVIN.—See XVII.

See also A., Dec., 1397, **Autoxidation of quinol** (DUBRISAY and SAINT-MAXEN). 1403, **Electrolytic chlorination of benzene** (JAYLES). 1436, **meso-Derivatives of anthracene and of 9:9'-dianthranyl** (MINAEV and FEDOROV). 1453, **Perylene and its derivatives** (ZINKE and KOLMAYR). **Linear pentacene series** (HERNLER and BRUNS; MACHEK). 1463, **Naphthoisoindigotins** (WAHL and LOBECK).

PATENTS.

Partial oxidation of gaseous hydrocarbons. H. R. CURME, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,729,711, 1.10.29. Appl., 26.4.22).—A mixture of normally gaseous homologues of methane with an oxidising gas, the former being in excess, is heated above 600° in contact with vitreous material with formation of formaldehyde. After separation of the formaldehyde the mixture is reheated with an additional quantity of the oxidising gas.

D. F. TWISS.

Dehydration of aqueous formic acid. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 303,742, 27.11.28. Belg., 7.1.28).—Aqueous formic acid, preferably concentrated to constant b.p., is continuously added, with an entraining liquid (e.g., isobutyl formate, isoamyl formate, benzene), to a diluent (e.g., *n*-amyl formate, isoamyl formate, butyl ether), which may be identical with the entraining liquid, in a distilling column. Water is removed as azeotropic mixture

with the entraining liquid, whilst a mixture of anhydrous formic acid and diluent is drawn off from the bottom of the column and rectified in a separate still, the heat of the formic acid vapour being utilised in the main column. Three variations are figured.

C. HOLLINS.

Manufacture of aliphatic anhydrides. H. DREYFUS (B.P. 318,662—4, 7.6.28).—As catalysts in the anhydriation of acetic and other aliphatic acids at 300—700° there are used: (A) chloroacetic acid, chlorinated or brominated acetic or propionic acids, sulphoacetic acid, or acetylphosphoric acids; (B) phenol, cresols, xylenols, or their acetates; or (C) glycerol, other polyhydric alcohols or their acetates. The amount of catalyst is 4—7% of the acid.

C. HOLLINS.

Production of benzoic acid. G. BARSKY and I. V. GILES, Assrs. to AMER. CYANAMID CO. (U.S.P. 1,734,029, 5.11.29. Appl., 8.3.26).—Equimolecular quantities of benzene, cyanuric chloride, and aluminium chloride are mixed and boiled.

F. G. CLARKE.

Manufacture of condensation products of the benzodiazine series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,076, 16.1.28. Addn. to B.P. 309,102; B., 1929, 935).—The process of the prior patent is applied to monochloroquinazolines. 4-Chloroquinazoline is condensed with H-acid or α -aminoanthraquinone; 4-chloro-6-methylquinazoline with 1-*p*-aminophenylpyrazolone-3-carboxylic acid; 2- or 4-chloroquinazoline with *p*-phenylenediamine-sulphonic acid; 4-chloro-6-nitroquinazoline with J-acid, β -naphthylamine, 5-aminosalicylic acid, 2:6-dichloro-*p*-phenylenediamine, ammonia, *p*-phenylenediamine (0.5 mol.), 5-sulphinosalicylic acid, or 4-nitro- α -naphthol-5-sulphonic acid; 4-chloro- $\beta\beta$ -naphthaquinazoline with H-acid; 4:4'-dichloro-6:6'-diquinazoly, m.p. 270°, with *N*-*p*-aminobenzoyl-H-acid (2 mols.).

C. HOLLINS.

Manufacture of emulsions from organic iso-colloids which have undergone a modifying treatment, and of new materials therefrom. L. AUER (B.P. 318,562, 4.5.28. Cf. B.P. 287,943; B., 1928, 398).—The "modified" oils of the prior patent are emulsified with water in presence or absence of emulsifying agents etc., by which process further "modification" is effected at once or on keeping. The emulsions may subsequently be treated with "modifying agents," or may be vulcanised. The aqueous medium may be removed by the usual methods and a rubber-like mass obtained.

C. HOLLINS.

Production of carbon disulphide. M. PIER and K. WINKLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,735,409, 12.11.29. Appl., 5.1.28. Ger., 12.1.27).—See B.P. 293,172; B., 1928, 634.

Producing simultaneously formates and gaseous mixtures rich in hydrogen. G. CLAUDE and A. H. GOSSELIN, Assrs. to SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE & PROD. CHIM. (U.S.P. 1,735,107, 12.11.29. Appl., 9.1.26. Fr., 17.1.25).—See B.P. 246,168; B., 1927, 796.

Vaporisation of formamide. E. MÜNCH, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,735,407, 12.11.29.

Appl., 17.1.28. Ger., 27.1.27).—See B.P. 301,974; B., 1929, 163.

Manufacture of acetic anhydride from acetic acid. F. A. HENGLEIN and F. SCHLEICHER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,735,433, 12.11.29. Appl., 12.9.27. Ger., 22.9.26).—See B.P. 301,562; B., 1929, 122.

Catalytic oxidation of organic compounds. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,735,763, 12.11.29. Appl., 8.8.27).—See B.P. 295,270; B., 1929, 806.

Polymerisation of diolefines (B.P. 320,960).—See XIV. Anaglyphs (B.P. 301,490).—See XXI.

IV.—DYESTUFFS.

See A., Dec., 1439, **Cleavage of azo dyes by sulphites** (ENGEL). 1460, **Sulphur dyes** (POLLAK and RIESZ). 1469, **Prodigiousin** (WREDE and HETTCHE). **Thiazole derivatives** (RIESZ and HÜBSCH). 1473, **Reaction between proteins and diazotised arylamines** (HEIDELBERGER and KENDALL).

Aniline recovery plant. KREBS.—See III. **Red pigments.** RASQUIN.—See XIII.

PATENTS.

Dyes and dyeing [sulphuric leuco-ester of anthraquinone-1:2-naphthacridone]. R. S. BARNES, J. E. G. HARRIS, B. WYLAN, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 318,090, 22.5.28).—Caledon-red BN or its leuco-compound is treated with pyridine-sulphuric anhydride (etc.) and copper in presence of pyridine at relatively high temperatures, e.g., at 90°.

C. HOLLINS.

Manufacture of [vat] dyes [of the dibenzanthrone series]. SOC. CHEM. IND. IN BASLE (B.P. 294,486, 23.7.28. Switz., 21.7.27. Addn. to B.P. 262,774; B., 1928, 517).—Blue vat dyes, fast to water, are obtained by chlorinating dibenzanthrone with more than 5 pts. of ferric chloride, e.g., in trichlorobenzene at 140–150°.

C. HOLLINS.

Vat dyes by condensation of 1-(4-benzanthronyl-amino)anthraquinones. H. WOLFF, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,711,710, 7.5.29. Appl., 2.7.27. Ger., 1.7.26).—The condensation products of 4-halogenobenzanthrones with 1-aminoanthraquinones are treated with acid condensing agents (sulphuric acid, aluminium chloride). Examples are the treated products from 4-chlorobenzanthrone and 1-aminoanthraquinone (orange-red), 4:9-dichlorobenzanthrone and 2 mols. of 1-aminoanthraquinone (brown) or 1 mol. of 1-amino-4-methoxyanthraquinone (brown), 4-chlorobenzanthraquinone and 6-chloro-1-aminoanthraquinone (red-brown) or 1:6-diaminoanthraquinone (0.5 mol.: violet-brown).

C. HOLLINS.

Green vat dyes [of the dibenzanthrone series]. B. STEIN, W. TRAUTNER, and R. BERLINER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,680, 21.5.29. Appl., 16.9.26. Ger., 20.10.25).—2-Phenylbenzanthrone, m.p. 199–200°, obtained by the action of aluminium chloride at 100–150° on the 3-isomeride, is fused with alkali to give a diphenyldibenzanthrone, which is a bluish-green vat dye.

C. HOLLINS.

Vat dyes [halogenated *ms*-benzodianthrones]. M. A. KUNZ and K. KÖBERLE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,714,427, 21.5.29. Appl., 16.6.27. Ger., 15.3.26).—8:9-Dimethyl-*ms*-benzodianthrene or its 5:10-dichloro-derivative is treated with sulphuryl chloride in nitrobenzene, whereby three or four chlorine atoms are introduced, to give bright orange vat dyes.

C. HOLLINS.

Vat dyes of the isodibenzanthrone series. A. LÜTTRINGHAUS, H. NERESHEIMER, and H. WOLFF, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,704,983, 12.3.29. Appl., 9.12.26. Ger., 25.10.24).—Monohalogenated isodibenzanthrones, giving reddish-violet to violet-blue shades, are obtained by condensing a monohalogenated 3:4-dibenzanthronyl, having positions 4 and 3' free, in presence of alkali.

C. HOLLINS.

Azo dyes. L. LASKA, F. KRECKE, and F. WEBER, Assrs. to GRASSELLI DYESTUFF CORPORATION (U.S.P. 1,709,989, 23.4.29. Appl., 29.9.27. Ger., 5.10.26).—2:6-Hydroxynaphthoic acid is coupled with diazo compounds, especially with diazotised *p*-chloroaniline-*o*-sulphonic acid (yellow-red on wool). Other diazo components mentioned are aniline (yellow-red), 5-nitro-*o*-aminophenol (brown, black-brown on chroming), 6-nitro-*o*-aminophenol-4-sulphonic acid (after-chromed violet on wool), benzidine-2:2'-disulphonic acid (red-brown chrome printed on cotton), *p*-toluidine-3-sulphonic acid (red barium lake).

C. HOLLINS.

Azo dyes [for wool] from hydroxynaphthazine derivatives. W. HERSBERG and H. OHLENDORF, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,705,974, 19.3.29. Appl., 4.10.26. Ger., 9.6.25).—Diazo compounds are coupled with the hydroxynaphthazines obtained by alkaline fusion from sulphonaphthazines. Examples are: 1:5-dihydroxy- α -naphthazine coupled with diazotised sulphanilic acid (yellow), naphthionic acid (red), 4-chloro-*o*-aminophenol-5-sulphonic acid (orange-brown, violet on chroming); 1:5-dihydroxy- $\alpha\beta'$ -dinaphthazine with naphthionic acid (red), 4-aminoazobenzene-4'-sulphonic acid (salmon-red), *p*-sulphobenzeneazo-Cleve acid (red-brown), β -naphthylamine-1-sulphonic acid (bordeaux-red). The 4-chloro-*o*-aminophenol-5-sulphonic acid coupling is specially claimed.

C. HOLLINS.

Monoazo dyes of the pyrazolone series. L. W. GELLER, Assr. to NAT. ANILINE AND CHEM. Co., INC. (U.S.P. 1,711,639, 7.5.29. Appl., 9.10.25).—An *o*-aminonaphtholsulphonic acid is diazotised and coupled with a 1-sulphoarylpyrazolone carrying a methyl or carboxyl group in position 3; e.g., 1:2:4-aminonaphtholsulphonic acid \rightarrow 1-*p*-sulphophenyl-3-methyl-5-pyrazolone (orange-brown on wool, bluish-red after chroming; silk not stained) or 1-(2-chloro-4-sulphophenyl)-3-methyl-5-pyrazolone (yellow-red).

C. HOLLINS.

Manufacture of azo dyes [pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 299,332, 22.10.28. Ger., 21.10.27).—An ester of 2:3-aminonaphthoic acid is diazotised and coupled in substance or on the fibre with β -naphthol or a 2:3-hydroxynaphthoic arylamide. Examples are: methyl or ethyl 2:3-aminonaphthoate with 2:3-hydroxynaphthoic *p*-aniside (bluish-red), β -naphthylamide (bluish-red), or 4-chloro-*o*-aniside (bluish-red).

C. HOLLINS.

Vat dyes [of the thioindigoid series]. K. THIESS, C. J. MÜLLER, K. SCHIRMACHER, and K. ZAHN, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,712,721, 14.5.29. Appl., 20.12.23. Ger., 27.12.22).— $\beta\beta$ -Naphthathioindoxyl is condensed with 5:7-dibromoisatin and the product (violet-red vat dye) is brominated in nitrobenzene for a bluish-claret (bromine enters position 9 of the naphthathioindoxyl residue). Similar dyes are obtained by brominating the product from $\beta\beta$ -naphthathioindoxyl and isatin, 5-bromo-7-methylisatin, or 6-chloro-7-methylisatin or by chlorinating the product from $\beta\beta$ -naphthathioindoxyl and 5:7-dichloroisatin. C. HOLLINS.

Dyes of the thioindigo series. E. HOFFA and H. HEYNA, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,709,982, 23.4.29. Appl., 16.12.26. Ger., 19.12.25).—*O*-Acylated thioindoxyls are condensed with isatins etc., the acyl group being lost during the reaction. Examples are $\beta\beta$ -naphthathioindoxyl *O*-acetate with 5:7-dichloroisatin chloride (blue), 5:7-dichloroisatin (brown-red), 6-chloro-4-methylthioisatin (greyish-blue), acenaphthaquinone (red). C. HOLLINS.

Manufacture of substitution products of sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,776, 22.5.28).—The brown sulphide dyes of B.P. 315,910 (B., 1929, 809) and 317,139 (B., 1929, 896) are converted into orange or bright red dyes by alkylation or aralkylation in substance or on the fibre, particularly by means of phenylbenzyltrimethylammonium chloride. The dyes obtained in substance can no longer be vatted. C. HOLLINS.

Manufacture of organic salts of dyes [lakes etc.]. I. G. FARBENIND. A.-G., G. KRÄNZLEIN, C. HARTMANN, and A. HARDT (B.P. 316,370, 22.5.28. Addn. to B.P. 277,371).—A non-nitrogenous (e.g., iodonium, phosphonium, sulphonium) base is combined with a dye acid to give insoluble pigments. Examples are: the diphenyliodonium salt of 1-amino-4-anilinoanthraquinone-2-sulphonic acid (blue) or of *m*-toluidino-1-benzamido-8-naphthol-4:6-disulphonic acid (red); di-*o*-tolylmethylsulphonium 1:4-di-*p*-toluidino-5:8-dihydroxyanthraquinonedisulphonate (green); *p*-tolyltriethylphosphonium salt of aminoazobenzene- β -naphthol-8-sulphonic acid. C. HOLLINS.

Dianthraquinonylaminesulphonic acid dye. W. MIEG and H. RAEDER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,735,123, 12.11.29. Appl., 17.8.25. Ger., 27.9.24).—See B.P. 240,492; B., 1926, 973.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Resistance of various celluloses to saccharification. P. LEONE (Annali Chim. Appl., 1929, 19, 432—443).—Hydrate celluloses exhibit lowered resistance to saccharification and greater capacity for reaction in other ways. The most readily saccharified are those precipitated from solutions in which the celluloses were dissolved as esters, then, come those precipitated from indifferent solutions, and finally mercerised celluloses, which yield, on the average, one half as much dextrose as the natural celluloses. The action of sodium hydroxide solution becomes appreciable at concentrations above 10%, increasing rapidly to 16—18% and slowly after-

wards. Hydrate celluloses prepared with salt (potassium iodide, zinc chloride) solutions behave, as regards saccharification, like natural celluloses, and the salt solutions do not enhance the action of the soda in mercerisation. The hydrocelluloses exhibit about the same resistance to saccharification as the natural celluloses, with appreciable differences for different types. Use of high pressure increases slightly the yield of dextrose if the saccharifying medium is sulphuric acid, but with hydrochloric acid the influence of high temperature and pressure is scarcely appreciable, and in some cases leads to marked diminution in the amount of dextrose formed. The oxycelluloses are hydrolysed rather more readily than natural celluloses, but their behaviour confirms the non-homogeneity of their constitution; they are partly soluble in alkalis, and the part precipitated from alkaline solution is hydrolysed more readily than the insoluble part, but always less easily than hydrate celluloses precipitated from their solutions. The action of zinc chloride on the saccharification of cellulose is always considerable, even on mercerised cellulose, and greatly exceeds that of the mercerising action itself. The insoluble matter obtained on saccharification of cellulose with sulphuric acid under pressure consists largely of partially hydrolysed products of reducing character. T. H. POPE.

Developments in nitrocellulose production. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 1044—1047).—Improvements in the methods in operation at the Union plant of the Hercules Powder Co. for handling raw materials and products etc. are described. A notable development is the use of chrome iron for the construction of equipment which comes in contact with strong acid, whereby its life is lengthened and contamination of the product is prevented. F. R. ENNOS.

[Cellulose from] lemon residues. MELIS.—See XIX.

See also A., Dec., 1427, **Fractional precipitation of cellulose acetate** (McNALLY and GODBOUT). 1428, **Acetylation and degradation products of lignin** (FRIESE). 1429, **Acetylated wood** (SUIDA and TITSCH). 1473, **Structure of silk fibroin** (ABDERHALDEN and BROCKMANN). 1498, **Determination of lignin by acid hydrolysis** (PALOHEIMO).

PATENTS.

Degreasing of animal fibres. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 311,394, 7.2.29. Ger., 11.5.28).—After saturation with water or an aqueous solution, the wet material is extracted by means of chlorinated hydrocarbons. F. R. ENNOS.

Treatment of jute fibre and analogous fibres. TEXTILES (NEW PROCESS), LTD. (B.P. 321,140, 18.4.29. Fr., 8.10.28).—The fibre is treated in a bath at 30—33° containing a culture of bacteria obtained from the fermentation of flax and/or hemp, together with potassium phosphate. After removal from this bath it is dried until it contains about 30% of water, passed through the breaker carding machine, mercerised, bleached, treated with an emulsifiable oil, and dried.

F. R. ENNOS.

Manufacture of artificial silk. O. FREHERR VON KOHORN (O. KOHORN & Co.), and A. PERL (B.P. 312,029, 26.7.28. Austr., 18.5.28).—In the cuprammonium stretch-spinning process, the formation of air bubbles in the precipitating liquid may be avoided if the latter is partly freed from its contained gases by keeping it before use at 10–15° above the spinning temperature, and by preventing the production of under-pressure in the spinning apparatus. F. R. ENNOS.

Preparation of artificial silk threads and fabrics. F. J. GAHLERT (B.P. 320,869, 19.5.28).—To produce crêpe yarns, an already prepared and twisted artificial silk thread is softened, sized under tension, dried, and rewound with additional twist. F. R. ENNOS.

Manufacture of cellulose from vegetable fibre. A. G. POLLARD and J. R. WHINCOP (B.P. 321,282, 25.9.28).—Soaked or moistened cellulose material is suitably teased or loosened, treated with chlorine gas, subsequently steeped in an alkali solution of less than 3% concentration, beaten, and washed. Surplus water is removed from the pulp and the chlorine treatment repeated, after which the pulp is washed successively in a slightly alkaline bath (0.1%), in water, in a dilute acid solution, and finally in water till neutral. The process is substantially cold throughout, and the pulp has a high α -cellulose content and is readily esterified. A. G. POLLARD.

Treatment of cellulose-containing materials with mineral acids. INTERNAT. SUGAR & ALCOHOL Co., LTD. (B.P. 299,844, 24.10.28. Ger., 2.11.27).—For the saccharification of cellulose-containing material with hydrochloric acid on the diffusion principle, the material is premashed with a portion of the total acid required and, after transferring the mixture to the diffuser, the treatment is continued with the balance of the acid. The proportions of acid used are such that the temperature reached in the diffuser is below 50°, preferably 25–30°. Premashing for 4 hrs. with half of the total acid or with the acid solution of partially hydrolysed cellulose from a previous diffuser treatment is suggested. A. G. POLLARD.

Production of a liquid- and moisture-proof transparent and flexible material. WOLFF & Co., KOMMANDIT-GES. AUF AKT., R. WEINGAND, and F. W. SPIECKER (B.P. 309,017, 27.7.28. Ger., 3.4.28).—Transparent foils of regenerated cellulose are coated with a cellulose ester lacquer, with which is incorporated a solution of a solid wax, a liquid wax or grease, and a natural or an artificial resin in a solvent miscible with the cellulose ester lacquer, the applied film being exposed for a time to a temperature above the softening point of the waxes. F. R. ENNOS.

[Treatment of] textile materials. R. P. FOULDS, J. T. MARSH, and F. C. WOOD, ASSRS. to TOOTAL BROADHURST LEE Co., LTD. (U.S.P. 1,734,516, 5.11.29. Appl., 2.11.28. Swed., 7.11.27).—See B.P. 291,474; B., 1928, 636.

Electrical insulator (B.P. 297,749).—See XI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Aniline recovery plant. KREBS.—See III.

PATENTS.

Treatment of felt and felt-hat bodies. E. BÖHM

(U.S.P. 1,729,474, 24.9.29. Appl., 2.9.26. Austr., 1.6.26).—The lustre of felt is increased by treatment with an aqueous solution of a complex compound of a heavy metal of the chromium group. W. G. CAREY.

Preparation of moth-proof articles. W. LOMMEL, H. MÜNZEL, H. STÖTTER, and B. WENK, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,734,682, 5.11.29. Appl., 2.7.27. Ger., 13.7.26).—See B.P. 274,425; B., 1928, 743.

[Apparatus for] printing textile fabrics. F. ADLER (B.P. 321,603, 18.1.29. Addn. to B.P. 317,122).

Treatment of textile materials in roll form with liquids. G. P. and G. R. ROBERTS (B.P. 321,183, 31.7.28).

Dyeing (B.P. 318,090).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

[Bromocresol-green as] indicator for ammonia titrations. L. H. BAILEY (Cereal Chem., 1929, 6, 454–456).—The various indicators for use in the Kjeldahl distillation of ammonia are briefly reviewed and their limitations described. The neutral points of ammonium chloride and ammonium sulphate are at p_H 5.1 and 5.7, respectively, and the indicator the colour change of which most nearly approaches these values is bromocresol-green. This indicator has a colour range of p_H 4.0–5.6, being yellow in acid solution and blue in excess alkali, passing through an intermediate green at about p_H 5.0. H. J. DOWDEN.

Investigations [of the Shanks system for lixiviation and crystallisation of caliche etc.]. R. GUILIER (Caliche, 1929, 11, 241–247).—Details are given of the technical application of the system. II. F. GILLBE.

Basic processes of manufacturing alumina. G. G. URAZOV and Y. E. VILYANSKI (J. Appl. Chem., Russia, 1928, 1, 271–283).—Sodium chloride has no appreciable effect on the reaction between alumina and sodium carbonate at 500–1050°; the rate of reaction increases with increase in the amount of sodium carbonate present, and depends on the nature of the raw material. Bauxite is preferably fused with sodium carbonate at 980° for 2 hrs. Quick leaching with water leaves 25% of the alumina insoluble; the yield of alumina is largely increased by addition of sodium hydroxide.

CHEMICAL ABSTRACTS.

Soda-lime process of manufacturing alumina from bauxite and aluminium silicates rich in silica. G. G. URAZOV, Y. E. VILNYANSKI, and Y. A. MORACHEVSKI (J. Appl. Chem., Russia, 1928, 1, 77–96).—Fusion of aluminium silicates with sodium carbonate affords principally the compounds 1–1.5Na₂O, Al₂O₃, 2–2.5SiO₂ and Na₂O, Al₂O₃. The insoluble residue has a composition corresponding approximately with Na₂O, Al₂O₃, 2SiO₂. The optimum fusion temperature is 850–950°. When nepheline was fused with lime and the mass leached with water the insoluble residue contained all four oxides. When clay was fused with sodium and calcium carbonates more water-soluble soda and alumina were formed than with nepheline. Conditions under which all of the alumina but no silica is extracted were not found. CHEMICAL ABSTRACTS.

Barium process of manufacturing alumina from bauxites and aluminium silicates rich in silica. G. G. URAZOV and A. V. CHITAEV (J. Appl. Chem., Russia, 1928, 1, 96—109).—By fusion at 1000° or 1200° with barium carbonate 55 or 88—98%, respectively, of alumina becomes soluble. In presence of sodium chloride 93% is so converted at 1000°. Maximum solubility (at 60°) corresponds with the ratio $\text{Al}_2\text{O}_3 : 2\text{BaCO}_3$; no compounds are formed in the system $\text{BaO}-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$. In the system $\text{Ba}(\text{OH})_2-\text{NaCl}-\text{H}_2\text{O}$ (at 60°) no compounds are formed; the highest solubility corresponds with a molecular ratio $\text{BaO} : \text{NaCl} = 1.17 : 1$. In the system $\text{BaO}-\text{Al}_2\text{O}_3-\text{NaCl}-\text{H}_2\text{O}$ (at 60°) the presence of sodium chloride has no effect on solubility. As compared with the fusion of bauxites with sodium carbonate, fusion with barium carbonate converts less alumina into a soluble form, but the product contains less silica.

CHEMICAL ABSTRACTS.

Analysis of lead peroxide. A. V. PAMFILOV (J. Russ. Phys. Chem. Soc., 1929, 61, 1131—1143).—A number of methods which have been suggested for the analysis of lead peroxide are reviewed. A modification of Figg's method (B., 1925, 215) is proposed, in which dissolution is accelerated by triturating 1 g. of lead peroxide with 40 c.c. of saturated sodium acetate in 5% acetic acid solution and adding 10 c.c. of 10% potassium iodide solution. Results obtained agree closely with those obtained by the iodometric methods of Glasstone (B., 1922, 98 A) and of Milbauer and Pivnička (B., 1914, 591), and the thiosulphate method of Liebig (B., 1901, 1027).

R. TRUSZKOWSKI.

See also A., Dec., 1376, **Mixed absorbents** (SCHILOV and others). 1388, **System water-carbon dioxide-ammonia** (JÄNECKE). **Equilibrium between water and the nitrates, chlorides, etc. of sodium and potassium** (CORNEC and KROMBACH). **System $\text{AlCl}_3-\text{FeCl}_3-\text{KCl}-\text{HCl}-\text{H}_2\text{O}$ at 25°** (MALQUORI). 1400, **Oxidation of ammonia to calcium nitrate** (LESCHIEWSKI and others). 1401, **Active oxygen** (COPELAND). 1402, **Electrolytic production of fluorine** (FREDENHAGEN and KREFFT). 1411, **Potentiometric determination of sulphuric acid** (LINDA and ETTINGER). 1498, **Iodine from Black Sea weeds** (AVERKIEV). **Iodine content of marine algæ from Galician coasts** (PONDAL).

Preservation of hydrogen peroxide. TELLERA.—See XX.

PATENTS.

Sodium cyanide compound and its production. K. F. COOPER (U.S.P. 1,734,562, 5.11.29. Appl., 1.11.22).—To produce sodium cyanide from the crude compound derived from crude cyanamide, the last-named is dissolved in water and soluble calcium salts are precipitated therefrom by adding a suitable sodium salt; the liquor is then filtered, sodium chloride added, and the solution concentrated and cooled to separate sodium cyanide dihydrate, which is dried under reduced pressure.

W. G. CAREY.

Alkali metal [sodium] compound of a suboxide of titanium and derivatives therefrom. F. VON BICHOWSKY (U.S.P. 1,731,364, 15.10.29. Appl., 4.6.23).—Titanium cyanonitride or carbide is heated at 800° with sodium carbonate whereby a mixture of sodium cyanide

and the blue compound, $2\text{Na}_2\text{O} \cdot \text{Ti}_2\text{O}_5$, is obtained. This compound is decomposed by water yielding the compound, $\text{Na}_2\text{O} \cdot 2\text{Ti}_2\text{O}_5$, as a lavender-coloured powder which on roasting in air affords a voluminous white mass of sodium hexatitanate ($\text{Na}_2\text{Ti}_6\text{O}_{13}$) of value as a pigment.

A. R. POWELL.

Mercuric cyanide tartrate and its manufacture. M. MIYAHARA (U.S.P. 1,732,106, 15.10.29. Appl., 4.6.28. Jap., 9.3.28).—A solution of 1 mol. of mercuric oxycyanide and 1 mol. of tartaric acid is dissolved in alcohol and the solution is evaporated. On cooling, needles of the compound $\begin{matrix} \text{CH}(\text{OH})\text{CO}_2 \\ | \\ \text{CH}(\text{OH})\text{CO}_2 \end{matrix} > \text{Hg}, \text{Hg}(\text{CN})_2, \text{H}_2\text{O}$

separate. The same substance may be prepared from aqueous solution if a small quantity of sodium tartrate or aluminium borate is added to prevent hydrolysis during evaporation.

A. R. POWELL.

Manufacture of dispersions containing copper. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H., F. KAUFER, and F. X. SCHWABEL (B.P. 321,260, 23.8.28).—Suspensions of copper oxochloride are prepared by treating cupric chloride solution in contact with copper with air or other gases containing oxygen in the presence, if desired, of dispersion-promoting colloids, e.g., dextrin, sulphite-cellulose waste liquor. Such dispersions find application as plant-spraying materials.

L. A. COLES.

Manufacture of lead compounds [oxides]. C. H. BRASELTON (U.S.P. 1,734,285, 5.11.29. Appl., 20.12.20).—A mixture of air and ozone is passed through molten lead maintained at a high temperature, the degree of oxidation being controlled by the quantities and temperature of the mixed gases.

W. G. CAREY.

Manufacture of iron carbonyl. M. MÜLLER-CUNRADI, ASST. to I. G. FARBENIND. A.-G. (U.S.P. 1,725,619, 20.8.29. Appl., 19.10.25. Ger., 5.1.25).—Carbon monoxide under pressure is passed downwards through a heated mass of coarse-grained iron.

A. R. POWELL.

Manufacture of aluminium chlorosulphate. H. BLUMENBERG, JUN. (U.S.P. 1,734,196, 5.11.29. Appl., 18.5.27).—A dehydrated mixture of aluminium oxide and aluminium sulphate is treated with hydrogen chloride fumes.

H. ROYAL-DAWSON.

Preparation of thorium hydride. H. S. COOPER, ASST. to KEMET LABS. Co., INC. (U.S.P. 1,730,723, 8.10.29. Appl., 3.10.25).—Pure thorium powder is heated at 300—375° in an atmosphere of hydrogen.

A. R. POWELL.

Treating [refining] sulphur. E. F. WHITE (U.S.P. 1,734,006, 5.11.29. Appl., 7.4.26).—Sulphur is vaporised and the vapour subjected to mechanical shock to cause partial condensation, the remainder being then cooled.

H. ROYAL-DAWSON.

Production of phosphorus [penta]chloride from phosphate rock. C. G. MINER (U.S.P. 1,730,521, 8.10.29. Appl., 6.7.26).—Briquettes made of phosphate rock, silica, and carbon are heated at a white heat in a reducing atmosphere in the presence of excess chlorine, and the issuing gases are cooled below 160°, whereby

the trichloride combines with the excess chlorine to form the pentachloride. A. R. POWELL.

Production of alkaline or alkaline-earth salts of oxalic acid and saturated monobasic fatty acids. KEMIKAL, INC., Assees. of I. S. MELLANOFF (B.P. 307,784, 1.8.28. U.S., 13.3.28).—See U.S.P. 1,681,156; B., 1928, 857.

Halogen-resistant refractories (B.P. 304,694).—See VIII. **Electrolytic production of oxygen and hydrogen** (B.P. 320,388). **Cells for production of gases** (B.P. 318,734). **Cells for electrolysis of water** (B.P. 292,131).—See XI.

VIII.—GLASS; CERAMICS.

Fuel utilisation in the ceramic industries. A. T. GREEN and A. J. DALE (J. Inst. Fuel, 1929, 3, 102—122).—The influence of the firing operation on clay goods, the thermal reactions involved, the firing of refractory products and of glazed and decorated pottery wares are discussed. Various types of intermittent and continuous kilns and improved methods for their better thermal insulation are described.

C. B. MARSON.

Plasticity of clay. L. E. JENKS (J. Physical Chem., 1929, 33, 1733—1757).—The relative plasticities of different commercial clays and ceramic materials have been measured by a method which depends on the time taken for a ball of the clay to disintegrate when immersed in water, and the effects of the amount of water used in moulding the ball and the time of drying on the relative plasticity have been investigated. The plasticity of kaolin is noticeably increased by the chlorides of the alkalis and alkaline-earths only after a critical concentration, specific for each salt, has been reached, but with the exception of lithium salts the high plasticity does not survive subsequent treatments of the kaolin. The carbonates of the alkalis and alkaline-earths also confer increased plasticity and at lower concentrations than the corresponding chlorides. The addition of lithium chloride to dialysed gelatinous alumina appears to confer a plasticity similar to that of a clay, but this is not the case with gelatinous silica. The effect of the addition of mixtures of electrolytes on the plasticity of kaolin may be additive in certain cases, but, in general, it is greater or less than the sum of the individual effects. The action of flocculating agents in peptising solutions on various clays has also been examined. Criteria of plasticity are discussed, and from the above results it is concluded that the cause of plasticity is the formation in the moistened clay of a continuous, gelatinous, external phase through the action of water on the material forming an enveloping surface around the non-gelatinous particles and aggregates present in clay. The presence of organic matter, carbon dioxide, or electrolytes in the supernatant water or in the clay itself is not the cause of plasticity; these are agents forming the necessary gelatinous material.

L. S. THEOBALD.

Silica refractories for coke ovens. R. RICHARDS (Proc. South Wales Inst. Eng., 1929, 45, 419—461).—The advantages of silica over fireclay refractories

are: greater refractoriness under load, resistance to effect of salty coals, higher heat conductivity, and less tendency to spalling. Each stage of manufacture is considered, particular attention being given to "crushing," as this determines the porosity of the finished product. Correct mixing of particle sizes produces a material of low porosity (22—24%), with a consequently higher conductivity and lower impermeability to gases. The constitution of various raw materials and the changes occurring during the "burning" process are illustrated by photomicrographs. The specifications and methods of test applied by a Continental firm include refractoriness (with and without load), sp. gr., porosity, and thermal expansion.

J. A. SUGDEN.

See also A., Dec., 1407, **Effect of heat on crystalline aluminium hydroxide** (ROOKSBY).

PATENTS.

Manufacture of compound glass. J. H. ROBERTSON (B.P. 321,157, 27.7.28).—A coating of gelatin in phenol on the glass sheets is allowed to set, and its surface is then softened in a bath of ethyl or propyl alcohol. A celluloid sheet is then sandwiched between the sheets of glass and the whole placed in a heated press. Alternatively, the phenol-gelatin layer may be coated with a thin layer of a solution of celluloid in amyl acetate, or the celluloid replaced by cellulose acetate coated with gelatin or celluloid. A. COUSEN.

Non-splintering glass. C. H. FIELD and D. HASLETT (B.P. 321,178 and 321,189, 27.7.28).—(A) Adhesion between glass and cellulose ester sheets is secured by means of phenol-formaldehyde, phenol-formaldehyde-acetone, phenol-formaldehyde-glycerin, phenol-urea-formaldehyde, or glycerin-polybasic acid condensation products, or mixtures of these. The resins may be applied alone or with other materials such as Canada balsam, euphorbium resin, or cellulose ester solutions. (B) The composite sheet, as formed, is submitted simultaneously to pressure, in order to force the sheets together, and to a vacuum, to draw the air and solvent vapours from between the sheets. A. COUSEN.

Bonded refractory. J. D. MORGAN, Assr. to DOHERTY RES. Co. (U.S.P. 1,734,595, 5.11.29. Appl., 17.2.27).—A mixture of zircon and monazite sand is treated with a mineral acid and the mixture is fired. W. G. CAREY.

Manufacture of [halogen-resistant] refractory vessels etc. N.V. ELECTROCHEM. IND. (B.P. 304,694, 23.1.29. Holl., 24.1.28).—Apparatus for the treatment of compounds containing oxygen simultaneously with reducing agents and chlorine is lined with or constructed of insoluble halides, e.g., calcium fluoride, silver chloride. L. A. COLES.

Grinding or abrading means. ARTIFEX CHEM. FABR. G.M.B.H. (B.P. 309,441, 12.2.29. Ger., 10.4.28).—Friable abrasive blocks comprising a caoutchouc substitute (factice) as binder are prepared by adding a liquid which does not react with sulphur or sulphur chloride, e.g., hydrocarbons, their halogen substitution products, hydrogenated naphthalene, to a mixture of

a fatty oil, *e.g.*, rape-seed oil, with sulphur or sulphur chloride and an abrasive powder. L. A. COLES.

Refractory product. I. HARTER and A. M. KOHLER (U.S.P. 1,734,726, 5.11.29. Appl., 25.5.23).—See B.P. 216,514; B., 1925, 718.

Apparatus for feeding of molten glass. T. F. PEARSON (B.P. 321,195, 4.5.28).

IX.—BUILDING MATERIALS.

Reactions in burning cement. A. C. DAVIS (Cement, 1929, 2, 303—314).—Existing knowledge of the heat changes in the dehydration of clay and the formation of cement clinker is reviewed. The latter has been investigated (*a*) by determination of the heat of formation of the component silicates and aluminates, (*b*) by direct reaction in a bomb calorimeter, (*c*) by use of heating curves, and (*d*) by measuring the heat of dissolution of the raw materials and of clinker in acids. The latest determinations (Coghlan, Nacken; by methods *a* and *d*, respectively) give the values 99.16 and 100 kg.-cal. evolved per kg. of clinker formed. R. WENTWORTH.

Spray process of slurry feed. S. J. M. AULD (Cement, 1929, 2, 291—296).—In the Rigby spray-feed for rotary kilns a slurry containing 40% of water is sprayed in counterflow to the hot gases, sufficient evaporation taking place before the spray touches the walls to avoid adherence. Dried slurry is removed from the kiln gases by a lute in the smoke chamber. A decrease of 15—20% in fuel consumption per unit weight of cement is reported, and an increase in output of 20—30% per kiln. R. WENTWORTH.

Manganese in cement clinker. Constitution of cement. A. GUTTMANN and F. GILLE (Zement, 1929, 18, 500—505, 537—541, 570—574; Chem. Zentr., 1929, ii, 206).—In ordinary Portland cement clinker the manganese is present as Mn_2O_3 ; more than 3.3% is undesirable. In high-lime clinker the manganese is contained in the celite. In cements rich in manganese but abnormally poor in alumina quadrivalent manganese is present. A. A. ELDRIDGE.

Physico-chemical principles underlying methods of testing the constancy of volume of Portland cement. I. A. ALEKSANDROV (J. Appl. Chem., Russia, 1929, 2, 303—316).—Fajja's method is not sufficiently severe; Heinzel's method is misleading. Le Chatelier's method is acceptable. Drying at 120° followed by treatment with steam affords promise of a practical method. Storage under water for 28 days yields valuable results. CHEMICAL ABSTRACTS.

PATENTS.

Waterproofing of stone. S. S. SADTLER, Assr. to AMESITE ASPHALT CO. OF AMERICA (U.S.P. 1,730,245, 1.10.29. Appl., 24.3.27).—See B.P. 309,634; B., 1929, 520.

[Grooved rock-asphalt] road pavements and the like. HIGHWAYS CONSTRUCTION, LTD., and C. W. RHODES (B.P. 321,544, 6.11.28).

Concrete building blocks [lined with sugar-cane fibre] and their manufacture. H. S. PALMER (B.P. 321,435, 7.7.28).

Cooling apparatus (B.P. 321,036).—See I. Pitch composition (B.P. 295,687).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Corrosion of metals and its prevention. H. E. YERBURY (Proc. Inst. Mech. Eng., 1929, 545—558).—A review of known facts as to the causes of corrosion, with especial reference to engineering materials, and an account of modern methods of preventing corrosion by the development of materials more highly resistant to corrosive media and by the use of suitable protective coatings. A. R. POWELL.

Pickling [of iron and steel]. K. TAUSSIG (Arch. Eisenhüttenw., 1929—30, 3, 253—266; Stahl u. Eisen, 1929, 49, 1655—1656).—The rate of scale removal from iron and steel by sulphuric acid of concentrations between 2 and 20% with and without the addition of inhibitors has been determined at temperatures between 10° and 80°. Ferrous sulphate retards the dissolution of iron in the pickling liquor only when the acid concentration is less than 10%. Feebly acid liquors at low temperatures remove scale only slowly, so that it is preferable to work with hot acid of medium concentration (*e.g.*, 20%) until the concentration falls to 10%. The liquor is then cooled to allow ferrous sulphate crystals to separate and the mother-liquor is used again in making up a fresh bath. A. R. POWELL.

Volume change of cast iron during solidification. K. HONDA, T. KASE, and Y. MATUYAMA (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 527—538).—The results of Honda and Endo (A., 1926, 897) have been verified. F. J. WILKINS.

Nature of the fracture of steel and the significance of the transition region in the notched-bar impact test. F. FETTWELLS (Stahl u. Eisen, 1929, 49, 1621—1627).—As a result of a review of the earlier work of himself and others on the variation of the impact strength of notched steel bars with the conditions of testing, the author concludes that the transition zone in the impact-test curves, in spite of its varying shapes, always conforms to the same laws; thus, if in the curve showing the relation between impact strength and width of the test piece this zone is broad and irregular, it will similarly appear in the temperature-impact strength curve. The change from fibrous to granular fracture is always irregular even when the impact strength shows a gradual transition. The fibrous fracture is not connected with any definite crystal plane, but granular fracture takes place along crystallographically defined cleavage planes. The nature of the fracture depends entirely on the state of strain set up inside the metal in opposition to the breaking force. A. R. POWELL.

Volumetric determination of carbon in difficultly combustible iron and steel alloys. E. ZINDEL (Chem.-Ztg., 1929, 53, 891).—As a catalyst in the combustion of ferro-alloys and complex alloy steels the addition of soft iron or mild steel turnings of known carbon content is recommended in place of the usual bismuth oxide. Complete combustion of all the carbon is effected in a short time at the temperature required

for a plain carbon steel, and there is no action on the walls of the tubes such as is obtained when using bismuth oxide.

A. R. POWELL.

Determination of manganese and iron by successive titration with permanganate. I. S. TELETOV and N. N. ANDRONNIKOVA (*J. Appl. Chem., Russia*, 1929, 2, 289—292).—The solution, containing ferric and manganous ions, is treated with sodium hydroxide and hydrogen peroxide, the precipitate being washed with water and dissolved in 0.1*N*-oxalic acid solution containing sulphuric acid. For the determination of iron the solution is filtered through reduced cadmium and again titrated with permanganate. **CHEMICAL ABSTRACTS.**

Smelting in the lead blast furnace. I. Approximation of the form of the lead in slag and other products. II. Gases from the top of the furnace. G. L. OLDRIGHT and V. MILLER (*U.S. Bur. Mines, Rep. Invest.*, 1929, No. 2954, 8 pp.; 2957, 18 pp.).—I. The sample (0.5–5 g.) is dissolved in ammonium acetate solution, diluted to 100 c.c., and boiled for 10 min.; the solution is filtered and the residue washed. The filtrate contains lead originally present as oxide, sulphate, or basic sulphate. The residue is treated for 1 hr. at the ordinary temperature with 10% silver nitrate solution (10–20 c.c.), the mixture being then filtered and the residue washed. The filtrate contains the lead originally present as metal. The residue is treated at 25–28° for 12 hrs. with 25–30 c.c. of saturated aqueous sodium chloride containing ferric chloride (60 g. of hexahydrate per litre). The filtrate contains lead originally present as sulphide, and the residue that as silicate.

II. The furnace gases contain very little sulphur. The carbon monoxide content is similar to that of iron blast-furnace gas. Analyses of samples from different positions are recorded.

CHEMICAL ABSTRACTS.

Double [metal] as a constructional material for apparatus. G. DURST (*Chem.-Ztg.*, 1929, 53, 837–838).—For the construction of large vessels with an acid-resisting inner surface, double metal, consisting of silver or 18-carat gold on a backing of copper or bronze, provides a material having a long life. In order to prevent diffusion of the silver or gold into the base metal a thin layer of nickel or a nickel-iron alloy is interposed between the precious and base metals during the welding operation, which is effected by a pressure of 100 atm. at 700–800°. After rolling into sheet the latter may be fabricated into apparatus in the usual way; joints may be made by welding the inner surface with the appropriate precious metal, after which the outside of the joint is filled in with silver solder or simply brazed.

A. R. POWELL.

Electrolytic preparation of zinc. G. EGER (*Chem.-Ztg.*, 1929, 53, 857–858, 878–879).—Technical practice in the preparation of electrolytic zinc in Europe and America is briefly reviewed. The two methods which have been tried, using soluble anodes, as in copper refining, and inert anodes, are discussed. In modern operations the latter only is used. The electrolyte may be either of zinc chloride or sulphate, but difficulties encountered when employing the former, such as utilisation of the anodic chlorine and the need of diaphragms,

have resulted in the adoption of the sulphate method only. Modern practice, employing a zinc sulphate solution, lead anodes, and aluminium cathodes, is considered under the headings: (a) the roasting of the ore, (b) the leaching of the roasted ores and purification of the liquors, (c) the electrolysis and production of metallic zinc, (d) the melting of the zinc cathode, and casting in bars. For the production of a good zinc deposit the heavy-metal impurities should not exceed (mg. per litre of electrolyte): copper 10, arsenic 1, iron 30, cadmium 12, cobalt 1. A current density of 340 amp. per m.² of cathode with a current efficiency of 90% is desirable, the zinc deposit being stripped from the cathodes every 24–48 hrs. A current strength of 8000–11,000 amp. at 500 volts (total) is applied across all the cells in series, giving a potential drop across each cell of 3.2–3.6 volts. Flow sheets are given for this and for the Tainton processes. In this latter process more concentrated (30%) sulphuric acid is used for leaching the roasted ores and a current density of 1000–1100 amp. per m.² is employed. The cells are not in cascade, but on the same level, whilst the solution flows in and out of each one separately, the energy consumption being 4–4.5 kw.-hrs. per kg. of zinc. The anodes in this case consist of lead-silver alloy; addition of certain organic and inorganic substances to the solution is found to prevent the anodic evolution of gases containing acid.

G. E. WENTWORTH.

Cadmium plating. I. T. WATANABE and C. TSUCHIMOTO (*J. Min. Met. Japan*, 1929, 7, 3–10, 34–40).—When cadmium is deposited on iron from cyanide solutions the cathode current efficiency scarcely changes up to the point of gas evolution (1.75 amp. per dm.²). If the current density is too low the deposit is crystalline. The concentration has little effect on current efficiency, but the current density necessary to produce a good deposit tends to increase with concentration. The character of the deposit is good when the potassium cyanide concentration is above 1.5*N*. The effect on the deposit of the addition of various substances has been studied. Cadmium is more effective than zinc in protecting iron.

CHEMICAL ABSTRACTS.

[Treatment of] manganese ore by the Bradley process. C. ZAPFFE (*Min. and Met.*, 1929, 10, 428–429).—The separation of manganese and iron in mixed ores of varying composition is effected by roasting the ore and leaching out manganese oxide by means of a solution of ammonium sulphate. Iron oxide and silica are present in the residue, from which iron oxide is separated by magnetic wet concentration. Manganese hydroxide is precipitated from the solution by means of ammonia, evolved and collected from the leaching process, re-forming ammonium sulphate in solution, which after concentration is used again for leaching; the process thus becomes cyclic. In practice the extraction is made in two stages, freshly roasted ore being treated with liquor already heavily charged with manganese sulphate and later with a fresher solution of ammonium sulphate to complete the extraction, which is of the order of 90%. Excess of ammonium sulphate in solution covers possible variation in the composition of the ore. A plant of commercial size is proposed.

C. A. KING.

Lime process for coating aluminium. L. McCULLOCH (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 3 pp.).—A dead-white coating can be produced on aluminium by boiling in an aqueous suspension of lime and calcium sulphate. The coating appears to consist mainly of hydrated aluminium oxide with a certain proportion of calcium sulphate. It is extremely fine-grained and adherent, but not so hard or resistant to corrosion as that produced by anodic treatment of aluminium. Some advantages of the process are pointed out.

H. J. T. ELLINGHAM.

Inverse segregation in duralumin. S. M. VORONOV (Z. Metallk., 1929, 21, 310—316).—Commercial aluminium, even that containing 99—99.5% Al, and all solid-solution aluminium alloys exhibit the phenomenon of inverse segregation on casting into ingots. With aluminium containing 0.4—0.5% Fe and 0.3% Si the outer skin of the ingot frequently contains 1.3—1.7% Fe and 0.75% Si. Small exudations which appear on solid-solution alloys of aluminium with copper, magnesium, or nickel may contain 3—4 times as much of the alloying element as does the remainder of the ingot. Some tests on duralumin showed a gradually decreasing copper content from 5.05% at the outside of the ingot to 3.65% in the middle when cast under normal conditions; after casting from 750° the difference between outside layer and core amounted to 0.28% Cu only. Casting in a mould preheated at 200° still further reduced the segregation owing to the slower rate of cooling permitting diffusion to take place. The occurrence of inverse segregation in a duralumin ingot leads to defects in the finished material after rolling and heat treatment; to avoid segregation the metal should be somewhat overheated and cast into preheated moulds as rapidly as possible. In any case removal of the casting skin by planing is to be recommended before rolling.

A. R. POWELL.

Non-porous aluminium alloy for vacuum-chamber castings. E. C. NICHOLS (J. Opt. Soc. Amer., 1929, 19, 164—165).—The alloy consists of 95% Al and 5% Si. It has an ultimate strength of 20,000 lb./in.², elongation 4.6%, Brinell hardness 40. It should be cast at 677°, and is rendered closer grained by annealing at 400°. A vessel made thereof maintained a vacuum of 0.008 mm. of mercury practically unchanged for a week.

C. A. SILBERRAD.

Chemical resistance of aluminium coated with anodic films. S. SETOH and A. MIYATA (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 900—916).—Aluminium may be oxidised anodically in a dilute solution of oxalic acid. Corrosion and pitting may be prevented by superposing an alternating current on the direct current. If the film-coated aluminium is treated with high-pressure steam for a few minutes, it is waterproofed and its chemical resistance is increased. The chemical resistance is limited by the porosity of the film, and perfect protection can be obtained only with pure aluminium.

C. W. GIBBY.

Centrifugal casting. H. SIMON (Z. Metallk., 1929, 21, 302—304).—Some examples are given of the value of the centrifugal casting process in producing high-

strength castings of relatively small size. For example, sand castings of red brass with 84.8% Cu, 5.1% Sn, 3.9% Pb, and 6.2% Zn have tensile strength 12 kg./mm.² and elongation 9%; centrifugal castings of the same alloy have tensile strength 35 kg./mm.², elongation 27%. The corresponding figures for aluminium are: (a) sand-cast, 13 kg./mm.², 10%; (b) centrifugal cast, 21 kg./mm.², 18%; for copper: chill-cast, 25 kg./mm.², 30%; (b) 32 kg./mm.², 32%; and for 63:37-brass: (a) 32 kg./mm.², 24%; (b) 46 kg./mm.², 34%.

A. R. POWELL.

Casting texture of metals and alloys. F. C. NIX and E. SCHMID (Z. Metallk., 1929, 21, 286—292).—From X-ray examination of the casting structure of various metals, it is shown that, except in the case of the hexagonal metals zinc and cadmium, most metals have a fibre structure with a simple crystallographic direction parallel to the longitudinal axis of the radial crystals which spread inwards from the cooling surface. In metals with a cubic structure this direction is the (100), in tetragonal metals the (110), and in rhombohedral metals the (111). In hexagonal metals the basal plane is parallel to the axis of the radial crystals and the metals have a ring-fibrous structure. In the aluminium-silicon eutectic, only the aluminium has the characteristic casting structure, the silicon being irregularly oriented, but in the cadmium-zinc eutectic both metals have their characteristic casting structure.

A. R. POWELL.

Segregation in certain non-ferrous alloys. R. LAW (J. Soc. Chem. Ind. Victoria, 1927, 27, 1414—1431).—The only alloy of copper and silver which is homogeneous after cooling is the eutectic mixture containing 71.8% Ag. When the silver content is greater, more copper is found at the exterior of the ingot than the known average content, and if the silver content is less than 71.8%, the surfaces are found to be enriched in silver. Examples of segregation of gold and lead and of gold and arsenic are given, and the present theories of segregation are summarised.

C. A. KING.

Coke-oven gas in industry. EVANS. Gas producers for metallurgical furnaces. ATKINSON.—See II. Physico-chemical properties of fatty acids. LEDERER.—See XII.

See also A., Dec., 1929, Vanadium-carbon system (OYA and OSAWA). Thermomagnetic analysis of oxides and minerals (HUGGETT). 1370, Effect of iron on magnetic susceptibility of aluminium (MASON). 1373, Volume changes during solidification of metals and alloys (GOODRICH). 1374, Eutectic and eutectoid alloys in binary systems (SALDAU). 1375, Solubility of copper in ferro-nickels (CHEVENARD). 1402, Separation of chromium from solutions (LIEBREICH and DUFFEK). Electrodeposition of molybdenum (MERRILL and RUSSELL). Electrodeposition of tungsten (JACKSON and others). Electrolysis of molten ternary alloys (KREMANN and others). 1408, Preparation of rhenium (NODDACK and NODDACK). 1413, Potentiometric determination of chromate (MAYR and BURGER). 1414, Volumetric determination of manganese (KOLTHOFF and SANDELL). 1416, Determination of lead (HOLMES and MORGAN).

PATENTS.

[Open-hearth] furnace. A. SIMONET (U.S.P. 1,733,829, 29.10.29. Appl., 14.2.27).—The furnace is provided with a trough embedded in the walls at the height of the normal melting level. The bottom of the trough is inclined downwardly to the wall of the furnace through which cooling pipes pass. An auxiliary longitudinal pipe discharges a cooling liquid against the outer walls of the furnace. C. A. KING.

Operating a hearth-type furnace [for lead ores]. H. R. MACMICHAEL, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,730,582, 8.10.29. Appl., 3.6.27).—The charge of ore and fuel is fed into a reverberatory furnace with a well-shaped hearth terminating in an inclined portion on which a mechanical shovel operates to throw back continuously that portion of the charge which rolls on to it from the back of the well.

A. R. POWELL.

Smelting furnace and method of smelting. R. A. WAGSTAFF, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,729,408, 24.9.29. Appl., 27.6.25).—A reverberatory furnace for matte smelting comprises a shelf composed of fired lumps of chromite on to which the charge is fed against a water-cooled back wall. The flames from an oil- or powdered coal-fired burner play on to the front of the charge in such a manner that the molten material runs forward into a settling chamber indirectly heated by the hot gases in the melting zone.

A. R. POWELL.

Furnaces [for heating billets etc.]. G. and D. M. NAISMITH (U.S.P. 1,732,137—8, 15.10.29. Appl., [A] 12.12.27, [B] 19.12.27).—The furnace described in (A) is regenerative and that in (B) is recuperative. In (A) all the gases pass from one set of regenerators through an immediately adjoining goods chamber, and most pass to the other set of regenerators; some of the gases, however, are taken from the goods chamber, through a preheating chamber (which is in an extension of it), and pass directly to the stack. In (B) both gas and air pass horizontally through the recuperator, mix in a vertical passage, then pass through a lower inlet port (or ports) to the goods chamber, through which they circulate upwardly and leave by an upper outlet port, thence downwardly through the recuperator.

B. M. VENABLES.

[Blast-furnace] tuyère. G. G. CRAWFORD (U.S.P. 1,727,337, 10.9.29. Appl., 1.2.26).—A copper casting having a chamber for circulation of a cooling medium is joined to a cover by a fused-metal joint. The walls of such casting are uniformly thick, and their interior surfaces are polished in order to reduce the deposit of sediment.

J. S. G. THOMAS.

Apparatus for treating [iron] ores. C. A. BRACKELSBURG, Assr. to MAGUIRE, INC. (U.S.P. 1,729,534, 24.9.29. Appl., 17.6.27).—The ore mixed with sodium carbonate and a small proportion of sodium hydroxide is formed into small spheres, which are fed into the upper part of a blast-heating device through which is blown a current of hot, compressed air; thus the ore is roasted under pressure and the alumina and chromic oxide contents are converted into sodium aluminate

and chromate. Means are provided for charging and discharging the roasting chamber while maintaining a high pressure therein and for returning the waste gases to a regenerative heating unit.

A. R. POWELL.

Treatment of ores and metallurgical products. H. G. HEINE, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,731,898, 15.10.29. Appl., 23.4.27. Ger., 28.4.26).—The material is heated in a kiln in which zones of uniform reaction temperature are formed by locally reducing the cross-section of the kiln so that the height of the material is varied, e.g., by the introduction of steps.

A. R. POWELL.

Reduction of [iron] ores. B. CHRISTIANSEN (U.S.P. 1,728,784, 17.9.29. Appl., 19.12.28. Swed., 23.12.27).—The ore is passed successively through (A) preheating, (B) pre-reduction, and (C) final reduction zones in a long tunnel kiln. In A the ore is heated by waste gases from C, which then pass through a glowing mass of carbon at 1500° in a carbonising furnace until the temperature falls to 1200°, the carbon monoxide being passed through zone C of the reduction furnace. Steam is passed into the carbonising furnace until the temperature falls to 800°, and the resulting water-gas is used for the pre-reduction in B. Finally, air is blown through the carbonising furnace to reheat it to 1500°. Three carbonising furnaces are used for one reducing kiln, the furnaces being used in such a manner that one is being reheated while the next supplies carbon monoxide and the third water-gas.

A. R. POWELL.

Roasting and reducing [iron] ores. B. CHRISTIANSEN (U.S.P. 1,732,672, 22.10.29. Appl., 6.1.28. Norw., 29.1.27).—The ore is roasted by means of the heat generated by the combustion of the waste gases from the pre-reduction zone of the three-stage furnace described in U.S.P. 1,728,784 (preceding). Both the pre-reduction and complete reduction of the ore are effected by means of producer gas from recarbonising furnaces fed with part of the waste gases from the reducing furnace.

A. R. POWELL.

Puddling of iron. J. B. SCHLOSSBERG, Assr. to AMER. CHAIN Co., INC. (U.S.P. 1,730,044, 1.10.29. Appl., 26.4.27).—Mixed iron and steel scrap in quantity sufficient for several puddling furnaces is melted, samples are analysed from time to time for phosphorus, silicon, manganese, and carbon, and the requisite quantity of these elements is added to the molten metal to give it the desired composition. As required, quantities of iron are withdrawn into a rotary or an oscillating puddling furnace and the stock is made up by addition of more scrap etc., so that a continuous supply of metal of regular composition is available for the puddling furnaces.

A. R. POWELL.

Treatment of moulds. H. C. BUGBIRD, Assr. to S. D. WARREN Co. (U.S.P. 1,730,801, 8.10.29. Appl., 1.11.27).—A mould dressing comprises an unctuous powder prepared by boiling precipitated calcium carbonate with oleic acid until the particles become coated with calcium oleate, filtering, and drying.

A. R. POWELL.

Treatment [quenching] of metals. D. M. TILLER and W. W. FERREE (U.S.P. 1,731,641, 15.10.29. Appl.,

16.8.26).—The tensile strength of copper, aluminium, silver, gold, and steel is claimed to be materially increased by quenching the metal from 250–500° in a solution of sodium chloride and mercuric iodide in dilute acetic acid or in a mixture of fish oil and mercuric chloride.

A. R. POWELL.

Treatment of malleable [iron] castings. I. R. VALENTINE, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,734,928, 5.11.29. Appl., 20.5.26).—The casting is coated successively with copper and with zinc, and aged at a temperature above 100° but below that which would cause the zinc to peel off.

F. G. CROSSE.

Making composite [steel-cast iron] castings. C. M. WALKER, Assr. to R. L. McELROY and J. E. SHEPHERD (U.S.P. 1,729,848, 1.10.29. Appl., 10.11.26).—A steel reinforcing rod is coated electrolytically with a thin deposit of copper, immersed in borax solution, dried, and placed in the mould, which is then filled with cast iron. The copper coating acts as a solder between the two metals and ensures a sound union between steel and cast iron.

A. R. POWELL.

Cast-iron alloys [containing molybdenum]. MOLYBDENUM CORP. OF AMERICA, Assees. of H. L. GREENE and C. TAYLOR (B.P. 289,075, 21.4.28. U.S., 21.4.27).—The alloys contain 1–4% Ni, 0–1% Mo, 0–2.5% Si, and 2–4% C. The preferred composition is 1.5% Ni, 0.25% Mo, 2.5% Si, and 2–4% C. [Stat. ref.]

A. R. POWELL.

[Chromium] alloy steel. F. F. McINTOSH, Assr. to CRUCIBLE STEEL CO. OF AMERICA (U.S.P. 1,730,780, 8.10.29. Appl., 12.1.28).—The steel contains 0.1–1.0% C, 8–18% Cr, 0.9–1.3% Mn, 0.08–0.15% P, and 0.05–0.15% S.

A. R. POWELL.

Removal of foreign substances from metal [steel] surfaces. C. F. DIXLEY (U.S.P. 1,729,765–7, 1.10.29. Appl., [a] 11.2.25, [b] 14.8.26, and [c] 30.11.27. Renewed [b] 7.12.27).—(a) Rust and oil are removed from steel surfaces by spraying them with a mixture of a solvent for the rust, an organic solvent for grease, an absorbent mineral powder, and iron phosphate to act as a suspending agent to keep the mass in the form of a smooth paste. The paste is allowed to dry on the surface and then brushed off. The mixture comprises (b) phosphoric acid 120 g., methyl ethyl ketone 81 g., raw sienna 135 g., powdered charcoal 45 g., and water 230 g.; or (c) ferric phosphate 32 g., 75% phosphoric acid 45 c.c., fuller's earth 125 g., kaolin 75 g., alcohol 250 c.c., and water 250 c.c.

A. R. POWELL.

Production of a hard surface on [cutting] tools etc. F. L. SCOTT, Assr. to HUGHES TOOL CO. (U.S.P. 1,733,241, 29.10.29. Appl., 6.8.23).—Particles of tungsten carbide are sintered on to the cutting edges of the tool.

C. A. KING.

Case-hardening process. G. W. SCHWAB and F. J. SCHWEIZER, JUN. (U.S.P. 1,730,247, 1.10.29. Appl., 12.1.26).—Hydrocarbon gas is passed through a cracking chamber provided with baffles to collect the carbon deposited, and the issuing hot gases are passed into the carbonising vessel over a nickel-iron alloy, which is claimed to act as a catalyst in the case-hardening operation.

A. R. POWELL.

Manufacture of finely-divided metals [iron]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,336, 19.11.28).—Ferric chloride vapour mixed with hydrogen in large excess is passed downwards through a vertical tube, heated at 1000° by means of a stream of nitrogen, in such a manner that the ferric chloride is reduced to iron in the central part of the tube without coming into contact with the walls. The ratio $H_2 : HCl$ in the issuing gases should exceed 10 : 1.

A. R. POWELL.

Recovery of copper from ores etc. H. LAVERS and B. TAPLIN (B.P. 321,213, 2.8.28).—The coarsely ground ore is heated to redness in the presence of a small quantity of a carbonaceous material and a chloridising agent, e.g., sodium chloride, and the cooled mass is subjected without further grinding to a flotation treatment. The process is especially suitable for the concentration of mixed oxide-sulphide ores.

A. R. POWELL.

Winning of metals from metal-bearing materials. R. RODRIAN (U.S.P. 1,728,735, 17.9.29. Appl., 22.6.27).—The finely-ground ore is treated with a hot, concentrated, alkaline sulphide solution which dissolves any sulpho-acids and converts the sulpho-bases into finely-divided sulphides which remain in suspension. The thick suspension is separated from the sediment of non-metallic substances and diluted to allow the suspended sulphides to settle; the clear liquor is electrolysed for the recovery of soluble metals and the sulphide precipitate is smelted.

A. R. POWELL.

Recovery of tin and similar functioning metals from materials containing them. H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,729,196, 24.9.29. Appl., 20.4.27).—Slag containing tin is blown in a converter with a blast containing sufficient carbonaceous material to maintain the temperature and small quantities of powdered sodium or calcium chloride, whereby the tin is volatilised as stannous chloride. Addition of lime to the slag assists in the liberation of the tin.

A. R. POWELL.

Removal of certain constituents from metal-bearing materials. H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,730,548, 8.10.29. Appl., 20.4.27. Cf. U.S.P. 1,729,196, preceding).—Copper, lead, zinc, silver, and gold are recovered from molten materials containing them by blowing the material in a converter with a blast containing powdered fuel and a halidising agent, e.g., sodium chloride, whereby the metal is volatilised as the corresponding halide.

A. R. POWELL.

Extraction of copper-nickel matte. W. J. HARSHAW and G. L. HOMER, Assrs. to HARSHAW CHEM. CO. (U.S.P. 1,729,423, 24.9.29. Appl., 28.4.26).—Bessemerised nickel-copper matte is ground to about 10-mesh and boiled with dilute sulphuric acid until no more nickel dissolves. The residue is dried, roasted at 300–500°, and leached first with very dilute sulphuric acid to dissolve copper sulphate and then with more concentrated acid to dissolve nickel sulphate and more of the undecomposed nickel sulphide. The process is repeated until no further copper and nickel are extracted.

A. R. POWELL.

Freeing copper from copper oxide. H. S. LUKENS and R. P. HEUER (U.S.P. 1,730,775, 8.10.29. Appl., 29.12.24).—The copper is melted in an electric furnace under a layer of slag which acts as a solvent for the copper oxide. As the slag becomes saturated with copper oxide it is removed, treated with a reducing agent in a second furnace to recover the copper, and returned to the first furnace for use again. A. R. POWELL.

Treatment of ores etc. containing acid oxide-forming metals, e.g., chromium, manganese, molybdenum, titanium, uranium, for the formation of salts thereof and useful by-products. R. W. STIMSON (B.P. 320,845, 21.4.28).—The ore is roasted at 700° with an alkali nitrate under about 2 atm. pressure, the nitric oxide evolved is cooled and mixed with air to form nitrogen peroxide, and this is passed into a second reaction vessel containing further quantities of ore and a base forming a soluble compound with the acidic oxide of the ore. The roasted products are leached with water and the solution is evaporated for the recovery of the soluble salts; the iron oxide residue from leaching is in a porous form suitable for smelting in an iron blast furnace after briquetting. A. R. POWELL.

Treating ores [by chloridising roasting]. R. F. MEYER, ASSR. to MEYER MINERAL SEPARATION CO. (U.S.P. 1,730,584, 8.10.29. Appl., 16.4.26).—Sulphide ores are roasted, at a temperature below that at which the sulphate of the contained metal decomposes, in a furnace provided with means for advancing the charge through its entire length. During its progress sodium chloride is added in small portions and the charge is subjected at 450° alternately to the action of hot moist air and of air, steam, and sulphur dioxide. The product is subsequently leached for the recovery of the valuable metal as chloride and sulphate. A. R. POWELL.

Treatment of arsenious ore. A. R. LINDELAH (U.S.P. 1,729,351, 24.9.29. Appl., 2.12.27. Swed., 7.7.27).—Arsenic trioxide obtained by roasting ores containing arsenic is converted into calcium arsenate by roasting with lime in excess, and the product is then smelted with a basic slag which converts the arsenic into an insoluble compound. A. R. POWELL.

Flotation process [for oxidised lead-silver ore]. H. R. WILSON (U.S.P. 1,733,570, 29.10.29. Appl., 3.4.28).—Oxidised ores containing silver associated with lead and the mineral argentorjarosite are floated first with amyl xanthate to obtain a rich silver concentrate, then with amyl xanthate and sodium sulphide to recover the remainder of the silver together with the lead. A. R. POWELL.

Manufacture of bearing metals. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 308,983, 2.4.29. Ger., 2.4.28).—Bearing metal capable of absorbing lubricant is produced by sintering into a porous mass metal, such as iron or steel, in the form of small particles or powder. F. G. CROSSE.

[Non-corrodible copper] alloys. BARRER ASPHALT CO., ASSEES. of A. B. DAVIS (B.P. 294,233, 30.5.28. U.S., 21.7.27).—The alloy comprises 80–90% Cu, 2–10% Ni, and 1–8% Sn, together with silicon in an amount

sufficient to saturate the alloy. The toughness and hardness are increased by the addition of 0.1–2.0% Cr and/or 0.2–5% Mn. Sharp castings are produced by adding 0.5–2% Sb. A. R. POWELL.

Apparatus for refining metals [lead]. H. W. YORK, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,733,522, 29.10.29. Appl., 9.2.27).—In the apparatus described in U.S.P. 1,706,722 (B., 1929, 687) the revolving table on which the reaction between the sulphur and the lead takes place is replaced by a series of sloping baffles arranged in a vertical direction alternately on either side of the reaction vessel. A. R. POWELL.

Refining of lead. J. P. WALKER (U.S.P. 1,731,365, 15.10.29. Appl., 25.8.28).—Molten lead is agitated with dried slaked lime and air whereby zinc, arsenic, and antimony are rapidly oxidised. A. R. POWELL.

Cleaning [tinned] metal surface. H. ADLER, ASSR. to DIVERSEY MANUF. CO. (U.S.P. 1,734,706, 5.11.29. Appl., 29.3.26).—The surfaces are treated with a solution of trisodium phosphate in the presence of sodium dichromate; corrosion is thereby inhibited. H. ROYAL-DAWSON.

Manufacture of metallic substances with a fibrous structure. KAISER-WILHELM-INST. F. EISENFORSCHUNG (B.P. 296,986, 27.8.28. Ger., 10.9.27).—Wood, bone, asbestos, etc. is treated in any suitable manner for the extraction of non-fibrous substance, heated at 240°, and immersed in a bath of molten tin under 50 atm. pressure for a few seconds. The product is capable of taking a high polish, may be split and grained, and does not bend or kink in thin sections. A. R. POWELL.

Extrusion of metal. BRIT. INSULATED CABLES, LTD., and J. G. LAUDER (B.P. 320,928, 24.7.28).—Metal rod, tube, or wire is coated with aluminium or an analogous metal by passing the wire etc. through aluminium in a plastic condition under pressure and extruding the plastic metal around the wire through a die. The wire is protected from contact with the aluminium until almost entering the die, by means of a straight tubular sheathing passing through the wall of the metal container and extending nearly to the die aperture. C. A. KING.

Manufacture of chromium articles. ELECTRICAL RESEARCH PRODUCTS, INC., ASSEES. of S. R. MASON (B.P. 309,539, 28.3.29. U.S., 12.4.28).—Tubular articles or dies for drawing wire are made by depositing chromium on a core which is afterwards removed. The core may be of a highly polished metal of low m.p. C. A. KING.

Storage of alkali and other readily oxidisable metals. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of D. H. BROPHY and W. A. RUGGLES (B.P. 296,347, 22.8.28. U.S., 29.8.27).—Cesium is obtained by distillation of a mixture of cesium chloride and calcium in *vacuo* in a nickel container heated in an induction furnace. The distillate is collected in a glass bulb and transferred to a narrow tube-still in a vacuum. The still is filled with a dry hydrocarbon oil and the vacuum broken. The metal is melted below the oil and transferred to numerous small capillary tubes by placing the capillary in a closed tube below the surface of the molten metal, breaking the end of the outer

tube, and drawing the metal into the capillary by suction. The latter is removed from the liquid and sealed.

A. R. POWELL.

Reclamation of scrap metals. T. D. STAY, Assr. to ALUMINUM Co. OF AMERICA (U.S.P. 1,729,631, 1.10.29. Appl., 28.10.21. Renewed 24.3.27).—The metal, *e.g.*, aluminium or one of its alloys, is treated with a magnet to remove iron, then fed centrally into a bath of similar metal melted in a kettle provided with a stirring gear which produces upward and downward currents in the metal bath. The dross is removed, crushed, and treated on shaking screens to remove metal particles, which are returned to the main part of the scrap for re-treatment. A. R. POWELL.

Treatment of aluminium or other light metals [to prevent corrosion]. ELECTROLUX, LTD., Asses. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 294,237, 15.6.28. Ger., 22.7.27).—The metal, after anodic treatment in a bath containing a fluoride, *e.g.*, in a sodium fluoride solution containing 1–5% of ammonium carbonate, is submitted to anodic treatment in an electrolyte, *e.g.*, a solution of ammonium carbonate. Subsequently the surface of the metal is cleaned by being used, for a short time, as cathode.

J. S. G. THOMAS.

Producing a coating of chromium on objects of iron, steel, or aluminium, and particularly on knives, fork, and spoons. H. LEISER (B.P. 294,484, 23.7.28. Ger., 21.7.27).—Metal articles are coated with molten tin (which is smoothed by pressing or other means), then with another metal or alloy, *e.g.*, brass, and heated; a final coating of chromium is deposited electrolytically.

C. A. KING.

Electrolytically depositing chromium. Soc. CHIM. DE LA SEINE, and V. SZIDON (B.P. 320,952 and 320,959, 28.7.28).—(A) The electrolyte comprises a 25% solution of chromic acid containing 1–4% of chromic sulphate and 1–2% of sodium carbonate. A current density of 130 amp./ft.² at 4–6 volts is employed. (B) The articles to be plated are heated electrically before immersion in the electrolyte (cf. B.P. 320,440; B., 1929, 1020).

A. R. POWELL.

Production of electrolytically deposited gold in film or leaf form. W. F. GRUPE (U.S.P. 1,731,415, 15.10.29. Appl., 23.2.27).—Gold is electrodeposited on a continuous band or strip of silver, which is then caused to adhere, on the plated side, to a similar strip of paper coated with resin. After dissolving the silver in dilute nitric acid the gold-coated paper may be used as a transfer, the paper being readily removed by warming until the resin melts.

A. R. POWELL.

Blast furnace. Y. MURAKAMI (U.S.P. 1,735,293, 12.11.29. Appl., 14.9.26. Jap., 28.9.25).—See F.P. 621,588; B., 1928, 127.

Mechanical roasting furnace. G. BALZ, Assr. to BALZ-ERZRÖSTUNG GES.M.B.H. (U.S.P. 1,734,552, 5.11.29. Appl., 7.6.27. Ger., 22.2.27).—See B.P. 288,823; B., 1928, 451.

[Manufacture of] pure iron of small grain size. W. MEISER, W. SCHUBARDT, and O. KRAMER, Assrs. to I. G. FARBEIND. A.-G. (U.S.P. 1,735,405, 12.11.29.

Appl., 28.8.28. Ger., 10.9.27).—See B.P. 306,215; B., 1929, 329.

Steel alloy. A. FRY, Assr. to F. KRUPP A.-G. (U.S.P. 1,735,744, 12.11.29. Appl., 28.7.27. Ger., 23.8.26).—See B.P. 276,317; B., 1928, 451.

Manufacture of ductile bodies from highly refractory metals [tungsten]. J. A. VAN LIEMPT, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,728,814, 17.9.29. Appl., 16.1.25. Holl., 24.1.24).—See B.P. 228,131; B., 1925, 750.

Production of metals and alloys. T. R. HAGLUND (U.S.P. 1,731,976, 15.10.29. Appl., 16.10.26. Swed., 23.10.25).—See B.P. 239,404; B., 1925, 887.

Coating aluminium or aluminium alloys with aluminium oxide. T. KUJIRAI and S. UEKI, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,735,286, 12.11.29. Appl., 2.12.24. Jap., 19.12.23).—See B.P. 226,536; B., 1925, 725.

Rustproofing process. W. H. ALLEN, Assr. to PARKER RUST PROOF Co. (Re-issue 17,484, 12.11.29, of U.S.P. 1,654,716, 3.1.28).—See B., 1928, 235.

[Die] casting molten metal under pressure. R. H. V. CHRISTENSEN (B.P. 306,958, 25.2.29. Denm., 29.2.28).

Covering surfaces of metal pipes with a mixture of fibrous and cement substances, or forming pipes of such a mixture. A. ROCCA (B.P. 321,471, 14.8.28).

Metallised coke (B.P. 296,443).—See II. **Melting furnace** (B.P. 305,952). **Coating aluminium electrolytically** (U.S.P. 1,727,331).—See XI.

XI.—ELECTROTECHNICS.

Effect of drawing on the temperature coefficient of the electrical resistivity of constantan. R. S. J. SPILSBURY (J. Sci. Instr., 1929, 6, 357–358).—The variability of the temperature coefficient of resistance of commercial constantan wire has been traced to the effect of drawing. The temperature coefficient becomes more negative with progressive drawing.

C. W. GIBBY.

Low-temperature carbonisation and electricity production. SMYTHE and WEEKS.—See II. **Zinc.** EGER. **Cadmium.** WATANABE and TSUCHIMOTO. **Aluminium.** SETOH and MIYATA.—See X.

See also A., Dec., 1392, **Weston normal standard cell** (SUMMERS and GARDINER; GARDINER and HULETT). **Preparing standard cells** (GARDINER and HULETT). 1402, **Electrolytic production of fluorine** (FREDENHAGEN and KREFFT). **Separation of chromium from solution** (LIEBREICH and DUFFEK). **Electrodeposition of molybdenum** (MERRILL and RUSSELL). **Electrodeposition of tungsten** (JACKSON and others). **Electrolysis of molten ternary alloys** (KREMANN and others). 1403, **Electrochemical behaviour of gold and behaviour in hydrochloric acid solutions** (GRUBE and others). **Chlorination of benzene** (JAYLES). 1414, **Colorimetric reactions determined electrochemically** (FRITZ). 1415, **Thermo-electric temperature scales** (ROESER). **Thermostat** (HUME). 1416, **Regu-**

lation of temperatures (COOKE and SWALLOW). **Electrochemical switchboard** (MUNN; HOLMES and MORGAN). **Valve for p_H determinations** (LJUNGGREN and ELMQVIST). 1417. **Valve for electrometric titrations** (EHRHARDT).

PATENTS.

Electric melting furnace. ELECTRIC FURNACE CO., LTD., Assees. of HIRSCH, KUPFER- & MESSING-WERKE A.-G. (B.P. 305,952, 11.2.29. Ger., 11.2.28).—The charge is heated by induction or by a resistor, these heating means being used either separately or together. Thus the charge is arranged to flow from the induction furnace to the resistance furnace, so that the solid metal is heated by induction, and the liquid metal by the resistance elements. A pyrometer cuts off the inductive heating when the charge begins to melt.

J. S. G. THOMAS.

Electric [induction melting] furnace. V. STOBIE (B.P. 319,802, 31.3.28).—A portion only of the charge angularly surrounds a part of the magnetic circuit of the furnace which passes through an interlinking path composed of magnetic iron. Part of the energy of the furnace is induced in an annular circuit through a path of length and cross-section determined by the shape of the crucible, the remainder of the energy being induced in the charge as eddy currents.

J. S. G. THOMAS.

Protecting carbon electrodes from consumption in the atmosphere. I. G. FARBENIND. A.-G. (B.P. 302,891, 2.11.28. Ger., 23.12.27).—Carbon electrodes for use in furnaces are soaked in or coated with phosphoric acid. [Stat. ref.]

J. S. G. THOMAS.

Electrodes for electric arc-welding or soldering. (A) ALLOY WELDING PROCESSES, LTD., Assees. of LA SOUDURE ELECTRIQUE AUTOGENE SOC. ANON, and (B) ALLOY WELDING PROCESSES, LTD., and E. J. CLARKE (B.P. 307,760, 12.3.29. Fr., 12.3.28, and B.P. 320,596, 13.3.29).—(A) One or more oxyacid salts of the alkali and alkaline-earth metals, *e.g.*, 2–3% of the nitrates, chlorates, bromates, nitrites, chlorites, and corresponding per-salts, are added to the electrode coating to raise the m.p. of the electrode. Thus 3% of potassium nitrate may be added to a coating consisting of calcium carbonate (35%), magnesium carbonate (30%), and siliceous chalk (35%). (B) A powdered alloy of two or more non-ferrous metals is added to the usual fluxing materials and/or slag-forming materials used for coating the electrode in order to form with the metal of the core the desired alloy steel. Thus the coating may contain a powdered alloy of two or more of the metals nickel, chromium, iron, tungsten, molybdenum.

J. S. G. THOMAS.

Electric heating apparatus for heating bituminous or other materials. J. A. REAVELL (B.P. 319,298 and 319,391, [A, B] 19.6.28).—Intercommunicating jackets (one or more in number) surrounding a vessel containing material to be heated are provided with heating elements whereby heat-absorbing fluid contained in the jackets is heated and caused to circulate. In (B) constant circulation of the fluid is maintained by means of a force pump.

J. S. G. THOMAS.

Electrical insulation [material]. I. G. FARBENIND. A.-G. (B.P. 297,749, 26.9.28. Ger., 26.9.27).—Paper or

textile fabric is saturated with a solution of a cellulose ester of a higher fatty acid, other than cellulose oleate, or a mixture of such esters, or an ether-ester of cellulose with a higher fatty acid.

J. S. G. THOMAS.

Electric resistance material. T. A. SWARTZ and W. P. UHLER (U.S.P. 1,734,309, 5.11.29. Appl., 24.9.23).—The material comprises a vulcanised composition consisting of rubber, sulphur, barium sulphate, and finely-divided carbon, the relative proportions of the last two determining the specific resistivity of the product.

F. G. CROSSE.

Manufacture of magnetic cores of high permeability. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,854, 14.7.28).—Metallic powder, *e.g.*, iron, preferably prepared from a metal carbonyl, is impregnated with a condensation product of urea or its derivatives and formaldehyde, dried, and compressed.

J. S. G. THOMAS.

Manufacture of magnetic cores of high permeability. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,611, 14.7.28. Cf. B.P. 319,854; preceding).—Pitch-like or asphaltic materials prepared either in accordance with B.P. 302,710 (B., 1929, 233) or by treating asphalt, tar, pitch, etc. with vegetable or animal oils or fats and natural or artificial resins, at a high temperature with oxygen or gases containing oxygen, are impregnated with a metallic powder, *e.g.*, iron, preferably prepared from the carbonyl. Alternatively, solutions of pitch-like or asphaltic materials, *e.g.*, in benzene, may be so impregnated, dried, and pressed, or coumarone resin heated with pitch, asphalt, etc. and solid absorbing materials may be so treated.

J. S. G. THOMAS.

Solution for chemical rectifiers. F. T. BOWDITCH, ASSR. to NAT. CARBON CO., INC. (U.S.P. 1,727,462, 10.9.29. Appl., 21.6.24).—A solution containing citrate and phosphate radicals in the ratio 3–15:1 is employed. Thus a solution containing about 42.5% of ammonium citrate and about 7.5% of ammonium phosphate may be used.

J. S. G. THOMAS.

Polarising electrolyte [for rectifiers or condensers]. P. E. EDELMAN, ASSR. to E. BANNING (U.S.P. 1,730,725, 8.10.29. Appl., 19.3.28).—A syrupy solution of gum arabic containing a small quantity of borax is used as the electrolyte of an aluminium rectifier.

A. R. POWELL.

Rectifier compound. F. H. MACKENZIE, ASSR. to M. H. LANE (U.S.P. 1,727,656, 10.9.29. Appl., 12.4.26).—A positive (aluminium) and a negative (lead) electrode are immersed in an aqueous solution of lead acetate and ammonium dihydrogen phosphate.

J. S. G. THOMAS.

Electrolytic apparatus [for production of oxygen and hydrogen]. A. E. KNOWLES (B.P. 320,388, 14.4.28).—An electrolyte feed-tank comprises a shallow tank of relatively large horizontal dimensions arranged below two gas-washing chambers, the combined superficial areas of the chambers and of the intervening space being equal to the superficial area of the feed tank. Liquid is drawn off from each chamber into the tank by a U-tube placed in the tank and having limbs of unequal length, the longer limb passing upwards through the bottom of the chamber and being vertically

adjustable to vary the level at which liquid is maintained in the chamber. J. S. G. THOMAS.

Cells for production of gases by electrolytic processes. A. LANE (B.P. 318,734, 21.8.28).—The vertical edges of the flanges of a vertical plate of substantially channel-shaped cross-section are welded to the line of fold of the succeeding similar plate so as to form a bell or funnel for the collection of gases evolved during the electrolysis. J. S. G. THOMAS.

Prevention of excess voltages in electrolytic cells for the electrolysis of water. A. VOGEL (B.P. 292,131, 14.6.28. Ger., 14.6.27).—A coating of colloidal or finely-divided material, *e.g.*, hydroxide or other compound of iron or of other metals of the iron group, is formed upon the cathodes, preferably roughened or covered with wire gauze. J. S. G. THOMAS.

Apparatus for electrolytic and similar treatments. F. T. TAYLOR, Assr. to HANSON-VAN WINKLE-MUNNING Co. (U.S.P. 1,727,736, 10.9.29. Appl., 23.9.25).—Separate workholders are conveyed along parallel paths through their respective separate tank compartments, the conveying mechanism providing a circuit connexion of one polarity for both compartments, whilst separate circuit connexions of the other polarity lead to the respective compartments and produce different potential drops therein. J. S. G. THOMAS.

Secondary electric cells. A. SISMANOPOL (B.P. 296,990, 6.9.28. Roum., 10.9.27).—A positive electrode of lead peroxide and a negative electrode of lead coated with electrodeposited copper, and having less than half as much active surface as the positive, are arranged in a container, the positive electrode being placed above the negative, which extends to the bottom of the container. J. S. G. THOMAS.

Electric storage batteries or accumulators. A. N. HAZLEHURST (B.P. 319,400, 21.6.28).—Positive plates of lead peroxide enveloped by a copper plate and negative plates of copper are interleaved and immersed in an electrolyte composed of sulphuric acid and/or a soluble sulphate (*d* 1.14), the copper electrodes being without contact with air in presence of the electrolyte. The capacity of the positive plates in relation to the volume of electrolyte is such that the latter does not become a supersaturated solution of copper sulphate. J. S. G. THOMAS.

Electrolyte for galvanic elements or [Leclanché] cells. G. HANEKOP and W. SCHMIDT (B.P. 320,526, 15.10.28).—A slightly alkaline, neutral, or weakly acid electrolyte is composed of a large excess of magnesium chloride and magnesium oxychloride to which is added a neutral aluminium salt solution, *e.g.*, the chloride, fluoride, or sulphate of aluminium, or double salts thereof, *e.g.*, aluminium fluochloride. If desired, zinc chloride, alkali chlorides, manganese chloride, or ammonium salts may be added. J. S. G. THOMAS.

Electroplating tank. C. E. JONES, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,734,909, 5.11.29. Appl., 15.10.25).—Current is supplied to the walls of a tank containing electrolyte and an anode composed of metallic particles and a wire-mesh retaining device connected electrically with opposite sides of the tank. J. S. G. THOMAS.

Coating of aluminium electrolytically. C. L.

BEAL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,727,331, 10.9.29. Appl., 17.1.27).—An aluminium surface, after treatment in a dilute aqueous alkaline bath (*e.g.*, hot 5% solution of trisodium phosphate), is used as cathode, under non-oxidising conditions, in an acid, dilute aqueous bath, and is then electroplated.

J. S. G. THOMAS.

Electron discharge device. INTERNAT. GEN. ELECTRIC Co., Inc., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 299,857, 2.11.28. Ger., 2.11.27).—For the introduction of alkaline-earth metals into receiving or transmitting valves for wireless telegraphy etc., a mixture of aluminates or silicates of alkaline-earth metals, preferably coated with paraffin oil, with reducing agents which do not volatilise appreciably at the temperature of reaction is heated within the envelope, subsequent to evacuation of the valve. As reducing agents, aluminium, beryllium, or a rare-earth metal may be used. To moderate the reaction, if necessary, diluting agents suitable for the purpose are described.

J. S. G. THOMAS.

Introducing highly active metals [caesium] into sealed containers [radio valves]. WESTINGHOUSE LAMP Co., Assees. of J. W. MARDEN and E. A. LEDERER (B.P. 296,781, 24.8.28. U.S., 9.9.27).—A mixture of caesium permanganate or dichromate (1 pt.), silicon (3 pts.), and 1% of aluminium is introduced as a fine powder into the valve and, after evacuation, the powder is heated whereby metallic caesium is liberated in an active form which serves as a getter and produces a filament having a high emission. A. R. POWELL.

Rectifier. Electric discharge device. C. G. SMITH, Assr. to RAYTHEON, Inc. (U.S.P. 1,731,944—5, 15.10.29. Appl., [A, B] 22.1.27).—(A) Solid alkali metal is inserted within an anode which almost completely encloses an electron-emitting electrode in an evacuated vessel. (B) Lithium is placed within an isolated chamber formed of electrodes, one of which is hollow and receives the others. J. S. G. THOMAS.

Taking X-ray photographs with a short exposure. A. E. O'DELL, From SIEMENS-REINIGER-VEIFA GES. F. MEDIZIN. TECHN. M.B.H. (B.P. 320,540, 29.10.28).—One or more condensers are suddenly discharged through a tube operating on direct current and having an incandescent cathode, and the primary current is interrupted at the beginning of the condenser discharge by an overload release device in the primary circuit, *e.g.*, by a delay-action relay or fusible cut-out.

J. S. G. THOMAS.

Electric furnace. V. SORREL and L. A. LAFONT, Assrs. to UGINE-Infra (U.S.P. 1,734,536, 5.11.29. Appl., 4.8.28. Fr., 29.9.27).—See B.P. 297,826; B., 1929, 985.

Electron-emitting cathode. G. L. HERTZ, Assr. to N. V. PHILIPS' GLOBELAMPFABR. (U.S.P. 1,735,080, 12.11.29. Appl., 31.12.23. Holl., 12.1.23).—See B.P. 209,730; B., 1924, 822.

Electrodes [with helical core] for electric arc-welding or soldering. ALLOY WELDING PROCESSES, LTD., and E. J. CLARKE (B.P. 319,487, 20.9.28).

Cleaning the electrodes of electrical gas-cleaning apparatus [by jarring]. LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 321,571, 27.11.28).

Electric discharge tubes. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 320,358, 9.7.28).

Heating and mixing apparatus (B.P. 321,006).—See I. Aluminium etc. (B.P. 294,237). Chromium-plating knives etc. (B.P. 294,484). Chromium (B.P. 320,952 and 320,959). Gold leaf etc. (U.S.P. 1,731,415).—See X.

XII.—FATS; OILS; WAXES.

[Detection of adulteration in] cacao butter. W. SCHMANDT (Z. angew. Chem., 1929, 42, 1039—1040).—Differentiation between pure cacao butter obtained by pressing and the lower grades obtained by extraction or from the waste material may be effected by means of ultra-violet light. A sample of the butter (0.5 g.) is dissolved in 20 c.c. of pure light petroleum and examined under a quartz-mercury lamp. If only "press" butter is used, the solution remains dark and appears greyish in colour, but if 1% or more of cacao extract or waste butter is present the solution exhibits bright fluorescence. The presence of butter derived from the waste can be detected by a colorimetric test with acetic acid. Exactly 1 c.c. of the melted butter is added to 1 c.c. of acetic acid (98—99%) in a test-tube, the two layers being carefully heated in a glycerin-bath. At about 100° the liquids are mixed by shaking and the tube is then kept in water at 50° until two layers separate. A yellow or brown coloration of the acetic acid layer proves that "waste" butter is present. The adulteration of "press" butter with other fats can be detected by means of a special immersion refractometer, provided with a prism head, which can be heated. Using the sodium flame as illuminant, measurements at 40° could be duplicated within ± 0.05 of a scale division. The presence of 2% of foreign fat produced a change of more than 0.2 of a division. Figures are given to illustrate the change in refraction produced by 2% and 5% additions of such materials as palm-kernel oil, coconut oil, oleomargarine, and hardened arachis oil.

H. J. DOWDEN.

Cacao butter. SCHELLBACH (Pharm. Ztg., 1929, 74, 875—876).—Five samples of commercial (chocolate factory) cacao bean scrap, containing 30% of husks, were extracted with ether; the resultant fats had iodine values and refractive indices within normal limits. The author considers that the abnormally high values of the refraction found by Bodinus (B., 1929, 608) must have been due to material containing adulterants, to an abnormal proportion of shell fat, or to residual traces of the solvent used for extraction. E. LEWKOWITSCH.

Cacao oil. SCHELLBACH (Pharm. Ztg., 1929, 74, 1022).—The author re-asserts his opinion (cf. preceding abstract) concerning the abnormally high refraction found by Bodinus for extracted cacao butter, and adds that free fatty acids would lower the refraction, but their removal could not increase the value above the normal for neutral fat (cf. B., 1929, 608.)

E. LEWKOWITSCH.

Cacao oil. SCHELLBACH (Pharm. Ztg., 1929, 74, 1037—1038); BODINUS (*ibid.*, 1038).—A discussion (cf. preceding abstracts).

S. COFFEY.

Pulasan [*Nephelium mutabile*] and rambutan [*N. lappaceum*] fats. C. D. V. GEORGI and G. L. TEIK (Malayan Agric. J., 1929, 17, 364—366).—Pulasan and rambutan seeds yielded, respectively, 91.5 and 94.4% of kernels; these contained moisture 16.3, 16.9%, oil 62.7, 29.9%. Both fats are white solids melting at about 38° to pale yellow oils. The fats from pulasan and rambutan seeds respectively had d_{4}^{20} 0.8588, 0.8595; n_D^{20} 1.4570, 1.4587; saponif. value 194.5, 194.9; iodine value (Wijs) 35.2, 42.2; unsaponif. matter 0.3, 0.5%; acid value 0.2, 0.6. The fatty acids had titer value 52.8°, 56.9°; mean mol. wt. 285.3, 291.6; neutralisation value 196.6, 192.4; iodine value (Wijs) 37.2, 44.9. The high figures for the titer and mean mol. wt. of the fatty acids indicate the presence of fatty acids not usually occurring in vegetable fats. The fats are suitable for edible purposes or for hard soaps.

E. LEWKOWITSCH.

Liver oil from a young male shark "pèlerin" *Cetorhinus maximus*, Günner). Biological relations between the cholesterol and squalene. E. ANDRÉ and H. CANAL (Bull. Soc. chim., 1929, [iv], 45, 498—511).—The oil obtained (yield 50%) from the liver had d_4^{15} 0.9105, n_D^{20} 1.4865, $[\alpha]_D$ -6.9°, saponif. value 98.7, iodine value (Hanus) 155.2, being thus lighter and considerably less unsaturated than liver oil obtained from the adult fish. When hydrolysed the oil affords 3.21% of glycerol, 58.5% of fatty acids (m.p. 25—26°, n_D 1.4616, iodine value 111.1, mean mol. wt. 290.7), and 40.5% of unsaponifiable matter, the adult species containing 44—50% of unsaponifiable matter and 52—57% of fatty acids. Fractionation of the fatty acids by the usual methods yielded 15% of arachidic acid, 20% of myristic acid, 50—55% of cetoleic acid (m.p. 32—33°, iodine value 77.6; cf. Toyama, A., 1928, 154), and about 10% of an unsaturated acid, $C_{18}H_{28}O_2$, iodine value 249.8, for which the name *therapeutic acid* is proposed. About 32% of the acids are present as glycerides, the remainder being cholesteryl esters; the absence of free alcohols is shown by the negligible acetyl value of the oil. The unsaponifiable matter, m.p. 106—107°, iodine value 198.6, acetyl value 87.9, crystallised from acetone yields a fraction (22.5% of the original oil), m.p. 143°, $[\alpha]_D$ -35.83°, iodine value 84.6, acetyl value 145.7 (acetate, m.p. 114°), consisting of ordinary cholesterol, and a highly unsaturated cholesterol, the separation of which was incomplete on further fractionation from alcohol, and a liquid fraction d_4^{15} 0.851, n_D^{21} 1.4871, $[\alpha]_D$ +0.87°, iodine value 375, yielding on fractional distillation squalene and about 7% of pristane, $C_{18}H_{38}$ (d^{19} 0.8057, n_D^{21} 1.4500). Preparation and fractionation of the hexahydrobromides of the crude squalene indicated the presence of two hydrocarbons: $C_{30}H_{50}$ (hexahydrobromide, m.p. 109—110°) and $C_{31}H_{52}$ (hexahydrobromide, m.p. 142—143°). Addition of hydrogen bromide is, however, probably incomplete, and the iodine value, 420, of the fraction 230—235°/3 mm., d^{19} 0.8629, n_D^{21} 1.494, indicates the formula $C_{27}H_{44}$ for squalene, from which a genetic relationship with cholesterol can be traced. The highly unsaturated fatty acids which are always present in this and in other liver oils are regarded as the first stage of the physiological trans-

formation of saturated or slightly unsaturated acids into cholesterol. The low squalene content (18%) and high content of cholesterol, including a cholesterol with several double linkings, of the liver oil derived from the young pèlerin as compared with the high squalene (48%) and low cholesterol content (2%) of the liver oil derived from the adult species suggests that the transformation: fatty acids \rightarrow cholesterol \rightarrow squalene progresses with the growth of the animal. The same physiological transformation proceeding at ordinary temperatures and the presence in these marine animal liver oils of optically active alcohols in association with hydrocarbons lend further support to the marine-animal theory of the origin of petroleum.

R. BRIGHTMAN.

Marine animal oils. Oil of *Centrophorus granulosus*, Müller and Henlé. Comparative study of unsaponifiable matter taken from the egg, the foetus liver, and the liver of adult animals. E. ANDRÉ and H. CANAL (Bull. Soc. chim., 1929, [iv], 45, 511—524).—The yield of oil obtained from the eggs (29.6%), the foetus liver (56%), and the liver of adult *Centrophorus granulosus* (78—89%) increases with the maturity of the animal, the yields of unsaponifiable matter increasing in the same order. Thus the dark-coloured oil obtained from the eggs, d_{20}^{25} 0.9060, n_D^{20} 1.4880, iodine value (Hanus) 293.8, saponif. value 79.1, acetyl value 16.5, yields 3.9% of glycerol and 45.5% of fatty acids (iodine value 187.2, containing a large proportion of highly unsaturated acids of the clupanodonic group), and 55.1% of unsaponifiable matter. The last-named consists of 8.5—9% of at least two alcohols of the cholesterol group, one of which contains two or more ethylenic linkings, and 91% of squalene. Oil from the foetus liver, d_{20}^{25} 0.8857, n_D^{20} 1.4896, $[\alpha]_D^{20}$ —2.23°, saponif. value 66.5°, iodine value 298.4, acetyl value 28.8, contains 2.35% of glycerol, 32% of fatty acids of lower mol. wt. and less unsaturated (iodine value 178.8) than those present in oil from the eggs, and 66% of unsaponifiable matter, consisting of 5.7% of cholesterol, small amounts of batyl and selachyl alcohols (Toyama, A., 1924, i, 604), and 90—94% of squalene. Liver oil from a young immature female animal contained 84% of unsaponifiable matter, 1.6% of solid alcohols (cholesterol), and 15.8% of fatty acids (iodine value 121), the corresponding figures for oil from the fully-grown male or pregnant female being 90—93.6%, 0.8—1.0%, and 8.0—8.7%, respectively. The conversion from the clupanodonic glycerides into the unsaturated hydrocarbons (squalene) does not therefore appear to be direct, the cholesterol forming an intermediate phase as indicated by a study of the pèlerin liver oils (cf. preceding abstract). It is concluded that in passing from the eggs, through the foetus, and the young, to the adult *Centrophorus granulosus*, there is a progressive biological transformation: clupanodonic glycerides \rightarrow cholesterol \rightarrow hydrocarbons, which is reversed in passing from the liver of the pregnant female to the egg.

R. BRIGHTMAN.

Extraction of bones with the vapour of solvents. E. SCHLENKER (Chem.-Ztg., 53, 838—839).—For the extraction of fat etc. from bones in a cyclic process, four extraction vessels are required for each series of

operations and each series is divided into four 3-hr. period parts, viz., two for treatment with the vapour of the solvent, one for the final extraction with the boiling solvent, and one in which the solvent is drained away, the extracted bones are removed, and a fresh charge is put in. At any one time there is always one vessel at each stage of the process, and the vapour evolved from the final boiling is used for the first and second vapour-extraction stages.

A. R. POWELL.

Fatty acids of filter-press cake from spent soap lye. B. W. HOWK and C. S. MARVEL (Ind. Eng. Chem., 1929, 21, 1137—1138).—The filter-press cake, consisting of the iron salts of fatty acids, was acidified and esterified, and the mixed esters were fractionated under reduced pressure. In this way octoic, decoic, lauric, myristic, and palmitic acids were detected, the identification being completed by formation of the *p*-bromophenacyl esters.

F. R. ENNOS.

[Toilet] soap bases. J. DAVIDSOHN (Chem. Umschau, 1929, 36, 349—350).—German manufacturing practice is discussed; addition of coconut oil and rosin and the use of potash are considered, together with the specifications of other countries. The necessity for the absence of unsaponified fat is emphasised. E. LEWKOWITSCH.

Physico-chemical properties of fatty acids. E. L. LEDERER (Z. angew. Chem., 1929, 42, 1033—1035).—The corrosive action of fatty acids on metals has been studied by suspending sheets of copper and aluminium for 1024 hrs. under 12 atm. pressure in an autoclave during the refining of arachis oil. The corrodibility of copper and aluminium as shown by the loss in weight of the plates was 0.009 and 0.042 g./m.²/hr., respectively; the corrodibility of nickel at 192° in stearic acid (m.p. 56°) was 0.62 g./m.²/hr. The copper content of a commercial fatty acid would be about 0.00004%. The influence of light on the colour of fats during storage has been studied on a 2:1 mixture of stearic and oleic acids. Samples of the pure mixture and with additions of iron, nickel, copper, manganese, lead, zinc, and aluminium at concentrations of 0.00004, 0.0004, and 0.04% were exposed (a) in the dark, (b) to direct sunlight for 8 hrs., and (c) to ultra-violet light for 2 hrs. Manganese, aluminium, zinc, and lead produced no change at any of the concentrations by either method of exposure. In the sample containing 0.04% Ni a slight yellowing was caused by the sunlight, but not by the ultra-violet irradiation. The samples containing 0.04% Cu exhibited distinct yellowing, and those containing iron a considerable darkening, the effect being more pronounced in sunlight than in ultra-violet light. The samples containing no metallic adulteration were scarcely affected, and it therefore appears that the discoloration of fats after manufacture is due not to the action of light, but to traces of iron and copper. The conductivity of a number of fatty acids at elevated temperatures has also been determined, and values are given for decoic, myristic, palmitic, stearic, and oleic acids at temperatures between 100° and 200°. It was found that, over this range, the relationship between temperature and conductivity may be expressed by: $\log \lambda = -a + bt - ct^2$, wherein the value of *a* varies with the acid, and the values of *b* and *c* for the

acids investigated approximate to 0.028 and 0.000042, respectively. Although valid only for the range 100–200°, the formula has been used to calculate the conductivities of the acids at their m.p. and b.p.

H. J. DOWDEN.

Dependence of drying power of linseed oil on origin of seed. F. FRITZ (Farben-Ztg., 1929, 35, 287–288).—Previously published iodine values of linseed oils from various sources are recapitulated. Whilst the general impression is given that oil from seed grown in cold climates, e.g., Baltic seed, has a higher iodine value than oil from, e.g., La Plata and India, it is pointed out that other factors than climate must exercise influence, since Indian oil normally has higher drying power than South American oil.

S. S. WOOLF.

Drying of [fatty] oils. A. EIBNER (Farben-Ztg., 1929, 35, 285–287, 336–337).—Mainly a commentary on the work of Morrell and Marks (B., 1929, 860). The results obtained by earlier workers are discussed and the position in the various phases of the drying problem is summarised. It is considered that knowledge of "oxyns" has been very definitely advanced by the work of Morrell and Marks from the analytical aspect and of Staudinger from the constitutional viewpoint.

S. S. WOOLF.

Auer's gas coagulation theory. Drying process of drying oils. H. SCHMALFUSS and H. WERNER (Kolloid-Z., 1929, 49, 323–328).—Objections are raised to the gas coagulation theory of Auer (B., 1927, 145). The experimental work on which the theory is founded has been repeated and entirely different results have been obtained. In an atmosphere of air, drying oils do not increase in weight to an amount greater than that of the oxygen present, the oils do not dry in an atmosphere of nitrogen which is free from oxygen, and the pressure of air does not increase but decreases during the drying process.

E. S. HEDGES.

Gas coagulation theory and the control experiments of SchmalFUSS and Werner. L. AUER (Kolloid-Z., 1929, 49, 328–332).—A reply to SchmalFUSS and Werner (preceding abstract). The gas coagulation theory is maintained.

E. S. HEDGES.

Ratfish-liver oil as a source of vitamin-A. E. R. NORRIS and I. S. DANIELSON (Ind. Eng. Chem., 1929, 21, 1078).—The vitamin-A content of a commercial sample of the liver oil of the ratfish (*Chimara colliet*) was determined by the method of Sherman and Munsell (B., 1925, 609) by feeding experiments on albino rats. The amount of oil required daily for a total gain in weight of 25 g. in 8 weeks (one U.S.P. unit) is calculated to be 0.03 g.

F. R. ENNOS.

Oiticica oil. F. WILBORN and A. LÖWA (Farben-Ztg., 1929, 35, 388–389).—Previously published literature (B., 1919, 645 A) on the oil is summarised. A sample of oil extracted from Pernambuco oiticica nuts had saponif. value 186.3, acid value 3.0, iodine value 178 (Hanus), 152 (Wijs), n_D^{20} 1.5094. The oil dries with a similar appearance to tung oil and gelatinises on addition of a chloroform solution of iodine. Crystals separate on storage. The fatty acids isolated from these were examined by lead-soap methods, but gave

inconclusive results. It would appear, however, that an isomeride of β -elæostearic acid (and not, as was at first thought, the acid itself) is present. The marked tendency of the oil to change to solid products might render it unsuitable for use as a varnish raw material, but it is considered likely that "working up" with rosin etc. would overcome this difficulty.

S. S. WOOLF.

Saponification value. ANON. (Chem. Umschau, 1929, 36, 353).—It is most convenient for makers of both hard and soft soaps to express the saponif. value as the number of c.c. of *N*-alkali required to saponify 100 g. of fat ("normal saponif. value").

E. LEWKOWITSCH.

Report [on butter]. HARRISON.—See XVI.

See also A., Dec., 1396, **Saponification of oils, fats, waxes, and pure triglycerides** (McBAIN and others). 1424, **Sulphonated oils** (NISHIZAWA and SINOZAKI). 1490, **Fat-decomposing enzyme** (GYOTOKU and MATSUBARA).

PATENTS.

Extraction of oils from fish livers. MANUF. DE MACHINES AUXILIAIRES POUR L'ELECTRICITE ET L'INDUSTRIE (B.P. 314,505, 27.7.28. Fr., 29.6.28).—Extraction of the oil by grinding with water followed by centrifugal separation is facilitated by suitable treatment causing the liver cells to burst, e.g., by preliminary freezing, sudden rise in temperature (addition of hot water), fermentation, or by osmotic action induced by adding to the minced livers either salt water, with a salt concentration differing from that of the liver cells, or water as soft as, or softer than, sea-water.

E. LEWKOWITSCH.

Manufacture of sulphonated oils, fats, fatty acids, and waxes. W. SECK, Assr. to ERBA A.-G. (U.S.P. 1,734,050, 5.11.29. Appl., 3.2.28. Ger., 28.7.27).—See B.P. 294,621; B., 1928, 825.

Emulsifying fats etc. (B.P. 321,130).—See I. **Emulsions from isocolloids** (B.P. 318,562).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Bleeding" of [red] pigments. H. RASQUIN (Farben-Ztg., 1929, 35, 386–387).—The compositions of a number of red coal-tar dyes and their relative solubilities in linseed oil, mineral oil, turpentine, white spirit, and benzene are tabulated. "Bleeding" is bound up with solubility, and it is shown that the structure of the dye is an important factor in this connexion, the bleeding tendency increasing with basic character; thus, Para-red and Autol-red, from which acidic groups are entirely absent, are most prone to this defect, whilst Helio fast-red, of similar structure except for a *m*-substituted nitro-group conferring acidic properties, is on the border-line. Heliobordeaux BL, Litholrubin G, and other acidic dyes are free from bleeding. Faulty diagnosis of bleeding is sometimes attributable to "grinning" through a single covering coat of insufficient opacity or to insufficient drying of the red coat. In view of the difficulties associated with bleeding reds, it is suggested that paints containing them be applied in thin coats and allowed a lengthier

drying period than the normal. If this is not practicable, the use of an intermediate coat of spirit varnish is recommended. It would be desirable to dispense entirely with the use of other than "oil-fast" reds, but this is not at present feasible from economic considerations.

S. S. WOOLF.

Composition of turpentines from *Pinus sylvestris*. V. KRSTINSKI and F. SOLODKI (J. Appl. Chem., Russia, 1929, 2, 337—351).—Turpentine distilled from wood chips in presence of alkali contained α -pinene 43, Δ^3 -carene 38.5, terpinolene 6.5, alcohols ($C_{10}H_{17}\cdot OH$) 7, sesquiterpenes (probably cadinene) 3.5, residue 1.5%. Turpentine distilled in absence of alkalis contained α -pinene 42.5, Δ^3 -carene 32.5, terpinolene 4, alcohols 14, sesquiterpenes 3, residue 4%. CHEMICAL ABSTRACTS.

Colorimetric determination of turpentine in air. P. ANDREJEV and A. GAVRILOV (Chem.-Ztg., 1929, 53, 870—871, 889—891).—The air is drawn through 20 c.c. of concentrated sulphuric acid at the rate of 1.5—2 litres/min. and the colour of the resulting yellow to orange solution is compared with that of a standard solution of turpentine in sulphuric acid or with that of a solution made by adding to 93 c.c. of water 7 c.c. of a solution containing 200 mg. of diamine-yellow and 2.8 mg. of methyl-orange in 1 litre of water. This 100 c.c. of solution has the same colour as 100 c.c. of sulphuric acid containing 2.14 mg. of turpentine. A. R. POWELL.

Oiticica oil. WILBORN and LÖWA.—See XII.

See also A., Dec., 1456, **Fungi resins** (FRÖSCHL and ZELLNER). 1499, **Natural resins and resin acids** (BALÁS).

PATENTS.

Production of [non-lifting] lacquers. G. TRÜMPER (B.P. 294,261, 20.7.28. Ger., 21.7.27).—Non-lifting lacquers are obtained by adding water-miscible diluents, e.g., ethyl alcohol or other liquid boiling below 100°, to a preferably highly concentrated solution of one or more cellulose esters in water-miscible solvents (b.p. 125—200°), with which solution natural and/or artificial resins and plasticisers have been incorporated. The weight of non-solvent should be $2\frac{1}{2}$ — $3\frac{1}{2}$ times that of solvent, and the former should have higher vapour tension than the latter in order to favour gelatinisation of the lacquer. The lacquers so obtained can be rendered sprayable by the addition of hydrocarbons etc. having vapour tension above that of the solvents and below that of the non-solvents.

S. S. WOOLF.

Production of coloured material and coating [cellulose lacquer]. C. E. FAWKES, Assr. to QUAKER OATS Co. (U.S.P. 1,732,124, 15.10.29. Appl., 11.5.25).—The lacquer comprises a solution of cellulose nitrate in a suitable solvent together with a plasticiser, e.g., castor oil, shellac, and up to 20% of furyl alcohol, and an acid reagent, e.g., sulphuric or hydrochloric acid, or a substance which hydrolyses readily to yield an acid. As the lacquer dries the furyl alcohol becomes converted into a jet-black compound and a glossy black lacquer is obtained. (Cf. B.P. 234,862; B., 1925, 642.)

A. R. POWELL.

Pyroxylin [lacquer] composition. R. H. VAN SCHAAK, JUN., Assr. to VAN SCHAAK BROS. CHEM.

WORKS, INC. (U.S.P. 1,734,657, 5.11.29. Appl., 12.9.25).—The lacquer comprises a mixture of pyroxylin, a resin, butyl acetate and alcohol, toluene, and benzyl phthalate.

F. G. CROSSE.

Manufacture of intaglio printing colours. I. G. FARBENIND. A.-G. (B.P. 307,877, 18.2.29. Ger., 15.3.28).—Colouring materials in a sufficiently finely-divided or dissolved state, and, if desired, other substances, are incorporated with solutions of resinous products obtained from organic vinyl esters, or from dibasic acids and polyhydric alcohols, in volatile organic solvents, e.g., aliphatic alcohols containing 1—5 carbon atoms per mol., glycol monoalkyl ethers or ketones, etc.

S. S. WOOLF.

Manufacture of coating for wood, metal, and other surfaces. C. H. HOLMAN and O. W. HOOPPAW (U.S.P. 1,734,130, 5.11.29. Appl., 22.7.26).—The coating consists of magnesia, an aqueous solution of magnesium chloride and sulphate, a volatile oil, and an emulsifier.

H. ROYAL-DAWSON.

Manufacture of shellac substitutes. W. DUX (B.P. 300,956, 19.10.28. Ger., 21.11.27).—Condensation is effected, preferably by the aid of suitable catalysts (e.g., anhydrous aluminium chloride), between the chlorinated derivatives of unsaturated fatty acids and of "resinol" or hydroaromatic acids, or, alternatively, between the hydroxy-derivatives of either and the chlorinated derivatives of the other (e.g., trihydroxystearic acid and chlorinated abietic acid, or chlorinated fatty acids and hexahydrosalicylic acid). The products resemble natural shellac resin in all its chemical properties, solubility, etc.

E. LEWKOWITSCH.

Production of moulded masses from blood. F. HOMBERG and M. LANDECKER, Assr. to AMER. NUPLAX CORP. (U.S.P. 1,732,015—6, 15.10.29. Appl., 20.5.26. Ger., 7.7.25).—(A) Dry blood is moistened with 22% of its weight of a 2% solution of zinc chloride to obtain a uniform, moist, powdery product which is moulded under heat and pressure. (B) The blood is mixed with 5% of zinc oxide and 15% of water to a moist, pulverulent mass and then moulded.

A. R. POWELL.

Manufacture of film-forming solutions [from urea, or its derivatives, and formaldehyde]. K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,734,693, 5.11.29. Appl., 22.6.25. Austr., 31.3.23).—See B.P. 213,567; B., 1925, 681.

Emulsions from isocolloids (B.P. 318,562).—See III. **Titanium compounds** (U.S.P. 1,731,364).—See VII. **Non-splintering glass** (B.P. 321,178 and 321,189).—See VIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See also A., Dec., 1455, **Cryoscopic measurements with caoutchouc** (PUMMERER and others). **Iodine and oxygen values of sol and gel caoutchouc** (PUMMERER and MANN).

PATENTS.

Manufacture of plastic or elastic polymerisation products of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,960, 26.4. and 27.8.28).—Such products are obtained by heating diolefines

emulsified in suitable liquids in the presence of hydrogen peroxide, colloids of vegetable origin (*e.g.*, carrageen moss extract, gum arabic, molasses, linseed meal extract), and other substances favouring polymerisation by influencing surface tension, *e.g.*, higher alcohols and electrolytes, particularly buffer mixtures of definitely favourable p_H value. (Cf. B.P. 318,115; B., 1929, 903.) S. S. WOOLF.

Dispersing rubber in water. W. B. PRATT, ASSR. to DISPERSION PROCESS, INC. (U.S.P. 1,731,725, 15.10.29. Appl., 6.12.24).—A coagulated rubber mass is beaten up with water and a suitable agent, *e.g.*, bentonite, which affords a protective coating for the dispersed particles, and the suspension is treated with such a quantity of a flocculating agent, *e.g.*, aluminium sulphate, as to cause the rubber to set to a plastic mass which when spread in a layer and allowed to dry is tacky, coherent, and irreversible. A. R. POWELL.

Treatment of rubber dispersions. J. MCGAVACK ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,730,518, 8.10.29. Appl., 15.3.27).—A rubber latex of increased mechanical stability is obtained by adding to ordinary latex ammonia and a solution of resin in an alcohol or ketone in which rubber is insoluble but which does not coagulate the latex. A. R. POWELL.

Making reversible concentrates from natural milky juices, *e.g.*, [rubber] latex. P. SCHOLZ (U.S.P. 1,729,522, 24.9.29. Appl., 30.8.28. Ger., 11.7.27).—Rubber latex may be preserved and coagulation during concentration prevented by addition of 0.1–0.5% of salicylic acid together with potassium hydroxide to produce a neutral reaction. A. R. POWELL.

Improving the [ageing] properties of rubber goods. O. Y. IMRAY. FROM SOC. CHEM. IND. IN BASLE (B.P. 318,275, 31.5.28).—Anti-agers for rubber are obtained by condensing cyanuric chloride with 1, 2, or 3 mols. of ammonia or substituted ammonias; or with 1 or 2 mols. of these and 2 or 1 mol. of water, hydrogen sulphide, alcohols, phenols, thiols, or thiophenols. Examples are 2:4:6-trianilino-1:3:5-triazine, 4-amino-2:6-dianilino-1:3:5-triazine, and melamine. C. HOLLINS.

Reclaimed rubber. C. H. CAMPBELL, ASSR. to AMER. GLUE CO. (U.S.P. 1,729,706—7, 1.10.29. Appl., [A, B] 22.11.28).—Rubber scrap during the process of devulcanisation is subjected to the action of cleavage products of (A) collagen, (B) keratin. D. F. TWISS.

Rubber compositions. C. H. CAMPBELL, ASSR. to AMER. GLUE CO. (U.S.P. 1,729,708—9, 1.10.29. Appl., [A] 14.12.27, [B] 22.10.26. Renewed [A] 11.5.29).—(A) Reclaimed rubber is mixed with fission products of ox-blood by an alkaline hydrolytic process which renders the ox-blood and its fission products soluble. (B) The albuminate of a metal is incorporated in rubber before vulcanisation of the latter. D. F. TWISS.

Aqueous rubber dispersion. W. B. PRATT, ASSR. to DISPERSIONS PROCESS, INC. (U.S.P. 1,732,027, 15.10.29. Appl., 13.9.23).—See B.P. 233,370; B., 1925, 558.

Emulsions from isocolloids (B.P. 318,562).—See III.

XV.—LEATHER; GLUE.

PATENTS.

Sticking or cementing of leather [with celluloid solution]. J. GOLDACH (B.P. 321,171, 31.7.28).

[Work-operating tool for] machines for treating hides, skins, leather, etc. TURNER TANNING MACHINERY CO., LTD. FROM TURNER TANNING MACHINERY CO. (B.P. 321,217, 3.8.28).

XVI.—AGRICULTURE.

Report of the Imperial Agricultural Chemist. W. H. HARRISON (Agric. Res. Inst. Pusa Sci. Rep., 1927—1928, 30—39).—(1) Sugar cane fertilised with potash yielded juices which from November to February contained the largest amount of sucrose and the lowest of dextrose; ammonium sulphate gave low sucrose and high dextrose values. Superphosphate gave intermediate results, whilst unfertilised cane gave results inferior to those with potash but superior to those with other fertilisers. (2) For the detection of animal fat in butter, 1 g. of dry fat is dissolved in 3 c.c. of acetic anhydride and 4 c.c. of 93% ethyl alcohol. In presence of animal fat a precipitate is formed when the mixture is kept at 30° for 30 min. The test is effective for cow butter and for buffalo butter from animals not fed on cotton seed. For the latter the test is repeated with 4 c.c. and 3 c.c., respectively. (3) Uncured Indian tobacco leaves contain more nicotine and starch than American leaves, the upper leaves containing more than the lower. Curing has little effect on the nicotine, but markedly decreases the starch. (4) Agitation is not necessary in determination of the p_H of soils with the antimony electrode; in the quiescent liquid, $p_H = 2.04 \pm 0.469$. (5) Soil acidity is due to an insoluble colloidal acid which is associated with surface-active hydrogen and basic cations. The colloidal acid appears to be tribasic.

CHEMICAL ABSTRACTS.

Biochemistry of waterlogged soils. III. Decomposition of carbohydrates with special reference to formation of organic acids. V. SUBRAHMANYAN (J. Agric. Sci., 1929, 19, 627—648; cf. B., 1928, 26).—Percolation experiments on waterlogged Indian and Rothamsted soils indicate that, in the absence of decomposing organic matter, addition of nitrate leads to no loss of nitrogen by denitrification. Addition of 0.06–0.30% of dextrose led to rapid depletion of nitrates and oxygen, but no denitrification, to increase in acidity, bacterial activity, and to carbon dioxide formation. Most of the soluble nitrogen was assimilated by micro-organisms or otherwise converted, and most of the added carbohydrate was transformed into lactic, acetic, and butyric acids. Addition of calcium carbonate to the soils led to a distinct increase in lactic acid formation, but had little effect on the other acids. Organic acids were produced in the soils from a variety of carbohydrates, including glycerol, xylose, arabinose, lævulose, mannitol, sucrose, maltose, lactose, starch, maltodextrin, and cellulose in the form of cotton wool. Lactic acid was the first to be observed and appeared to be formed chiefly by direct splitting of the sugar; it decomposed readily giving acetic and butyric acids.

Some acetic acid was formed by direct oxidation of lactic acid, with pyruvic acid as the intermediate.

E. HOLMES.

Determination of soluble carbohydrates, lactic acid, and volatile fatty acids in soils and biological media. V. SUBRAHMANYAN (J. Agric. Sci., 1929, 19, 649—655).—Details are given of the specialised technique involved in the determination of minute quantities of carbohydrates and fatty acids in waterlogged soils (see preceding abstract).

E. HOLMES.

Composition of soil suspensions of various degrees of dispersion in the steppe, solonetz, and podsolised types of soil. L. P. BELYAKOVA (Leningrad Agric. Inst., Glinka Mem. Vol., 1928, 91—162).—The silica content of suspensions of various degrees of dispersion decreases with increase in the degree of dispersion and has no relation to the silica content of the soil; the other components resemble those of the soil. The humus, hygroscopic moisture, R_2O_3 content, sometimes manganese and magnesia, and loss on ignition increase with the degree of dispersion. The smallest fractions appear to contain free hydroxides and soluble salts. Observations with chernozem, solonetz, and degraded meadow soils are recorded.

CHEMICAL ABSTRACTS.

Determination of exchangeable bases in soils: magnesium, potassium, and total bases. R. WILLIAMS (J. Agric. Sci., 1929, 19, 589—599; cf. B., 1928, 723).—The method of determining exchangeable calcium in carbonate-free soils by leaching with 0.5*N*-acetic acid, already developed, has been extended to the determination of magnesium, potassium, and total bases. The results obtained for total bases differ from those obtained by summation of separate determinations by an amount equivalent to that of the sulphates and chlorides present. It is suggested that the total of exchangeable bases obtained by the present method gives a truer measure of the Hissink S-value than methods in which bases present as sulphates and chlorides are included.

E. HOLMES.

Determination of soil organic matter: wet combustion method. F. HARDY (J. Agric. Sci., 1929, 19, 727—733).—A gasometric method for determination of organic carbon in soil by the use of sulphuric-chromic acid mixtures at 100° is advocated. The method gives concordant but slightly lower values than the Kjeldahl method of Robinson, McLean, and Williams (B., 1929, 487), and has the disadvantage that nitrogen determinations cannot be made simultaneously.

E. HOLMES.

Microdetermination of carbon in soil. M. NICLOUX (Compt. rend., 1929, 189, 768—770).—The air-dried soil sample is ground to pass 120-mesh and diluted by admixture with anhydrous sodium sulphate similarly ground and sieved. The mixture is shaken in a thick flask with some steel balls (diam. about 9 mm.) until homogeneous. The portion utilised for analysis should contain approx. 3 mg. of carbon. The oxidation process is carried out with a mixture of sulphuric acid, potassium iodate, and silver sulphate, followed by the addition of potassium dichromate as previously described (A., 1927, 436).

A. G. POLLARD.

Plastometric studies of soil and clay pastes. B. A. KEEN and G. W. S. BLAIR (J. Agric. Sci., 1929, 19, 684—700).—The pseudo-viscosity of soil and clay pastes and their static rigidity (the energy required to cause the paste just to flow) have been examined. The latter quantity is related to the resistance of the soil to the passage of cultivating implements, the effect of chalk on soil resistance, and the moisture content at which a well-kneaded mass of soil becomes sticky.

E. HOLMES.

Variations in potassium content of lucerne due to stage of growth and soil type, and the relationship of potassium and calcium in plants grown on different soil types. J. F. FONDER (J. Amer. Soc. Agron., 1929, 21, 732—750).—Plants grown on light sandy soils contained most potassium, those grown on very heavy soils were intermediate, and on sandy loam contained least. The stems contained more than the leaves, but the expressed juices from the two portions contained equal amounts. The percentage of potassium decreased with advancing growth period. Repeated cropping did not deplete the soil-potassium to the extent that it controlled the plant-potassium. Little of the potassium in stems and leaves was present in the woody tissue. A physiological balance between potassium and calcium appeared to exist.

CHEMICAL ABSTRACTS.

Artificial manure from straw. R. C. COLLISON and H. J. CONN (New York State Agric. Exp. Stat. Bull. No. 573, 1929, 17 pp.).—The rotting of cereal straw by means of a nitrogenous nutrient medium has been studied under controlled conditions and the suitability of the resultant material as a fertiliser has been tested by measuring the growth of barley, rape, and peas in sand to which known amounts of the rotted straw had been added. A heap of mixed oat and wheat straw was impregnated with a mixture of salts in the following proportions per ton of dry straw: ammonium sulphate 60 lb., ground limestone 50 lb., superphosphate 30 lb., and potassium chloride 25 lb., and the stack allowed to ferment with periodical additions of water. A second heap was built under parallel conditions using the "Adco" material (B.P. 152,387; B., 1920, 827 A) developed by Richards and Hutchinson. In three or four months both piles had rotted completely and resembled decomposed farmyard manure. The vegetative tests showed that the injurious effects of fresh straw had been entirely eliminated and the material produced active growth in proportion to the amount added to the sand. A subsequent experiment indicated that, unless a very much longer fermentation period can be allowed, natural rainfall will not successfully replace the artificial watering, which amounts to about 800 gal. per ton of straw. The economics of the processes are discussed.

H. J. DOWDEN.

Action of "nitrophoska I.G.I." in comparison with fertiliser mixtures of various physiological reactions. H. SIBBE (Landw. Jahrb., 1929, 68, 755—773; Chem. Zentr., 1929, i, 2225).—A study of the effect of various fertilisers on the p_H value of soils.

A. A. ELDRIDGE.

Four-year field trial with six different nitrogenous fertilisers. L. MEYER (Fortschr. Landw.,

1929, 4, 167—169; Chem. Zentr., 1929, i, 2226).—A comparative study of ammonium sulphate, ammonium chloride, sodium nitrate, and calcium nitrate.

A. A. ELDRIDGE.

Determination of tervalent arsenic [in plant protective materials]. G. FESTER (Z. angew. Chem., 1929, 42, 1040).—In the analysis of plant-spraying materials, such as Schweinfürth-green, the method involving boiling with caustic soda followed by titration with iodine solution in presence of sodium bicarbonate leads to low values for arsenious oxide when calcium compounds are present. More accurate results are obtained if the material is dissolved in boiling hydrochloric acid, and the solution, largely diluted, is then treated with ammonium carbonate solution until the copper deposit is redissolved. The blue solution is then titrated with 0.1*N*-iodine until it turns green. The results for a material containing calcium sulphate were in good agreement with those obtained by vaporising the arsenic in a stream of hydrochloric acid, and a check analysis on pure arsenious oxide gave a result of 99.9% of the theoretical.

H. J. DOWDEN.

New solvents for removal of arsenical spray residue. R. H. ROBINSON (Ind. Eng. Chem., 1929, 21, 1132—1135).—The most satisfactory single substance for dissolution of lead arsenate residue is hydrochloric acid in approx. 0.01% concentration. By the addition of certain sulphates and chlorides to the acid larger amounts of lead arsenate may be dissolved; both with the mixed solvents and with hydrochloric acid alone dissolution is rapid, reaching its maximum in under 5 min. A combination of hydrochloric acid with sodium sulphate appears to be the most useful of the solvents studied, but on account of the few commercial washing tests so far completed general recommendations cannot yet be made.

F. R. ENNOS.

Lowering of the carbonate horizon in the soils in the vicinity of Odessa due to irrigation. V. G. TANFILEV (Pochvovedenie, 1928, 23, No. 1—2, 96—98).

See also A., Dec., 1411, **Micro-determination of iodine** (REITH). 1413, **Determination of aluminium in plants** (WINTER and others). 1497, **Determination of aluminium in plants** (WINTER and BIRD). 1498, **Luxury consumption of potassium by plants** (BARTHOLOMEW and JANSSEN).

PATENTS.

Disinfection of seeds. W. P. RALEIGH and C. B. DICKEY, Assrs. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,734,646, 5.11.29. Appl., 1.2.28).—A disinfectant for maize and similar seeds consists of a mixture of a solid, inert carrier and yellow mercuric oxide to give a product containing 8—10% Hg.

F. G. CROSSE.

Manufacture and use of insecticides. C. N. HAND, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,734,519, 5.11.29. Appl., 12.12.23).—An aqueous suspension of *s*-diphenylthiourea and starch is claimed.

F. G. CROSSE.

Dispersions containing copper (B.P. 321,260).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Laboratory apparatus for diffusion experiments [with sugar beet]. V. STANĚK (Z. Zuckerind. Czechoslov., 1929, 54, 37—41).—A laboratory slicing machine for producing normal cossettes was constructed, the roots being moved over fixed Goller knives. Two forms of laboratory diffusion batteries are described (capacity about 200 c.c. and 1 litre, respectively), these being capable of giving a normal juice with a good exhaustion of the slices.

J. P. OGILVIE.

Determination of the sugar content of carbonatation scums [filter-press cake]. J. KADLEC (Z. Zuckerind. Czechoslov., 1929, 53, 747—748).—The content of sugar in unwashed carbonatation filter-press cakes, as determined directly by the usual method of analysis (using zinc nitrate to assist dissolution), was 11.13%. On the other hand, by extracting the unsweetened sample of cake by digesting with a large volume of water at 82°, evaporating down the extracts, and polarising them, a value nearly double the above was obtained. This considerable difference was not due to the presence of dextrorotatory substances extracted during the digestion, the determination of sucrose by the double-polarisation method having eliminated this possible source of error.

J. P. OGILVIE.

Use of "carboraffin" [decolorising carbon] in the sugar industry. E. ZAPPELLI (Internat. Sugar J., 1929, 31, 430—431).—Raw sugar was refined in Java on the Somobito plantation by remelting it, clarifying the resulting liquor with lime and phosphoric acid, filtering, and passing through Scheibler bags coated with "carboraffin." Only 0.1% of the carbon was used, calculated on the sugar in solution, and an average 50% decolorisation was obtained. Sugar was produced which was described as "very good."

J. P. OGILVIE.

Factors affecting colour in sorghum syrup. J. J. WILLAMAN and S. S. EASTER (Ind. Eng. Chem., 1929, 21, 1138—1145).—The colour value of sorghum syrup, as measured by the Pfund colour grader, varies linearly with the logarithm of the concentration (in degrees Brix) and with the p_H . On heating the diluted syrup the colour produced is proportional to the temperature but independent of the initial p_H . The most important source of colour is levulose. In factory practice colour production depends on the degree of inversion of the sucrose and on the time and temperature of heating, provided the reaction never becomes alkaline.

F. R. ENNOS.

Treatment of low-grade beet-sugar products. V. I. KUZNETZOV (Zhur. Sakh. Prom., 1927, 1, 235—238).—By liming at 90°, defecating, and saturating with carbon dioxide, or by dissolving in syrup, liming at 90°, and sulphitation, the colour is decreased but the purity is scarcely affected.

CHEMICAL ABSTRACTS.

Treatment of low-grade beet products. G. S. BENIN and B. E. KRASILSHCHIKOV (Nauch. Zapiski, 1929, 8, 73—90).—Sulphitation is preferred to carbonatation; for heating at 75—95° for 5—10 hrs., p_H 7.0 (remelts) or 6.5 (green syrups) is advisable, since at lower p_H values inversion occurs unless a lower temperature is employed.

CHEMICAL ABSTRACTS;

Experiments on the rapid cooling of low-grade massecuites. A. H. WARREN (Sugar News, 1929, 10, 317—323).—Experiments are described demonstrating how much more rapidly low-grade massecuite can be centrifuged hot than when cold, the capacity of the machines being thus doubled at least. A temperature of 60° is recommended. J. P. OGILVIE.

Zamaron's method of clarifying molasses for analytical purposes. K. K. BHARGAVA (Internat. Sugar J., 1929, 31, 421—423).—For decolorising solutions of sugar previous to polarisation, calcium hypochlorite, hypochlorous acid, and chlorine were found to be unsuitable, each causing inversion. Sodium hypochlorite increased the polarisation, though no invert sugar was formed. J. P. OGILVIE.

Glycerin from molasses. P. V. GOLOVIN (Zhur. Sakharnoi Prom., 1927, 1, 22—25).—Equal volumes of molasses and water are boiled for 1 hr. with turf powder, the solution is diluted to d_{20}^{20} 1.074—1.091 and fermented with yeast, after addition of ammonium salts, phosphates, calcium, manganese, and sodium hydrogen sulphite, for 36—48 hrs. The fermented solution is distilled to remove alcohol and aldehyde, the sulphite is recovered with lime, and the solution filtered and neutralised. The crude glycerin is distilled with steam.

CHEMICAL ABSTRACTS.

Relation of the "quality" of potato starch to the formation of mucilage. SPROCKHOFF (Z. Spiritus-ind., 1929, 52, 341—343, 350—351).—The formation of mucilage is due to the hydration of the amylopectin fraction of the starch molecule. This process begins at about 57° and becomes marked at 63—65°. The starch grain absorbs a small quantity of water with the production of two phases consisting of starch with very little water surrounded by a very dilute solution of starch. As the temperature rises the starch grains swell, allowing the absorption of more water, until at 100°, with a 0.5% solution, almost all the water is taken up and only one phase remains. The viscosity is then so great that sedimentation does not take place on cooling and keeping. A high viscosity value is taken as a criterion of the capacity of a starch to form a satisfactory mucilage. The viscosity depends on the temperature and concentration of the starch solution, and for the practical test of the starch should be determined at a temperature which is rigidly fixed and with a 0.4—1% solution of starch. With dilute solutions the true viscosity is given by the measurement, the internal friction between the two phases which occurs with higher concentrations being absent. With starch concentrations ranging from 1.2 to 3.6% there is a maximum viscosity depending on the temperature: 2% solution, max. 90—95°; 2.8%, 80—85°; 3.2%, 75—80°. At higher concentrations the maximum viscosity cannot be measured. With concentrations less than 1% there is no maximum viscosity, but the value rises rapidly between 90° and 100°. The "quality" of the starch is measured by the Saare test, which determines the power required to withdraw a disc from the starch mucilage. The resulting curves, which show maxima varying with the temperature, resemble those obtained from the viscosity determinations. Just as the viscosity

curves show a very steep fall between 75° and 90°, so the Saare test, being very sensitive at the same range of temperature, also indicates such changes, and the omission definitely to fix the temperature of the test has led to the publication of conflicting results.

C. RANKEN.

Report [on sugar cane]. HARRISON.—See XVI. **Lemon residues.** MELIS. **Sugar-beet pulp as source of pectin.** CODLING and WOODMAN.—See XIX. **Sugar-factory effluents.** NOLTE.—See XXIII.

See also A., Dec., 1488, **Chemistry of starch** (POLAK and TYCHOWSKI).

PATENTS.

Refining of sugar. E. N. EHRHART, Assr. to J. J. NAUGLE (U.S.P. 1,731,237, 15.10.29. Appl., 4.3.26).—A relatively dilute sugar solution ($d_{1.14}$ —1.24) is partially decolorised with spent carbon, "suchar," from the final decolorisation of a concentrated sugar solution, and the sp. gr. of the filtered liquor is increased to that required in the boiling pans by the addition of a more concentrated solution of crude sugar. The mixture is decolorised and purified by boiling with fresh carbon and the filtered syrup is passed to the boiling pans, the carbon being used again for the preliminary purification of more dilute liquor. A. R. POWELL.

Treatment of cellulose-containing materials (B.P. 299,844).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Treatment of brewing waters. H. LÜERS [with W. SCHREMPF] (Woch. Brau., 1929, 46, 469—474).—Beers brewed with a decarbonated water originally containing 15° of hardness, of which 14.2° were due to carbonates, showed variations depending on the method of decarbonation. The greatest increases in the nitrogen content of the wort were produced when the water was decarbonated either by the addition of lime or by neutralisation by lactic acid. Nitrogen assimilated during fermentation was chiefly of the formol type. Worts prepared from water neutralised by lactic acid showed the highest alkalinity of the ash, the greatest amount of buffer substances, the greatest increase in the content of phosphate, and, during fermentation, the maximum assimilation of nitrogen. Untreated water yielded a beer with a dark red colour and a coarse, bitter flavour. Water decarbonated by lime yielded a bright yellow-coloured beer with a pleasantly bitter flavour. The addition of gypsum as well as lime accelerated the ripening of the beer but lowered its quality, giving a harsher flavour. The colour of the beer was palest when brewed with water corrected by lactic acid, but the flavour was so mild that a higher hop rate was necessary to obtain the desired bitterness. A comparison was made between a soft water and the same water artificially carbonated by passing carbon dioxide into a suspension of calcium and magnesium carbonates. The original water yielded a wort containing more total and formol nitrogen, and more nitrogen was assimilated during fermentation. With the hardened water the buffering was greater and the phosphates and ash were less. Maltose and final attenuations differed but little.

The hardened water gave a beer with a rounder and fuller flavour.

C. RANKEN.

Determination of lactic acid in wine by "step" titration. J. TILLMANS and E. WEILL (Z. Unters. Lebensm., 1929, 57, 515—519).—The volatile acids are steam-distilled from 50 c.c. of wine, and after addition of a few drops of phenolphthalein solution to the residue cold saturated baryta solution is added along with 5 c.c. of 10% barium chloride solution until a faint pink colour is produced. In order to hydrolyse any lactic anhydride present, the mixture is warmed for 10 min. on the boiling water-bath with 2—3 c.c. of baryta solution until a pink colour persists, neutralised with carbon dioxide, and evaporated on the water-bath to 10 c.c. The solution is made up to 25 c.c. with water, and then to 100 c.c. at 15° with neutral 96% alcohol, added in a thin stream with stirring. After frequent shaking during 2 hrs., the liquid is filtered, and 75 c.c. of the filtrate at 15° are treated with 25 c.c. of 5% sodium sulphate solution. The mixture is shaken, and after remaining in a closed vessel for 15 min. is filtered; then 20 c.c. of the filtrate at 15° are evaporated to 4 c.c. with addition of a little pure animal charcoal, and again filtered, with careful washing of basin and filter, into a colourless 50-c.c. glass cylinder, 13 cm. high and 2.5 cm. in diam. The filtrate should amount to 20 c.c. A few drops of phenolphthalein are added and 0.1*N*-sodium hydroxide to the end-point. A control at p_H 3.2 is prepared by dissolving 21.008 g. of citric acid in 200 c.c. of *N*-sodium hydroxide, diluting to 1 litre, and mixing 43 c.c. of this with 57 c.c. of 0.1*N*-hydrochloric acid. 20 c.c. of this are placed in a similar cylinder to the first, and to both solutions 0.3 c.c. of a 0.01% solution of dimethylaminoazobenzene in 90% alcohol is added. The solution in the first cylinder is titrated with 0.1*N*-hydrochloric acid to the colour of the control. The lactic acid, x (in grams), in 1 litre of wine is given by $x = 1.45a$, in which a is the number of c.c. of 0.1*N*-hydrochloric acid used after deduction of 0.20 c.c. for the acid equivalent of the indicator.

W. J. BOYD.

Colorimetric micro-determination of glycerol in wine. C. DE COQUET (Bull. Soc. Pharm. Bordeaux, 1928, 66, 235—250; Chem. Zentr., 1929, i, 2115).—The method is based on the development of a greenish-blue colour by the addition of codeine and sulphuric acid to methylglyoxal produced by the action of bromine water.

A. A. ELDRIDGE.

Detection of fruit wine by the sorbitol process. FIESSELMANN (Mitt. Lebensm. Hyg., 1929, 20, 45—46; Chem. Zentr., 1929, i, 2599).—The test is rendered more difficult by the presence of much invert sugar. Such a wine is preferably first fermented by a pure yeast culture. Coloured wines and wines high in extractives are preferably treated with "eponite." The validity of the test is confirmed (cf. B., 1929, 619, 834).

A. A. ELDRIDGE.

Extraction of colour from red-juice grapes. JOSLYN and others.—See XIX.

See also A., Dec., 1397, **Mechanism of enzyme action** (NORD and WEICHERZ). 1488, **Enzyme catalysts** (ARMSTRONG and HILDITCH). Amylase from

Cumbu (*Pennisetum typhoideum*) (NARAYANA-MURTI and others). Chemistry of starch (POLAK and TYCHOWSKI). Malt catalase (MATSUYAMA). 1489, **Enzyme action: tyrosinase** (NARAYANAMURTI and AYYAR). 1490, **Effect of electrolytes on glycerophosphatase** (INOUE). **Fat-decomposing enzyme** (GYOTOKU and MATSUBARA). **Action of papain on polarisation of gelatin** (GORE). **Proteolytic enzymes of green malt** (HOPKINS). 1493, **Endo-cellular enzymes of *B. coli communis*** (YOUNG). 1496, **Vitamin-B contents of fresh yeast and of the derived dried yeast** (SCHEUNERT and SCHIEBLICH). 1498, **Post-mortal alcoholic fermentation of peas** (ZALESKI and NOTKINA).

PATENT.

Manufacture of yeast. E. A. MEYER, Assr. to FLEISCHMANN Co. (U.S.P. 1,724,952, 20.8.29. Appl., 30.4.27. U.K., 4.5.26).—See B.P. 275,328; B., 1927, 792.

XIX.—FOODS.

Minerals of wheat. I. Sulphur and chlorine. B. SULLIVAN and M. HOWE (Cereal Chem., 1929, 6, 396—400).—Determinations of magnesium, calcium, phosphorus, chlorine, and sulphur were made on a hard spring wheat, on the products milled from it, and on bread baked from the patent flour. These materials also were ashed by ignition for 16 hrs. at 590—600° and similar analyses then made. The wheat products and the bread contained about 0.2% S, but in the ash only traces could be found. The chlorine content of the products as calculated from the percentage found in the ash shows that 93—99% of the chlorine is lost during ignition. Practically all the sulphur in wheat is present as a constituent of cystine, and it was found that the sulphur contents of the materials increased with the protein contents.

H. J. DOWDEN.

Colloid chemistry of gluten. H. L. B. DE JONG and W. J. KLAAR (Cereal Chem., 1929, 6, 373—378).—When sodium hydroxide solution is slowly added to a gluten solution in dilute hydrochloric acid containing 1.134 g. of protein per 100 c.c. at p_H 4.6, a second liquid phase separates. The clear solution becomes at first increasingly opalescent and at a certain concentration of alkali again becomes clear. The globules show Brownian movement, are deformed by pressure, and are filterable through a porous crucible. The droplets are relatively rich in protein and are deeply coloured by iodine, the surrounding liquid remaining pale. The phase equilibrium is shifted by addition of alcohol, glycerin, sucrose, or acetone, and a cloudy gluten solution becomes clear with an acetone concentration of $\pm 30\%$ and opalescent again at 50%. The viscosity of the gluten solution falls rapidly when small amounts of electrolytes are added, the drop in viscosity depending on the valency of the anion.

H. J. DOWDEN.

Report on methods of analysis [for cereals]. M. J. BLISH (Cereal Chem., 1929, 6, 423—429).—Collaborative studies have shown that in protein determinations, replicated tests may vary over a range of 0.2% (cf. Treloar, following abstract), and accordingly the Committee of the American Association of Cereal

Chemists recommends that protein results should be reported to the first decimal figure only. It is further recommended that all equipment should be checked periodically by a large number of determinations on a typical flour, the variability of the results being compared with the standards. Bromocresol-green as the indicator for ammonia titrations is advocated (cf. Bailey, B., 1930, 11). H. J. DOWDEN.

Statistical study of collaborative protein determinations. A. E. TRELOAR (Cereal Chem., 1929, 6, 429—453).—Samples of three different flours of high, low, and average protein content were distributed to a number of laboratories to be analysed by official methods. Twenty determinations and twenty blanks from each investigator were received, and the results subjected to statistical analysis. The variations from laboratory to laboratory show that discrepancies of 0.35% for the average of three determinations and 0.6% for a single determination may occur in the protein analysis. H. J. DOWDEN.

Effect of storage and of various bleaching agents on the carotene concentration of flour. C. G. FERRARI and C. H. BAILEY (Cereal Chem., 1929, 6, 457—482).—To investigate the natural bleaching of flour by atmospheric oxidation of the carotene, samples of a straight-grade flour were stored in stoppered bottles (a) in the air at room temperature, (b) in carbon dioxide at room temperature, and (c) in air at 0°, the carotene content being determined at intervals by measuring the transmittancy of the petroleum extract to light, $\lambda = 435.8 \text{ m}\mu$ (cf. B., 1929, 657). Temperature exerted a greater influence than carbon dioxide in inhibiting the bleaching of the flour, and at the end of 100 days the carotene contents of the three samples had fallen to 1.85, 1.91, 2.12 p.p.m., respectively, the original concentration being 2.46 p.p.m. of flour. The influence on carotene of gaseous bleaching agents was studied by means of a special apparatus, which permitted a known volume of the gas to be admixed with flour contained in a revolving wooden box. With chlorine doses of 253, 315, and 394 p.p.m. of flour, the measurements immediately after treatment showed that 50, 66, and 75%, respectively, of the carotene had been bleached. The action of chlorine is not instantaneous, and after the large initial effect bleaching continues at a rate greater than can be accounted for by natural ageing. At the end of 104 days the concentration of bleached carotene in the above samples had risen to 66, 71, and 80%, respectively. Doses of chlorine of the same magnitude were used on a Marquillo wheat of high carotene content, viz., 4.56 p.p.m., and produced the same percentage bleaching. Nitrogen trichloride is used commercially for bleaching in the Agene process. Experiments on a straight-grade flour, using dosages of 15.9 and 47.8 pts. of nitrogen trichloride per million pts. of flour, showed immediate bleaching of 36% and 47%, and after 100 hrs. 43% and 60%, respectively. On the high-carotene wheat, using higher concentrations of nitrogen trichloride, correspondingly greater bleaching effects were produced: 96 p.p.m. of flour effected a 96% bleach in 71 days. Experiments were also made with Novadel (benzoyl peroxide mixed with calcium phosphate). The pow-

dered material was mixed with a sample of flour (500 g.) by agitation in a large jar for 20 min. Using Novadel concentrations of 0.011, 0.0145, and 0.016%, the bleaching produced at the end of 4 hrs. was 24, 38, and 41%, respectively of the original carotene content, and by the end of 153 hrs. 60% had been bleached by all concentrations. The influence of Novadel at various concentrations on the wheat of high carotene content was also studied. It appears that with this reagent all flours are bleached the same proportional amount in the same time; thus, in 197 hrs., all samples with all doses of the reagent were bleached to about 60% of the carotene. Large doses of the reagent produce more rapid results, but the final bleaching effect after a long interval of time is almost identical for all doses. Ultra-violet light is also an effective bleaching agent, and samples of flour exposed to the mercury-quartz arc were bleached 47% in 7 hrs. H. J. DOWDEN.

Influence of environment on the moisture content of flour and wheat. T. H. FAIRBROTHER (Cereal Chem., 1929, 6, 379—395).—Investigations have shown that the moisture content and consequently the gross weight of flour is closely related to the atmospheric humidity, the variations in weight when packed in cotton or in paper bags being about the same. By exposing samples in a closed chamber under conditions of controlled humidity and temperature it was proved that flour containing 15% of moisture may lose over 4% in weight in a dry atmosphere. When flour which has been stored in a moist atmosphere is subjected to dryer conditions for a short period a change in the hydration capacity occurs which is independent of temperature conditions, since on re-exposure to the original R.H. the loss in weight is not entirely regained. A bag of flour weighing 693.3 g. at 75% R.H. lost 15 g. when exposed for 24 hrs. to R.H. 36%. After the humidity had been rapidly increased to 75% again the flour regained 11.3 g. by the end of 120 hrs. and thereafter remained unchanged. From observations on a sample of flour containing 13.06% of moisture it was found that between 15% and 90% R.H., a linear relationship exists between moisture content and R.H. On mixing a wet wheat and a dry wheat an exchange of moisture occurs, and for a 1:1 mixture equilibrium is reached in 3 days, but equality of moisture is never attained, the final difference being about 2%. H. J. DOWDEN.

Influence of flour moisture on the Pekar test. A. W. ALCOCK and N. J. EDIGER (Cereal Chem., 1929, 6, 410—413).—When the moisture content of unbleached flour is lowered, the flour appears whiter as judged by the Pekar test, the original colour being resumed as the moisture is restored. By measuring the weight of crust per unit area, it was found that the dough formed by the flour of high moisture content is definitely thicker than that from the flour of low moisture content, even if the samples are dipped for a considerable time to allow both flours to imbibe equal amounts of water. Consequently the colour of the high-moisture flour dough is darker and it would be judged of low quality. It is recommended that before flours are compared by the Pekar method they should be brought to approximately the same moisture content. H. J. DOWDEN.

Plasticity of flour-water suspensions. J. L. ST. JOHN (Cereal Chem., 1929, 6, 400—410).—An attempt has been made to correlate flour quality with the plasticity of flour-water suspensions. The suspensions were made by adding the exact amount of water to a weighed sample of flour, stirring gently in a beaker, and finally working into a smooth suspension in a pestle and mortar, the whole procedure requiring 10 min. The author's plastometer was used (cf. B., 1928, 71), and it was found that duplicate measurements were in close agreement. The results obtained with ten different flours, expressed in terms of mobility, show that there is a gradual though irregular increase in mobility up to 8 hrs. from the time of mixing. There is also an increase in mobility with decrease in concentration of the suspension. The manometer pressures were maintained within narrow limits at 40 cm. and 60 cm. of water, but subsequent experiments showed that great accuracy of control is not essential. There is little change in mobility with age of the flour up to 2 years, after which it is not suitable for use in baking. Baking tests showed that there is a progressive increase in mobility as the loaf volume decreases, but there is no relationship between loaf volume and protein content or p_H of the flour.

H. J. DOWDEN.

Acetonedicarboxylic acid as a leavening agent.

E. O. WING (Ind. Eng. Chem., 1929, 21, 1145—1146).—A mixture of starch and acetonedicarboxylic acid when used as a leavening agent is equally effective in raising the baked product as is a commercial baking powder yielding the same amount of carbon dioxide. Such mixtures are moderately stable—one containing 15% of available carbon dioxide lost only 3% in 12 months—and possess the advantage of leaving no solid residue, although in one instance acetone was detected in the baked product. (Cf. A., 1928, 963.) F. R. ENNOS.

Panification [of flour] in presence of de-acidifying agents. D. MAROTTA and F. DI STEFANO (Annali Chim. Appl., 1929, 19, 459—466).—Bread was made from a high-percentage flour (85%) with additions of small proportions of either lime water, calcium glucosate, or sodium hydrogen carbonate. The first of these renders the bread less soft and less porous, and lowers the acidity by about 1°; the second leaves the bread soft and porous, but has little effect on its acidity; the third gives a soft bread with increased porosity, and pleasant smell and taste, the acidity being lowered by about 1°. Hence addition of sodium hydrogen carbonate is recommended when the flour used contains an exceptionally high percentage of the whole wheat corn. The acidity of the corn, discarding the germ, increases from the centre to the periphery, reaching a maximum in the grey zone preceding the bran proper. The acids present appear to be largely fatty acids and the grey zone contains a solid acid. T. H. PORE.

Points for consideration in baking tests. C. G. HARREL (Cereal Chem., 1926, 6, 414—423).—A survey of the results obtained over one year with the standard experimental baking test has shown that differences in moulding treatment introduce wide variations in the results, and will probably necessitate the use of moulding machines. A standard practice must be adopted

with regard to pan-greasing and greater attention given to oven-temperature control. H. J. DOWDEN.

Determination of sand in rice meal. E. WALTZINGER (Chem.-Ztg., 1929, 53, 891).—The extraction of the ash of the material with 5—25% sodium carbonate solution gives a result for sand which is much too high. Accurate results are obtained by boiling the meal, without ashing, with a 1.25% solution of sodium carbonate and washing the residue with 1.25% sulphuric acid.

A. R. POWELL.

Differentiation of cow's and goat's milk. F. G. KOHN (Z. Fleisch- Milch-hyg., 1929, 39, 279—281; Chem. Zentr., 1929, ii, 232—233).—When mixed with amyl alcohol and sulphuric acid (in the butyrometer) the casein of cow's milk forms larger and more voluminous flocks than does that of goat's milk; with the latter the layer is more mobile and the interface appears horizontal. The distinction is not observed with mixtures or with diluted cow's milk. A. A. ELDRIDGE.

Influence of neutral salts on alcohol coagulation of fresh milk. K. MITAMURA (Sapporo J. Forestry Agric., 1927, 19, 1—36).—Neutral salts (0.001N-solution) increase the sensitivity of fresh milk to coagulation by alcohol. The effect increases with the valency of the cation. Dialysis of milk decreases the sensitivity to coagulation by alcohol, which is not related to the p_H .
CHEMICAL ABSTRACTS.

Effect of heat on the antineuritic vitamin of milk. A. L. DANIELS, M. L. GIDDINGS, and D. JORDAN (J. Nutrition, 1929, 1, 455—466).—In superheated milk, except that dried by the roller process, there is considerable destruction of vitamin-B. In the closed method of pasteurisation vitamin-B is not destroyed; the slight loss in the open method is much less than that with evaporated or spray-dried milk. If milk is boiled quickly and cooled rapidly there is little loss of vitamin-B; temperature and aeration are important factors.

CHEMICAL ABSTRACTS.

Bacteria that survive and grow during pasteurisation of milk and their relation to bacterial counts. P. S. PRICKETT and R. S. BREED (New York State Agric. Exp. Sta., Bull. No. 571, 1929, 25 pp.).—The development of pin-point colonies in the plating of pasteurised milk is shown to be due to thermophilic and heat-resistant bacteria. Ordinary media and methods of incubation do not reveal these bacteria, so that microscopical examination is necessary to furnish a true bacterial count. Seven groups of organisms (three thermophilic and four heat-resistant) were isolated during the examination of milk at various stages of production and of farm and pasteurising plant and buildings. All these groups were isolated from pin-point colonies, and by adopting special media and appropriate incubation temperatures they were made to yield active cultures. The evidence tends to prove that the presence of these organisms is due to contamination at the farm, and measures are recommended for their control, such as more sanitary conditions of feeding and bedding and the disinfection of utensils. At the milk plant repasteurisation of tailings and returns will infect subsequent batches and the practice should be discon-

tinued. Foam should be entirely removed between batches of milk. Since thermophiles are sensitive to cold, refrigeration after pasteurisation will lower the bacterial count of the milk. H. J. DOWDEN.

Nature and significance of the volatile substances present in foodstuffs. J. KÖNIG and E. KRACHT (Z. Unters. Lebensm., 1929, **57**, 377—412).—Various foodstuffs were heated with water at 100° in the apparatus described by König and Schreiber (B., 1927, 344). The use of the expressed juice of vegetable products instead of the sliced material and the substitution of 5% salt solution for water gave slightly larger yields of volatile material, but neither of these modifications is recommended. The substitution of calcium hydroxide-hydrogen peroxide solution for ammoniacal silver oxide solution in the fourth Peligot tube was necessary in order to oxidise all the volatile reducing substances. In general, these yielded on oxidation a variable mixture of formic, acetic, and butyric acids, with two non-volatile acids, probably malonic and valeric acids. On heating various vegetable products at 100° or milk and flesh at 101—102° no volatile ammonia could be detected. Drying of the product at 50—60° produced changes in the yields of volatile substances. Carbon dioxide and formaldehyde are to some extent volatilised at these temperatures. Carrots and apples yielded increased quantities of hydrogen sulphide and mercaptans after being dried, whereas dried white cabbage yielded less of these substances than the fresh product. It is concluded that the labile substances occur in both a looser and a firmer combination. After storage of carrots in the earth or of apples in a cellar for 4 months, the volatile sulphur compounds had almost entirely vanished, carbon dioxide and formaldehyde had decreased, and the less volatile aldehydes had accumulated. The labile substances and their volatile products appear to be retained better in presence of organic acids (as in sauerkraut) than in the neutral or alkaline state. The behaviour of certain products occurring in foodstuffs on being heated with water at 100° was studied. Malonic acid yields acetic acid and carbon dioxide, but no reducing substances. Maleic and fumaric acids are stable. Oleic and stearic acids are slightly volatile in steam, but the former yields also reducing substances, including traces of formic acid and, after being heated for 10 hrs., considerable quantities of butyric acid. Cholesterol heated with water at 100° with a current of air or nitrogen passing through the liquid yields more reducing substances than does ergosterol. Formic acid was always detectable among the products. Lecithin yields formaldehyde and other volatile aldehydes on heating with water at 100° and a small quantity of a volatile phosphorus compound at 101—102°. Nuclein yields under the same conditions no formaldehyde nor volatile phosphorus compound and only traces of other volatile reducing substances. Irradiation with ultra-violet light had no influence on the yields of volatile substances from these compounds. W. J. BOYD.

Vitamin content of honey and honeycomb. H. B. KIFER and H. E. MUNSELL (J. Agric. Res., 1929, **39**, 355—366).—Examination of various samples of

honey and of the comb revealed in no case detectable quantities of vitamin-A, -B, -C, or -D.

A. G. POLLARD.

[Use of the] formol titration in examination of honey. A. GOTTFRIED (Z. Unters. Lebensm., 1929, **57**, 558—560).—The observations of Tillmans and Kiesgen (B., 1927, 456) are confirmed, in that the formaldehyde titration values of honey in general correspond with the results of the Ley and Fiehe tests and of the Lund precipitation method. When 20 g. of honey in 100 c.c. of water require about 1 c.c. or less of 0.1N-sodium hydroxide, the presence of artificial honey is indicated. W. J. BOYD.

Vitamins in canned foods. VIII. Home canning and commercial canning contrasted in their effect on vitamin values of pears. M. M. KRAMER, W. H. EDDY, and E. F. KOHMAN (Ind. Eng. Chem., 1929, **21**, 859—861; cf. B., 1929, 450).—The growth, survival periods, and severity of scurvy of rats have been used to study the vitamin contents of Kieffer and of Bartlett pears in the raw state and after canning by commercial and by domestic methods. As has been shown in the case of apples (cf. B., 1925, 81), if precautions are taken to remove oxygen, Kieffer pears can be canned commercially without any apparent loss of vitamin-C, although by domestic methods vitamin-C is almost completely destroyed (cf. B., 1927, 638). The effect of the maturity of the pears at the time of canning is somewhat obscure. The vitamin-C content of Bartlett pears is relatively low, and is approximately equal to that of Kieffer pears or apples. Canning destroys a definite though small proportion of the vitamin-C content of Bartlett pears, and the situation is not improved by removal of oxygen. This variety of pear is not rich in vitamins-A and -B. H. J. DOWDEN.

Composition of West Indian seedling avocados. J. TILT and M. WINFIELD (J. Home Econ., 1928, **20**, 43—46).—Maximum, minimum, and average values for different varieties and stages of maturity are: H₂O 81.65, 83.92, 83.02; protein 1.12, 1.16, 1.14; fat 6.43, 8.09, 7.33; carbohydrate 6.72, 6.92, 6.82; crude fibre 1.16, 1.19, 1.17; ash 1.02%. The ash (g. per 100 g. of fresh avocado) contained P 0.0477, 0.0507, 0.0492; Fe 0.0054, 0.0072, 0.0063; Ca 0.0368, 0.0381, 0.0370; Mg 0.0323, 0.0407, 0.0365.

CHEMICAL ABSTRACTS.

Quality and maturity of apples. J. L. ST. JOHN and O. M. MORRIS (J. Agric. Res., 1929, **39**, 623—639).—Chemical analysis of Jonathan apples in 1923—25 showed that the amounts of sugar etc. vary in different years, and that although the amounts of dry matter, ash, nitrogen, and reducing sugars show no progressive variation during the growing season, the total sugars increase due to increase in sucrose, and the percentage of acid tends to decrease. For dessert quality in apples the amount of sugar present is of more importance than that of acid. The decrease in the percentage of alcohol-insoluble, acid-hydrolysable fraction during the growing season is striking and needs further investigation.

E. HOLMES.

Sampling cleaned apples for determination of arsenical spray residue. J. W. BARNES and C. W.

MURRAY (Ind. Eng. Chem., 1929, **21**, 1146—1147).—For apples carrying not more than 0.01 grain $\text{As}_2\text{O}_3/\text{lb.}$, a sample of six apples taken at random from a lot all parts of which have had the same treatment will give an accuracy of ± 0.0015 grain/lb. F. R. ENNOS.

Chemical treatment of lemon residues. B. MELIS (Giorn. Chim. Ind. Appl., 1929, **11**, 399—404).—In America these residues are worked almost exclusively for the extraction of the pectins, but the procedure here described yields acid glucose extract (from the internal residues), bitter glucose extract (from the skins), and fats, nucleoproteins, pectins, and cellulose from both classes of residue. The main operations consist of drying of the finely-minced material, alcoholic extraction, and treatment of the residual pectocellulose to extract pectins and cellulose. The alcohol extraction is carried out in a reflux apparatus with 80% alcohol, and is repeated until the alcohol remains colourless. The combined extracts are concentrated under reduced pressure and the alcohol is recovered, the alcohol-free residue being taken up in water, and collected on a filter to remove separated fats and proteins, which are washed with hot water. Concentration of the filtrate yields about 6% of dense glucose extract (calculated on the original material) having, for the inner (outer) residues of the fruit, the mean composition: citric acid 23.2 (2.45), dextrose 31.27 (35.40), ash 3.26 (1.65), pectins 0.40 (—), water and undetermined matter 41.87 (60.50)%. The material filtered off represents about 0.6% of the fresh residues and contains about 25% of fats and 75% of nucleoprotein. The pectocellulose residue from the alcohol extraction, when freed from alcohol by distillation, forms about 9 (15)% of the inner (outer) residues. To separate the pectin and cellulose, it is steeped for 20 min. with cold 0.1% chlorine water, which is then heated to boiling and stirred. In this way the whole of the pectin passes into solution, leaving a white porous cellulose through which the dense, almost syrupy pectic liquors readily filter. Colloidal precipitation of the pectin, as practised in America, is attended by various inconveniences, and it is found preferable to precipitate by means of an aluminium salt and ammonia and to treat subsequently with alcoholic hydrochloric acid. The pectin thus obtained contains not more than 0.4% of ash and gives perfect jellyfication with 200 times its weight of sugar. A method of analysis of pectocellulose, based on the above procedure, is described. The results of various analyses give the mean percentage composition: pectin 37.6, cellulose 34.6, ash 4.3, undetermined 23.5. T. H. POPE.

Effect of temperature and time of heating on extraction of colour from red-juice grapes. M. A. JOSLYN, H. B. FARLEY, and H. M. REED (Ind. Eng. Chem., 1929, **21**, 1135—1137).—The intensity of the colour extracted increases slowly as the temperature is raised from 20° to 70°, but more rapidly from 70° to 90°; the rate of increase is greater at the higher than at the lower temperatures. The tannin content of the juice is similarly affected by time and temperature of heating.

F. R. ENNOS.

Organisms found in spoiled tomato products. C. S. PEDERSON (New York State Agric. Exp. Sta., Tech.

Bull. No. 150, 1929, 46 pp.).—Bottles etc. of spoiled tomato products, e.g., ketchup, have been examined and the organisms causing the spoilage have been isolated, identified, and characterised. Of the six most commonly-occurring species, five were of the *Lactobacillus* type, viz., *L. lycopersici*, *gayoni*, *pentoceticus*, *mannitopoeum*, and *plantarum*, and the sixth was *Leuconostoc pleofructi*. Of these, none survived for more than 15 min. at 76°, so that they should all be destroyed in properly conducted canning operations. A key for the identification of the organisms has been developed.

H. J. DOWDEN.

Control of spoilage in tomato products. C. S. PEDERSON and R. S. BREED (New York State Agric. Exp. Sta., Bull. No. 570, 1929, 16 pp.).—The organisms causing spoilage of tomato products are of the *Lactobacillus* type (cf. preceding abstract), the products of which, though not poisonous, are disagreeable to the taste and slimy in appearance. Owing to the viscosity of these materials a longer period of processing during canning operations is necessary to allow the centre of the container to attain the destructive temperature. Removal of unripe or rotten fruits during sorting and more careful washing will help to eradicate the organisms, whilst closer attention to temperature control, especially after breakdowns, and the elimination of wooden holding vats are most necessary. The stoppers of bottles, jars, etc. should be sterilised, and material which has been allowed to cool before sealing should be resterilised. For the storage of tomato pulp, wooden barrels should be abolished and the 5-gal. cans must be carefully handled and adequately supported by staging between tiers.

H. J. DOWDEN.

Evaluation of preserved crab products and detection of crab constituents. G. BÜTTNER and A. MIERMEISTER (Z. Unters. Lebensm., 1929, **57**, 431—437).—It is not possible to distinguish the natural pigment of crab products from added artificial dyes by means of ultra-violet light, as the luminescence attributed to the former is due to another component of crab tissue; nor is it possible to detect artificial pigments in presence of the natural pigment by the wool-stain test. By reduction with stannous chloride and hydrochloric acid the artificial pigments may, however, be destroyed and the natural pigment left unchanged. The latter is slowly decomposed by light, especially by ultra-violet light, but with proper precautions the natural colour of preserved crab products may be retained unimpaired.

W. J. BOYD.

Detection of castor bean in feeding-stuffs. M. WAGENAAR (Z. Unters. Lebensm., 1929, **57**, 413—418).—The finely-powdered material is heated for 1 hr. on the boiling water-bath with 4 g. of potassium chlorate and 50 c.c. of 2N-hydrochloric acid for every 1 g. of sample. Then 50 c.c. of 4N-sodium hydroxide are added for every 1 g. of sample, and steam is passed through the mixture for 1 hr. The undissolved residue, consisting almost entirely of platelets of *Ricinus* palisade cells, is separated by means of the centrifuge and mixed with a syrup of invert sugar prepared from 70 g. of sucrose, 30 g. of water, and 1 g. of citric acid. The area of palisade platelets in the whole suspension is found by

measuring the area of these in an aliquot portion under the polarising microscope, using a calibrated cover-glass (1500 mm.² = 1 g. of fat-free castor bean). Under polarised light the platelets appear as opaque areas surrounded by a bright fringe.

W. J. BOYD.

Luminescence of creatinine. G. REIF (Z. Unters. Lebensm., 1929, 58, 28—32).—Creatinine ordinarily exhibits a blue luminescence in ultra-violet light, but when its solution in butyric acid is heated at 165—170° for 10 min. and the crystalline mass which separates on cooling is recrystallised from alcohol and the crystals are washed with ether, they show marked yellowish-green luminescence. Repeated crystallisation does not affect the intensity of the luminescence. The crystals have the elementary composition of creatinine. The abnormal luminescence changes to the original bluish luminescence in acid solution, but is stable in alkaline solution. Only fatty acids, particularly butyric acid, produce the change to the yellowish-green luminescence; inorganic, tartaric, and lactic acids do not produce it. It is probably due to transformation into the enol form. Other amino-acids, amides, and organic bases do not behave like creatinine in this respect. The simultaneous occurrence of creatinine and fatty acids in many foodstuffs may have an influence on the luminescence of these foodstuffs.

W. J. BOYD.

Sugar-beet pulp as a source of pectin. A. J. CODLING and H. E. WOODMAN (J. Agric. Sci., 1929, 19, 701—714).—Dried sugar-beet pulp contains about 67% of nitrogen-free extractives of which a large proportion is pectose. Successive digestions of pulp with 0.5% ammonium oxalate at 100° extracts 34.5% of pectin calculated on the dry weight of pulp; a single prolonged digestion gives 32.2%. Acid digestion with 0.5% oxalic acid, 0.6% tartaric acid, 0.05N-hydrochloric acid, etc. leads to greater extraction owing to quicker hydrolysis of pectose to pectin, but does not necessarily enhance pectin yield. Prolonged digestion with water at 100° leads to satisfactory extraction of pectin. Beet-pulp pectin cannot impart a jelly condition to syrups of sucrose and free acid, but this inability is connected neither with the mineral impurities nor with changes in the pulp pectose during factory drying of the material. The pectin of beet pulp so far extracted has, therefore, no technical significance.

E. HOLMES.

Protein content as a factor in grading wheat. H. M. TORV and others (Nat. Res. Council Canada Bull., 1929, 13, 7—60).—An interim report.

CHEMICAL ABSTRACTS.

Cause and prevention of cloudiness [in beverages]. E. WALTER (Destillateur u. Likörfabr., 1929, 42, 119; Chem. Zentr., 1929, i, 2596).

Ammonia titrations. BAILEY.—See VII. **Cacao butter.** SCHMANDT; SCHELLBACH. **Pulasan and rambutan fats.** GEORGI and TEIK.—See XII. **Report [on butter].** HARRISON.—See XVI.

See also A., Dec., 1496, **Nutritive value of garbanza pea** (MITCHELL). 1497, **Production of avitaminosis; vitamin-C** (RANDOIN and LECOQ).

PATENTS.

Dough composition and its manufacture. J. R. WHITE (U.S.P. 1,729,409, 24.9.29. Appl., 22.3.28).—Yeast for bread making is propagated in a series of fermentable liquors, *e.g.*, molasses, each containing a higher concentration of sodium chloride and of a bromate or iodate than the preceding, and the final liquor containing 3% of sodium chloride and 0.75% of potassium iodate or bromate together with 1—1.75% of mucic acid.

A. R. POWELL.

Distillation and complete recovery of alcohol produced in baking ovens. N. DE NAVROTZKY (B.P. 305,223, 31.1.29. Fr., 2.2.28).—Vapour from the oven is drawn off and the alcohol condensed by passing through a vertical fractionating column containing an air-cooled condenser followed by a series of scrubbing chambers and a condensing chamber. Provision is made for returning water and dilute liquors to the oven. The air current used for cooling the condenser is actuated by the chimney draught of the oven.

E. B. HUGHES.

Preservative treatment of fresh fruit. E. M. BROGDEN, Assr. to BROGDEN Co. (U.S.P. 1,732,118, 15.10.29. Appl., 2.3.25).—Citrus fruit is pickled in an alkaline hypochlorite solution and the surface well rubbed to remove spores of blue mould, then dried, and coated with a thin film of waxy material.

A. R. POWELL.

Preparation of milk, skimmed milk, or butter-milk free of milk sugar. J. POHLMANN and J. R. F. RASSERS (U.S.P. 1,732,026, 15.10.29. Appl., 10.11.28. Holl., 26.8.27).—See B.P. 320,497; B., 1929, 1030.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of small traces of carbon monoxide in ethylene. B. S. WALKER and O. E. ALLEY (Anesth. and Analg., 1929, 8, 227—229).—Manchot and Scherer's reagent (silver oxide in pyridine) is specific for carbon monoxide in presence of ethylene under certain conditions of alkalinity. The pyrotannic acid method of Sayers, Yant, and Jones is also recommended.

CHEMICAL ABSTRACTS.

Detection of lead and copper salts in citric and tartaric acids by the official (D.A.B. VI) method. P. SCHÜTZ (Pharm. Ztg., 1929, 74, 1127—1128).—The dilute standard lead acetate solution prescribed for this test must be freshly prepared because it often becomes weaker when preserved in glass containers, owing to interaction of the lead salt and glass. This has been noted previously for lead nitrate (*cf.* Bernard, B., 1925, 831). Glass vessels which satisfy the official specification may or may not take up lead in this way. The official method is also unsatisfactory because in making up the dilute solution (10 mg. Pb per litre) no allowance is made for the sp. gr. of a 10% lead acetate solution, so that actually the dilute standard solution contains 10.6 mg. Pb per litre.

W. A. SILVESTER.

Nipagin [methyl *p*-hydroxybenzoate] as a preservative for 3% hydrogen peroxide solution. G. TELLERA (Pharm. Zentr., 1929, 70, 727—728).—Addition of nipagin very greatly improves the keeping properties, whether the solution be exposed to the air or kept in sealed vessels.

S. I. LEVY.

Comparison of the official pepsins of various pharmacopœias: correspondence between the different titres. P. LEGRAND (J. Pharm. Chim., 1929, [viii], 10, 385—401).—The methods of preparing and standardising pepsin as given by such pharmacopœias as those of England, France, Germany, America, Belgium, Spain, etc. are reviewed. The factors for converting French standards into the English, German, American, and Belgian ones, and *vice versa*, are given.

A. A. GOLDBERG.

Standardisation of preparations containing vitamin-D. O. SCHULTZ (Deut. tierärztl. Woch., 1929, 37, 152—155; Chem. Zentr., 1929, i, 1972).—A definition of units.

A. A. ELDRIDGE.

Iodine content and biological activity of thyroid preparations. A. STASIAK (Ber. Ungar. pharm. Ges., 1928, 4, 385—391; Chem. Zentr., 1929, i, 1845—1846).—The iodine contents of tablets, corresponding with the same quantity of thyroid substance, varied considerably. Aqueous extracts of thyroid gland contain only traces of iodine and are devoid of physiological activity.

A. A. ELDRIDGE.

American "poison barley." P. W. DANCWORT (Deut. tierärztl. Woch., 1929, 37, 170—171; Chem. Zentr., 1929, i, 2435).—The results of chemical analysis scarcely deviated from the normal; hydrogen cyanide and alkaloids were absent.

A. A. ELDRIDGE.

Sterilisation of urotropine [hexamethylenetetramine] solutions. V. CAZZANI (Boll. Chim. farm., 1929, 68, 821—823).—A criticism of the method of sterilisation of hexamethylenetetramine solutions at 120° for 20 min. given by Leulier, Simeon, and Finck (cf. Cazzani, B., 1928, 68). After one sterilisation at 70° for 30 min. only traces of formaldehyde and no ammonia (Nessler's test) can be detected; after three such sterilisations the amount of formaldehyde is increased, but still no ammonia is detected, whilst after heating at 100° for 1 hr. a strong positive test for ammonia is obtained. At 120° polymerides of formaldehyde, formic and carbonic acids, methyl formate, and other products may be formed. Sterilisation is best effected at 80°.

J. W. BAKER.

Determination of nicotine in a cadaver. B. KROFT and G. STEINHOFF (Arch. Pharm., 1929, 267, 609—616).—The dipicrate method of Pfyl and Schmidt (B., 1927, 955) gave good results when applied to the determination of nicotine in the cadaver of a boy who died from nicotine poisoning. The best results, however, were obtained when the nicotine was extracted by ether from the alkaline suspension of the tissues, precipitated with an ethereal solution of picric acid, and the resulting ether-insoluble dipicrate weighed.

C. C. N. VASS.

Medicinals of the somnifen type. E. V. CHRISTENSEN (Arch. Pharm., 1929, 267, 589—599).—Greater accuracy in the determination of the total barbituric acid content in preparations containing this acid or its derivatives is attained if chloroform is used to extract the free acids instead of ether. Diethyl- and dipropylbarbituric acids are separated by fractional crystallisation from water. The allylbarbituric acid content is determined by bromination, which occurs at the allyl double linking, and subsequent iodometric titration. Diethylamine, as the salt of the barbituric acid, is deter-

mined by direct titration with hydrochloric acid, using methyl-red as indicator.

C. C. N. VASS.

Oil of petitgrain. L. S. GLITCHITCH and R. NAVES (Parfums de France, 1929, 7, 60—66; Chem. Zentr., 1929, i, 3045).—The oil obtained by steam-distillation from the leaves and branches of Sicilian *Citrus [Bigaradia]* had d_{20}^{15} 0.875, α_D^{20} 21° 25', n_D^{20} 1.4739, acid value 1.12, ester value 42.7. The following constituents were detected: citral, camphene, *d*- α -pinene, cineole, linalool, geraniol, nerol, sesquiterpenes, a cyclic sesquiterpene alcohol, and a phenol (phenylurethane, m.p. 133.5—134°; benzoyl derivative, m.p. 107—108°). Hydrolysis afforded geranic and acetic acids, and traces of nitrogenous bases. The approximate composition is: terpenes 55, free *l*-linalool 4, free geraniol and nerol (1:1) 3, linalool and terpineol as acetate 2, geraniol and nerol as geraniate and acetate 10, citral 15, sesquiterpene substances 2, combined acids (geranic and acetic) 3%.

A. A. ELDRIDGE.

Vitamin-A in ratfish-liver oil. NORRIS and DANIELSON.—See XII. Report [on tobacco]. HARRISON.—See XVI. Lemon residues. MELIS.—See XIX.

See also A., Dec., 1410, Nephelometric determination of "argyrol" (RIMATTEI). 1462, 1-Phenyl-3-methyl-5-pyrazolone (BRUNNER and MOSER). 1470, Strychnos alkaloids (LEUCHS and KRÖHNKE). 1471, β -Anilinopropionamide-4-arsinic acid etc. (HAMILTON and SIMPSON). Pyridine and quinoline derivatives (BINZ and others). 1490, Nephelometric determination of pepsin (VAN ARKEL). 1496, Concentration of vitamin-B (GUHA and DRUMMOND). 1497, Production of avitaminosis: vitamin-C (RANDOIN and LECOQ). 1499, Protein complexes in tobacco (GAVRILOV and KOPERINA). Determination of protein in tobacco (GAVRILOV and TARANOVA).

PATENTS.

Manufacture of active substances from vegetable organisms. SCHERING-KAHLBAUM A.-G. (B.P. 295,361, 9.8.28. Ger., 10.8.27. Addn. to B.P. 291,005; B., 1929, 911).—Extracts free from tissue and containing germinal gland hormones, prepared from vegetable organisms as described in B.P. 271,492 (B., 1928, 769), are subjected to irradiation with ultra-violet rays.

L. A. COLES.

Water-soluble substance containing colloidal silver chloride. O. NEUBERT, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,734,269, 5.11.29. Appl., 30.7.28. Ger., 9.8.27).—See B.P. 317,121; B., 1929, 911.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Preservative for photographic industry. W. LESZYNSKI (Phot. Ind., 1929, 27, 426—427; Chem. Zentr., 1929, ii, 121).—Experiments with methyl *p*-hydroxybenzoate as an emulsion preservative are described.

A. A. ELDRIDGE.

Photochemistry of mercuric iodide. LÜPP-CRAMER (Phot. Korr., 1929, 65, 97—100; Chem. Zentr., 1929, i, 3071).—With emulsions of red mercuric iodide solarisation is not observed, a final condition being attained by the photochemical reaction. Iodine acceptors are without influence. Mercuric iodide emulsions exhibit

phenomena corresponding with the Schwarzschild effect.

A. A. ELDRIDGE.

Silver iodide problem. A. STEIGMANN (Phot. Ind., 1929, 27, 375—376; Chem. Zentr., 1929, ii, 120).—The effect on the sensitivity of substitution of silver iodide by colloidal silver iodide in an emulsion is described.

A. A. ELDRIDGE.

Herschel effect in a fogged pankine-film. A. STEIGMANN (Phot. Ind., 1929, 27, 507—508; Chem. Zentr., 1929, ii, 120).—The Weigert and Herschel effects are considered to be interdependent. A. A. ELDRIDGE.

Action of developers. K. WENSKE (Phot. Ind., 1929, 27, 482—484; Chem. Zentr., 1929, ii, 121).

Silver iodide bleach-out layers. LÜPPO-CRAMER (Phot. Ind., 1929, 27, 506—507; Chem. Zentr., 1929, ii, 120).

See also A., Dec., 1382, **Growth of silver in gelatin layers** (ARENS and EGGERT). 1404, **König-Marten spectrophotometer** (SCHAUM and others). 1405, **Action of polarised light on photographic plates** (COTTON). **Blackening surfaces of two slightly sensitive emulsions** (ARENS and EGGERT). **Photochemistry of silver halides** (SCHMIDT and PRETSCHNER). 1431, **Photosensitiveness of nitroprussides** (LEFFMANN and PINES).

PATENTS.

Photographic production of differentially tanned colloid images. G. KÖGEL (B.P. 315,236, 3.4.28).—A light-sensitive dye or such quinone as is not a dye is incorporated in the colloid film, which is treated after exposure with a solution of potassium dichromate. After washing, any of the usual after-treatments applied to dichromated colloid layers may be used. In examples the use of sodium anthraquinone-2:7-disulphonate and of stilbenephénylazonium (1:2:3-triphenylquinoxalinium) chloride is claimed.

J. W. GLASSETT.

Preparation of anaglyphs. KALLE & Co. A.-G. (B.P. 301,490, 5.11.28. Ger., 1.12.27).—Light-sensitive diazo-compounds are used in the preparation of complementary-coloured stereo part-pictures. For example, separate celluloid films are coated with (a) 2-ethylaminotoluene-5-diazonium fluoroborate and H-acid for blue, and (b) 1-diazo-2-naphthol-4-sulphonic acid and 1-phenyl-3-methyl-5-pyrazolone for orange, and are exposed under appropriate diapositives. The part-pictures are superposed and viewed through eye-glasses coloured blue and orange (preferably with the same dyes as are developed in the prints).

C. HOLLINS.

X-Ray photographs (B.P. 320,540).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Modern mining explosives. W. CULLEN (Bull. Inst. Min. Met., 1929, No. 302, 36 pp.).—The lines on which the explosives industry has been proceeding in recent years are reviewed.

S. BINNING.

Black powder. K. A. HOFMANN (Sitzungsber. preuss. Akad. Wiss., 1929, 25, 509—515).—Experiments with black powder containing percentages of sulphur varying from 0 to 10 show that the strength of the mixture (c.c. of gas evolved \times heat evolved in g.-cal.) remains practically constant, as an increase in the sulphur present diminishes the heat evolved but increases the volume

of gaseous products. For blasting powders an increase in the sulphur content to 15—20% is justified by the increased volume of the gaseous products. Sulphur lowers the ignition point of the charcoal-nitrate mixture, and this renders it more easily ignited by heat. Investigation of the part played by sulphur in the combustion of black powder shows that it exerts an autocatalytic effect on the reaction velocity.

S. BINNING.

Picric acid. DESVERGNES.—See III. **Nitrocellulose.** PARTRIDGE.—See V.

XXIII.—SANITATION; WATER PURIFICATION.

Treatment of sugar-factory effluents with chlorine. E. NOLTE (Z. Ver. deut. Zucker-Ind., 1929, 79, 463—469).—Chlorination has been applied with considerable success in the treatment of waste waters from beet-sugar factories, in particular where the water is returned to the factory. For example, the disposal of flume and wash waters has been in part solved by using them repeatedly with periodic chlorination at the rate of 10—15 g. of chlorine per cub. m. every 4 or 5 days. It is not practicable to destroy organic matter in the effluents so as to prevent subsequent fermentation, although hydrogen sulphide can be oxidised. Chlorination of fresh waste waters will check decomposition, so that in using the waters again the impurities introduced into working are only those natural to the processes, and not the much more highly melassigenic products of fermentation and putrefaction, such as organic acids.

J. H. LANE.

Lime-aggressive and rust-prevention inhibiting carbonic acid in natural water. J. TILLMANS (Z. Unters. Lebensm., 1929, 58, 33—52).—A review of the author's work on this subject. The varying rôles of carbon dioxide and of oxygen dissolved in water in relation to the action of natural waters on calcareous and iron structures are exhaustively discussed.

W. J. BOYD.

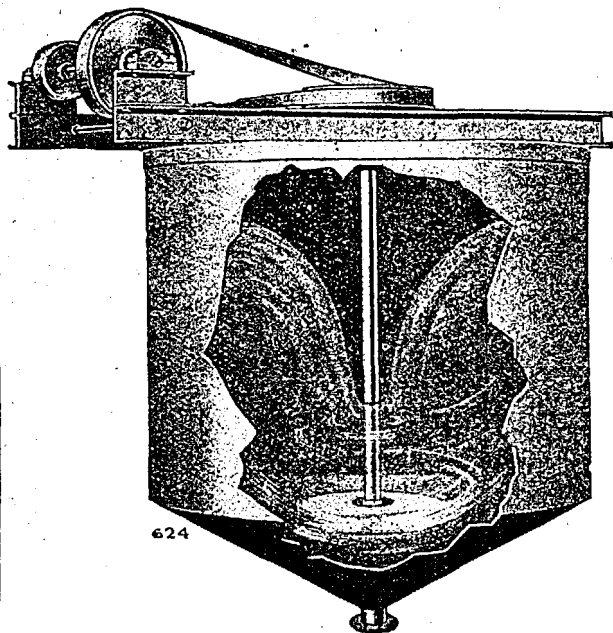
Detection of nitrites in water. S. VERGNOUX (Bull. Sci. pharmacol., 1929, 36, 146—147; Chem. Zentr., 1929, ii, 82).—Sulphuric acid (80 g.) is added, with cooling, to a filtered solution of neutral-red (2 g.) in water (18 g.), and the mixture is protected from light. Water (100—150 c.c.) is treated with 5 drops of the reagent; if more than 0.5 mg. per litre of nitrous acid is present, the green colour becomes blue or, with smaller quantities, violet. The sensitivity is 0.1 mg. per litre.

A. A. ELDRIDGE.

Direct enumeration on solid medium of *B. coli* contained in a large volume of water. DIENERT and ETRILLARD (Ann. Inst. Pasteur, 1929, 43, 1278—1281).—The *B. coli* in 100 c.c. of the water are separated by shaking with alumina, which, after centrifuging off and mixing with a dilute gelatin solution, is spread on a suitable solid medium. The colonies which then develop on incubation are counted. W. O. KERMAK.

Brewing waters. LÜERS.—See XVIII.

See also A., Dec., 1411, **Micro-determination of iodine** (REITH). **Micro-determination of nitrates and nitrites** (WORDICH). 1494, **Disinfecting properties of alkylphenols; *n*-butylphenol** (RETTGER and others).



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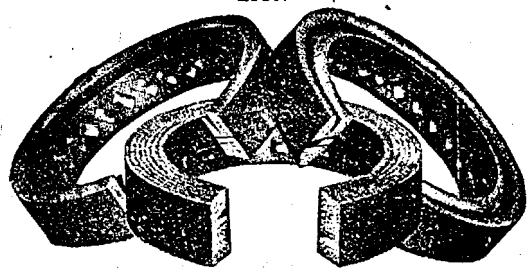
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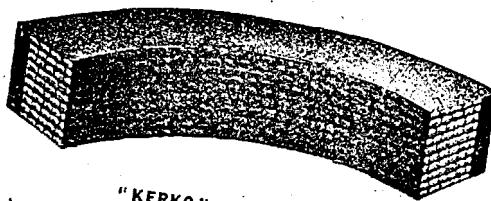
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BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JAN. 17 and 24, 1930.*

L—GENERAL; PLANT; MACHINERY.

Flow of pseudoplastic materials. R. V. WILLIAMSON (Ind. Eng. Chem., 1929, 21, 1108—1111).—The resistances to viscous and plastic flow exhibited by pseudoplastic materials are separated by a graphical treatment, and the following equation of flow is developed from these considerations: $F = fS/(s + S) + S/\phi_x$, where F is shearing stress, S is rate of shear, ϕ_x is apparent fluidity at infinite rate of shear, f is stress required to overcome plastic resistance at infinite rate of shear, and s is a constant which determines the curvature of the shear-stress curve for any given values of f and ϕ_x . The value of ϕ_x represents the slope of the asymptote to the shear-stress curve and corresponds to Bingham's mobility constant. The ratio of "plasticity constant" to "viscosity constant" gives another constant which appears to be a measure of that property commonly known as "false body." S. S. WOOLF.

Evaporation of water in open pans. G. W. HIMUS (Inst. Chem. Eng., Dec. 6, 1929, 53—61. Advance copy).—Formulae previously proposed by Hinchley and Himus for the rate of evaporation of water from open pans are, for still air, $W = 0.02(p_s - p_d)^{1.2}$, and in currents of air $W = (0.031 + 0.0135v)(p_s - p_d)$, where W is the evaporation in kg./m.²/hr., p_s the vapour pressure of the water, p_d that of the moisture in the air in mm. of mercury, and v is the air velocity. The index 1.2 is an expression of convection effects. The constants show a certain variation between different pans, and the second formula is evidently unsatisfactory. Considering the evaporation in a current of air to be equivalent to that in still air with an addition due to the movement, a formula of the type $W = 0.02 \times (p_s - p_d)^{1.2} + X$ is preferred; X is found to be in linear relationship with $(p_s - p_d)^{1/1.2}$. The factor of relationship, m , is found to vary directly with the draught velocity, but the relation of m to v is not linear. The evaporation is further affected by the shape of the pan and the section of the air duct. Within the limits, however, for which $X = m(p_s - p_d)^{1/1.2}$ the final formula proposed is $W = 0.02(p_s - p_d)^{1.2} + 0.0446v^{0.77} + (p_s - p_d)^{1/1.2}$. C. IRWIN.

Process and apparatus for recovery of volatile solvents. J. BODEWIG (Chem. Fabr., 1929, 471—473, 479—481).—The necessity for the recovery of the vapours of alcohol and ether from the atmosphere of the factory first arose in connexion with the manufacture of nitrocellulose and artificial silk by the Chardonnet process. Three possible courses are: (1) condensation, (2) absorption by solid reagents as activated carbon or silica gel, (3) absorption by liquid reagents. Con-

densation alone can only give a possible yield of 40—50%, according to the solvent and the temperature range. It results also in the simultaneous condensation of water vapour. It is a practical method only when dealing with large quantities of solvents and working the whole process in an enclosed apparatus. The use of activated carbon also involves the recovery of the solvent mixed with water, and it is chiefly suitable for solvents insoluble in water. Its employment requires careful control if fires are to be avoided. Efficiencies up to 100% are theoretically possible. Sulphuric acid was the first liquid absorbent used for the recovery of volatile solvents, but has many disadvantages, and cresol is far to be preferred as an absorbent for alcohol, ether, and esters. These form complexes with the cresol which are decomposed at 130°. In the "Cheminoval" process the vapour-charged air is treated with the absorbent in Feld gas washers. The absorbent passes through a heat exchanger to a heating column, the vapours from which are condensed and redistilled. Recoveries up to 95% are possible under practical conditions, and fire risks are negligible. The method has also been applied to the recovery of benzene, benzol, acetone, etc. C. IRWIN.

Recovery of volatile solvents (Brégeat process). A. HOCK (Inst. Chem. Eng., Dec. 6, 1929, 43—46. Advance copy).—Cresol has been found to be an almost general absorbent for volatile solvents miscible with water. Compounds are formed which are decomposed by heat at a temperature above the b.p. of the solvent, but below that of cresol. A cooling system is required in the scrubber used. This method is extensively used for ether-alcohol vapours, also for acetone etc. Cresol can also be used for solvents immiscible with water, but for such cases Brégeat prefers tetralin. This compound as an absorbent for benzol has the advantage that it does not resinify or increase in viscosity. Its absorptive capacity is greater than that of coal-tar oil. Experiments on the recovery of gasoline from natural gas are also described in which tetralin gave better results than spindle oil or gas oil. C. IRWIN.

Analysis of gases. TRAUTZ and others.—See VII. **Apparatus for determining degree of fineness.** BERG.—See VIII.

PATENTS.

[Boiler] furnaces. AMER. ENG. CO. (B.P. 304,677, 7.5.28. U.S., 24.1.28).—Air is drawn by a fan from the space between the boiler top and roof of the building, then, in a circuitous manner, through spaces between the main side walls of the boiler and outer walls or metal casing, and delivered as forced draught below the firegrate. B. M. VENABLES.

* The remainder of this set of Abstracts will appear in next week's issue.

Drying, heating, or cooling [of materials]. F. J. WARDEN-STEVENS (B.P. 321,169, 31.7.28).—Discrete material is allowed to descend between walls composed of louvres and is subjected to a transverse current of gaseous medium. Means are provided to spread the gaseous medium evenly and to catch any deposited moisture. To assist the descent of the material the walls may be reciprocated vertically and the slats moved pivotally. Transverse stirring rods or tubes are also provided. B. M. VENABLES.

Method of heat exchange, particularly for steam generators. E. C. R. MARKS. From A. T. HERPEN and E. SCHÜTZ (B.P. 321,198, 2.7.28).—A draught up to 250-mm. water gauge is applied to a boiler which has a normal combustion chamber but a heating surface with a resistance to flow of gases much greater than usual. The object is to balance the increased efficiency due to high velocity of gases against the power needed to drive the fan, thus obtaining the maximum net efficiency. Graphs of flue gas velocity against draught resistance and heating surface are exhibited. [Stat. ref.] B. M. VENABLES.

[Emulsions for] prevention of scale formation in steam generators, evaporators, condensers, etc. or the removal of scale therefrom. V. V. SAKS, and FILTRATORS, LTD. (B.P. 321,337, 14.5.28).—An extract of linseed or other oil-bearing material is prepared by first soaking the seed in steam under pressure and then injecting finely-divided water. The apparatus comprises a pressure-resisting container with lid and perforated support for the seed and necessary connexions for steam, water, and emulsion draw-off. It may be attached to the shell of the evaporator or other vessel of which the water is to be treated, the steam being taken from the vapour space and the water from the feed line. B. M. VENABLES.

Humidifying apparatus. Humidifying or conditioning materials. INDUSTRIAL DRYER CORP., Assees. of G. D. HARRIS (B.P. 293,471 and 293,692, [A, B] 7.7.28. U.S., [A] 8.7.27, [B] 9.7.27).—A current of air is circulated through the goods and over a bath of water contained in a shallow compartment below the goods chamber. The supply of heat to the water, or addition of cold water, is thermostatically controlled, and the temperature of the water serves to regulate the temperature and moisture content of the air. [Stat. ref. to (A).] B. M. VENABLES.

Cooler. E. LÜTSCHEN (U.S.P. 1,734,262, 5.11.29. Appl., 22.1.27).—A vertical shell of cylindrical or other shape is surrounded by a slightly larger shell of similar shape, and cooling water is passed through the space between the two. The compressed air, gas, or steam is passed through the inner shell countercurrent to the water, and is caused to impinge on the walls of the inner shell several times by means of a succession of transverse trays which are perforated only through their vertical rims. B. M. VENABLES.

Working fluid for refrigeration. A. A. KUCHER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,735,170, 12.11.29. Appl., 11.8.26).—A combined heat-absorbing and lubricating fluid comprises a mixture of castor oil and sulphur dioxide, which forms a physical

solution or homogeneous mixture in substantially all proportions. B. M. VENABLES.

Air-layer heat and sound insulation. RHEINHOLD & CO. VEREIN. KIESELGUHR- U. KORKSTEIN-GES. (B.P. 315,299, 26.2.29. Ger., 11.7.28).—Foil of comparatively expensive material (e.g., aluminium or nickel) having non-radiating properties is applied to sheets of less expensive material (e.g., tin plate) which act as a support. The foil may be on one or both sides, and may or may not be stuck on. B. M. VENABLES.

Grinding or crushing mills. F. ZWINZ (B.P. 321,501, 17.9.28).—In a disc-grinder both the discs are of the same pattern with a central hole which in the case of the fixed disc is utilised to accommodate the feed chute; this chute also serves to prevent axial and rotational movement of the disc, while permitting, if desired, a certain amount of freedom. The rotating disc is mounted with a certain degree of freedom on the driving shaft by means of a boss in the central hole. B. M. VENABLES.

Machines for separation of granular material. S. A. RAHAUGE and A. W. SIZER (B.P. 321,465, 13.5.29).—A number of overlapping plates are hinged to a conveyor chain, the slope of which is greater at the top than at the bottom. The full-size grains (e.g., of corn) are removed by hinged scrapers, whilst the half grains and dust roll down and pass on over the lower end. B. M. VENABLES.

Air separator [for solids]. T. J. STURTEVANT, Assr. to STURTEVANT MILL CO. (U.S.P. 1,735,479, 12.11.29. Appl., 4.2.28).—The solids are fed into the centre of a double-walled, funnel-shaped casing, the space between the two walls forming a settling chamber. The inner wall comprises upper and lower shells, the former acting as a separator, with an air passage between them which permits the air, which is circulated in the settling chamber, to be deflected up into the separator. The lower shell is provided with a screen-wall, so that the tailings falling from the separator are discharged in two grades. F. G. CLARKE.

Vessel for dissolving fine-grained substances. DORR CO. (G.P. 454,677, 24.8.22).—A circular tank is provided with a centrally-disposed hollow axle carrying at its lower end stirring arms fitted with shovels and with an internal heating coil. A. R. POWELL.

Apparatus for heating liquids in tanks. J. KOZELUH (U.S.P. 1,735,169, 12.11.29. Appl., 7.4.28. Czechoslov., 29.11.26).—A pipe coil is made foldable so that it can be inserted through the manhole, and is caused to lie with a slope as far as possible towards the outlet. B. M. VENABLES.

Washing or treatment of liquids with other liquids [of different sp. gr.]. ANGLO-PERSIAN OIL CO., LTD., A. E. HOLLEY, and O. E. MOTT (B.P. 321,200, 3.7.28).—A countercurrent decantation system suitable for the treatment of petroleum with alkali, acid, and alkali again comprises a number of alternate mixing and settling vessels so arranged that the flow is by gravity, with little or no assistance from pumps. In order to allow the heavy washing liquid to be present in greater ratio than the quantity actually supplied and withdrawn at the ends of the plant, a portion of the heavy liquid

separated in any settler is allowed to flow back to the mixer from which it came. When the lighter liquid passes from an alkaline section to an acid section of the plant additional settlers are inserted. In the mixers the liquids are agitated by rotating stirrers off the centre of the vessel. In the settlers provision is made for withdrawing solid matter collected at the interface of the liquids through an adjustable, rotatable, gallows-like pipe.

B. M. VENABLES.

Filtration of solutions [to render them homogeneous]. I. G. FARBERIND. A.-G. (B.P. 308,278, 20.3.29. Ger., 20.3.28).—The process claimed being one of homogenisation rather than of clarification, the solutions (such as those used for spinning artificial fibres) are continuously circulated from the bottom of a tank through a gear wheel or other type of pump, then through a filter, and back to the upper part of the tank, with a loaded valve-controlled by-pass from the pump to the tank. The tank is preferably tall so that the solution flows evenly downwards through it.

B. M. VENABLES.

Thickener. W. S. ORR, ASSR. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,734,044, 5.11.29. Appl., 5.4.28).—In a thickener with rotating rakes the latter are attached to a sub-truss which is suspended below a main truss or girder to which the power for rotation is applied. The connexion between the two trusses is by bell-crank levers which are operated by connecting rods so that the rakes may be raised without affecting the main girder.

B. M. VENABLES.

Process and apparatus for evaporation purposes. H. P. J. C. GORGEOT, and SOC. D'EXPLOIT. D'USINES MÉTALLURG. (B.P. 321,410, 1.8.28).—The liquid is evaporated in a partial vacuum in the form of a thin film flowing over a heated surface which may be either stationary or moving. The thickness of the film (e.g., 1–2 mm.) is so regulated that the pressure of the vapour bubbles is not substantially above that of the surrounding atmosphere, and the supply of heat is so regulated that no ebullition takes place. [Stat. ref.]

B. M. VENABLES.

Fractional condensation. F. D. FENHAGEN, F. H. RHODES, and T. M. HESSER, ASSRS. to BARRETT CO. (U.S.P. 1,735,455, 12.11.29. Appl., 12.2.21).—A mixture of vapours of substances that are normally solid is subjected to a first condensation by which practically only the highest-boiling constituent is obtained in the solid state, then to a second condensation in which a solid comprising a mixture of highest- and next lower-boiling constituents is obtained; this mixture is then moved countercurrent (by a worm-conveyer) to the vapour mixture, thereby reducing the proportion of lower-boiling constituent.

B. M. VENABLES.

Vapour condensers and scrubbers. A. E. WHITE. From INTERNAT. BITUMEN OIL CORP. (B.P. 321,204, 30.7.28).—A condenser is constructed from a low shell with flat top and bottom and having an internal, spiral guide-strip which makes contact with the top and bottom but is preferably not attached thereto; the convolutions of the spiral are kept apart by distance pieces, and the width of the passage may vary. The

shell may be ribbed for air-cooling or may be submerged in a bath of water. For fractional condensation a number of units are used in series; some may be heat-insulated, others cooled. When used as a scrubber, blocks of absorbent material may be inserted to soak up heavy oil which will absorb condensable vapours.

B. M. VENABLES.

Dust removal from gases. G. R. ATKINS, G. H. C. CORNER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 321,268, 4.9.28).—The gases are passed through counter-current water sprays and then through a series of baffles which are irrigated by water.

B. M. VENABLES.

Liquid and gas contact apparatus. BARTLETT HAYWARD CO., ASSECS. of F. H. WAGNER (B.P. 309,068, 21.11.28. U.S., 4.4.28).—A flat cylindrical chamber has a central gas inlet on the lower side and a corresponding gas off-take on the upper side. A stationary, perforated screen divides the chamber into a central compartment and a surrounding annular space. The central compartment is further divided into an upper and a lower part by a horizontal plate which is capable of rotation about a vertical axis. Nozzles are provided for spraying liquid into the spaces above and below the rotating plate, to which are attached ribbed rods to serve as beaters. Perforated screens extend from the gas inlet and off-take to the rotating plate, and so form the inner wall of the annular chamber through which the gases pass.

A. B. MANNING.

Preparing compressed gases under various controllable pressures from their liquid state. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 297,384, 21.8.28. Ger., 20.9.27. Addn. to B.P. 279,041; B., 1929, 500).—The apparatus described in the prior patent is modified so as to be able to produce pressures in excess of the critical. The gas discharged is led first through a coil exposed to atmospheric heat and then through a coil surrounding, or in, the pressure vessel.

B. M. VENABLES.

Pulveriser. H. WARING, ASSR. to ASSOCIATED LEAD MANUFRS., LTD. (U.S.P. 1,735,985, 19.11.29. Appl., 6.9.27. U.K., 13.9.26).—See B.P. 282,145; B., 1928, 143.

Homogenising mill. W. EPPENBACH, ASSR. to UNITED STATES COLLOID MILL CORP. (U.S.P. 1,738,288, 3.12.29. Appl., 19.7.27).—See B.P. 306,502; B., 1929, 497.

Separation of minerals and other substances. T. M. DAVIDSON (U.S.P. 1,736,111, 19.11.29. Appl., 14.9.26. U.K., 21.9.25).—See B.P. 268,043; B., 1927, 432.

Continuous centrifugal machine. T. LANGENBERG, ASSR. to C. A. FESCA & SOHN (U.S.P. 1,736,349, 19.11.29. Appl., 12.2.27. Ger., 1.10.26).—See B.P. 278,327; B., 1928, 143.

Centrifugal extractor. BRIT. THOMSON-HOUSTON CO., LTD., ASSECS. of C. H. KELSEY (B.P. 302,166, 10.12.28. U.S., 10.12.27).—See U.S.P. 1,689,490; B., 1929, 79.

Compact masses [for filter plates] (B.P. 321,394).—See VII. **Cleaning of gases** (B.P. 307,904).—See X. **Emulsions etc.** (U.S.P. 1,734,975).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Biology and coal research. R. LIESKE (Brennstoff-Chem., 1929, 10, 437—438).—Attention is directed to some investigations in coal and fuel research which also involve biological problems, *e.g.*, those dealing with the occurrence of bacteria in coal, the use of brown coal as a fertiliser, and chemical reactions, such as the oxidation of hydrogen to water, of sulphur or hydrogen sulphide to sulphuric acid, the conversion of carbon monoxide and hydrogen into methane, etc., which can be brought about by bacterial action. A. B. MANNING.

Coal as a material for organic chemical research. W. FUCHS (Brennstoff-Chem., 1929, 10, 438—441).—The concept of "pure coal" is discussed and recent progress in the elucidation of the constitution of the humic acids from brown coal is summarised (cf. Fuchs and Stengel, B., 1929, 771). A. B. MANNING.

Solved and unsolved problems in coal research. F. FISCHER (Brennstoff-Chem., 1929, 10, 444—447).—Recent developments in fuel research, with particular reference to the investigations carried out at the Kaiser-Wilhelm-Institut, Mülheim-Ruhr, are summarised. A. B. MANNING.

Temperature of inflammation of combustible solids. W. SWIENTOSLAWSKI, B. ROGA, and M. CHORAZY (Chim. et Ind., 1929, 22, 676—680).—The temperature of inflammation is defined as that temperature to which the substance together with pure oxygen must be heated to produce a vigorous interaction. Combustions were carried out in an aluminium or iron vessel to which oxygen was admitted by small ports enabling the gas to acquire the temperature of the vessel. Regular temperature readings were taken and a discontinuity on the time-temperature curve indicated inflammation. Results are not affected by the rate of oxygen feed, but are modified by the rate of heating. Tests were carried out with a rate of heating of 10°/min. on material of 2—3 mm. size, and the temperatures found varied from below 200° for some wood charcoals to 658° for Acheson graphite. It is shown that wood charcoal and activated carbon have temperatures of inflammation increasing regularly with the temperature of carbonisation and activation, respectively. C. IRWIN.

Determination of water content of solids, *e.g.*, brown coal. K. FISCHBECK and E. EINECKE (Z. Elektrochem., 1929, 35, 765—769).—The new and rapid method described, requiring about 5 min., depends on the ratio of water existing in the coal and in a definite amount of glacial acetic acid with which it has been shaken. About 20 g. of brown coal are shaken for 2—3 min. with 20—40 c.c. of glacial acetic acid; 2 c.c. of the liquid phase are transferred to a flask and distilled into a small receptacle containing two platinised platinum electrodes, about 1 mm. apart. The electrical resistance is measured and the water content ascertained from calibration tables. H. T. S. BRITTON.

Analysis of Iowa coals. H. L. OLIN, R. C. KINNE, N. H. HALE, and J. H. LEES (Bull. Iowa Geol. Survey, 1929, 19 pp.).—Mean results (dry) are: ash 13.6, volatile matter 42.0, fixed carbon 44.4, sulphur 4.8%;

thermal value 12,045 B.Th.U., unit coal 14,555 B.Th.U.; mean f.p. of ash 1108°. CHEMICAL ABSTRACTS.

Dirt in coal: its influence on carbonisation. W. E. PLEVIN (Gas J., 1929, 188, 653—654).—Coal from two pits was cleaned separately at the same plant and the refuse obtained was carbonised in an experimental retort holding 2.24 lb., and was also analysed. From colliery A the material contained 75.13% of ash, 15.08% of volatile matter, and 8.49% of fixed carbon; on carbonisation it gave 4450 cub. ft. of gas, or 16.46 therms per ton, the gas containing chiefly hydrogen and carbon oxides. The refuse from colliery B had 67.21% of ash, 16.84% of volatile matter, and 14.59% of fixed carbon; it gave 21.16 therms per ton, with a very similar gas analysis. Calculations are made which show that both water-gas and carbonisation gases are produced in heating these substances, and attention is called to the high sulphur content of the products. R. H. GRIFFITH.

Influence of added kaolin and graphite on the degree of swelling of coke obtained in determinations of volatile matter in coal. D. J. W. KREULLEN (Chem. Weekblad, 1929, 26, 547—548).—The height and volume of the cokes obtained increase to a maximum with increasing additions, and then begin to diminish. S. I. LEVY.

Low-temperature carbonisation of Moscow coal. A. P. SCHACHNO and I. B. RAPOPORT (Brennstoff-Chem., 1929, 10, 457—461).—Five South Moscow coals, including one boghead coal, were carbonised at 500° in the Fischer-Schrader aluminium assay apparatus. The cokes produced were pulverulent and of high ash and sulphur content; they were, however, with the exception of that from the boghead coal, of lower sulphur content and higher calorific value than the coals. The high yield of tar (45%) from the boghead coal made this particularly suitable for treatment by low-temperature carbonisation. From the other coals only a small yield of tar (10% of the ash-free, dry coal) of low value was produced. The gas from the boghead coal was of high calorific value, and could be used for heating, lighting, or enriching poor gases. The coals began to decompose at about 250°; they could be dried at any lower temperature without undergoing change. A. B. MANNING.

Industrial application of active carbon. K. EVANS, H. F. PEARSON, and E. REISEMANN (Inst. Chem. Eng., Dec. 6, 1929, 62—83. Advance copy).—In the Bayer process for the manufacture of active carbon, a paste formed of peat, sawdust, etc. with zinc chloride is pressed into rods which are broken and calcined in a rotary furnace. The Urbain process employs phosphoric acid, which on ignition at 1200° is reduced to phosphorus which distils over and is burned. Air activation at 350—450° or steam activation at 800—1000° in vertical retorts may also be used. The Carbo-Union (Bayer) solvent-recovery apparatus is described in detail. The requirements of the plant (fitted with steam economiser) are about 3 kg. of steam, 40 kg. of water, and 0.2 kw.-hr. of current per kg. of solvent recovered. This is for operation with a minimum of loss. Carbon consumption is taken as 1 kg./ton of recovered

product. The steam etc. for rectification is additional. The economiser is an evaporator working on the ejector-vacuum principle. The carbon loss will be greater in the case of gases containing sulphur. A transportable gasoline-recovery plant is described. Recovery plants are also employed for benzol recovery in the water-proofing industry. As they operate at low concentrations (15–20 g./m.³) they do not offer any additional fire risk. An 85–92% recovery is possible. A similar application is in the manufacture of artificial leather cloth, and a further one is for acetone recovery in the acetate silk industry. For gas-mask purposes the essential quality in a carbon is high capacity for gases at low concentrations, and the best carbon for the higher concentrations used in industry is not necessarily suitable. Some active carbons have catalytic properties which bear no definite relation to adsorptive capacity. These are employed in the desulphurisation of towns' gas. Sulphur separates out in the capillaries and is extracted with ammonium sulphide solution, which is afterwards boiled off. An air content of 3–4% and an ammonia content of 0.3 g./m.³ in the gas is necessary. It is considered that this process can compete with dry purification in large works. It is actually in use for the desulphurisation of water-gas for ammonia synthesis. A further application of active carbon is for the removal of offensive odours both of industrial origin and in halls etc.

C. IRWIN.

Adhesion tension values of different types of carbon black against water and against benzene. F. E. BARTELL and C. N. SMITH (Ind. Eng. Chem., 1929, 21, 1102–1106).—The adhesion tensions for seven different types of carbon black (after preliminary heat treatment and evacuation) against water and benzene were determined by the displacement method. The adhesion tensions observed are shown to vary with the nature of the black and with the preliminary heat treatment, the influence of crystalline structure, adsorbed impurities, and porosity being also discussed. It was found that reliable data could be obtained only when the carbon particles were so large that compressed membranes of them had pore radii greater than 2×10^{-6} cm.

S. S. WOOLF.

Modern apparatus for the detection of gases and vapours in air. G. SCHMITT (Brennstoff-Chem., 1929, 10, 424–426, 461–464).—The following types of apparatus for the detection of inflammable and other gases and vapours in air are briefly described: (1) Redwood's apparatus (cf. B.P. 187 of 1893; B., 1893, 375), (2) the Union recording apparatus (cf. B.P. 148,764; B., 1922, 238 A), (3) the Zeiss mine-gas interferometer, (4) the Vulkan gas testers, and (5) the apparatus recently designed by Williams and Johnson, of the Standard Oil Co., for the detection of petrol vapour in the air. The action of the Vulkan gas testers depends on the relative rates of diffusion of the different constituents of a gas through a membrane, and the resultant changes of pressure in the diffusion chamber. In the Standard Oil Co. apparatus a heated platinum wire brings about the combustion of the petrol in the air passing over it, and the change in the resistance of the wire due to its change in temperature gives a measure of the amount of inflammable vapour present.

A. B. MANNING.

[Gas] interferometer as an aid to factory control. H. A. J. PIETERS and J. A. MEYLINK (Chem. Weekblad, 1929, 26, 568–570).—A rapid and reliable method of determining crude benzene content of coke-oven gases with the aid of a simple gas interferometer is described. The apparatus is calibrated by employing purified gas, to which varying known quantities of the crude benzene extracted by means of adsorbent charcoal are added.

S. I. LEVY.

Recovery of benzol from coal gas, with particular reference to the use of active charcoal. H. HOLLINGS, S. PEXTON, and R. CHAPLIN (Inst. Chem. Eng., Dec. 5, 1929, 14–33. Advance copy).—Works-scale experiments carried out by the Gas Light and Coke Co. and extending over the last six years are described. The first experimental plant treated gas freed from hydrogen sulphide, containing 100 grains of benzol per 100 cub. ft., and saturated with water vapour and naphthalene at 15°, at a rate of 50,000 cub. ft./hr. A prefilter for naphthalene removal was used. The filter contained 2400 lb. of activated charcoal "A-Kohle-T." During 110 saturations and steamings a 70% benzol recovery was obtained; the charcoal lost 55% of its activity and increased 31% in weight. This depreciation would have rendered the process uneconomical. There was also a serious loss in the prefilter charcoal. It was therefore decided to remove naphthalene by oil-washing and conduct a laboratory investigation of the causes of charcoal depreciation. This showed that all types of charcoal deteriorate similarly under similar conditions, but that the fouling of the charcoal is reduced by passing the steam for distillation through in the opposite direction to that of the gas flow, and by turning on the steam supply to the cold, saturated charcoal at the commencement of the heating period. A larger plant was then constructed embodying these principles. Tests of this, though as yet incomplete, show an extension of the useful working life of the charcoal. The rate of depreciation, is, however, still considerable. The benzol freshly produced was used without trouble in motor-car engines, but gumming occurred after storage. The degeneration of the charcoal is itself due to the deposition of a gum within its capillaries. A considerable body of evidence is given which is consistent with the view that in each gas the presence of hydrogen cyanide is necessary to the formation of gum. Experiments using gas freed from hydrogen cyanide showed a definite but much slower rate of depreciation of charcoal, but no gum formation. Benzol obtained under those conditions did not resinify. When treated with aqueous hydrogen cyanide it suffered a slight reduction in mol. wt. and showed resinification. A method for the accurate determination of absorptive capacity of charcoal for benzol is described.

C. IRWIN.

Recovery of ammonia in gas manufacture—indirect, semi-direct, and direct processes. A. PARKER (Gas J., 1929, 188; Inst. Gas Eng. Suppl., 37–41. Cf. Hollings and Pexton, B., 1929, 630).—The costs of the indirect, semi-direct, and direct processes, together with their respective advantages, are examined. It is shown that (1) the direct and semi-direct processes are not so flexible because ammonium sulphate is their only product, whereas by the indirect

process sulphate, concentrated ammonia liquor, anhydrous ammonia, and certain other products may be obtained; (2) it is easier to produce sulphate of uniformly high quality by the indirect process; (3) if the power costs consumed in tar-fog removal from the gas before it enters the saturators of the direct and semi-direct processes, and other costs, are taken into account, there is no financial advantage in either process over the indirect process; if, however, the satisfactory disposal of effluent liquor should necessitate the removal of tar fog from the gas before the liquor condenses, then the direct process would be the cheapest; (4) the direct and semi-direct processes as operated in the coking industry would require modification for successful application to gas works, where the carbonisation units are usually much smaller; (5) it is possible to reduce the costs of the indirect process by utilising a concentrated virgin-liquor, thereby reducing distillation costs; this would require modifications in the existing condensing and scrubbing systems.

C. B. MARSON.

Liquid purification of coal gas by ammonia. E. W. SMITH (Gas J., 1929, 188; Inst. Gas Eng. Suppl., 41–42).—The capital and operating costs of liquid-purification processes using ammonia are examined, and it is shown that these are greater than the average costs for iron oxide box-purification; it is concluded that there is no likelihood of ammonia liquid purification replacing the present solid oxide box method.

C. B. MARSON.

Use of [coal-tar] creosote in the manufacture of carburetted water-gas. F. J. DENT (Gas J., 1929, 188; Inst. Gas Eng. Suppl., 21–27).—Results obtained in large-scale tests indicate that when using a light creosote in place of gas oil for carburetting, other conditions on the plant being unaltered, the thermal yield of oil gas obtained is 0.5–0.7 therm per gal., or about one third to one half of the yield usually obtained from a petroleum gas oil. Mixtures of light creosote and gas oil containing not more than 10% of creosote have been used continuously for three months, and no difficulties due to the deposition of naphthalene in the plant have been experienced, but there is no evidence to show that the thermal yield of gas obtained from such a mixture is greater than that given by the contained gas oil. The constitution of coal-tar creosote and its thermal decomposition are discussed, and methods of testing its value for carburetting water-gas are described.

C. B. MARSON.

Fractional adsorption of gases. J. S. MORGAN (Inst. Chem. Eng., Dec. 6, 1929, 47–52. Advance copy).—The first attempt to separate gas mixtures by the use of activated charcoal was made with the object of separating ethylene from coke-oven gas in 1918. Earlier experiments used a static plant in which the charcoal was stripped by heating to 300–400° by heated gas and similarly cooled. The requirements of gas for this were enormous. Dynamic systems in which the charcoal is moved halve the heat transfer required, as there is no container to be heated or cooled, and they also permit of fractionation. Such a system has now been devised in which the heating and cooling are effected by the addition of hot or cold iron shot, and, since

granulated charcoal would crumble, this is used in powdered form. An arrangement of inclined louvres regulates the downward course of the charcoal, and moving bands of flannel filter the gas from dust. Heating and cooling are so rapid that the effective capacity of the charcoal is greatly increased. A fractionating column is made up of alternately superimposed heaters and absorbers.

C. IRWIN.

Electrical treatment of gases. K. PETERS (Brennstoff-Chem., 1929, 10, 441–444).—A summary is given of those gas reactions which can be brought about by different types of electrical discharge. In particular, the conversion of methane and other hydrocarbons into acetylene, and of carbon dioxide into carbon monoxide and oxygen, by the action of an electrical discharge under reduced pressure are briefly discussed (cf. Fischer and Peters, B., 1929, 703).

A. B. MANNING.

Free carbon formation in coal tars and pitches. W. G. ADAM and J. S. SACH (J.S.C.I., 1929, 48, 337–341 r).—An attempt has been made to distinguish between the true suspended matter, C_1 , and the bituminous resin-like compounds, C_2 , precipitated by benzene, toluene, and like solvents from coal tars and pitches. Determinations of insoluble matter in coal tars and pitches, using various solvents, have been made, as a result of which pyridine is recommended for the direct determination of C_1 , and benzene or toluene for C_1+C_2 . The effect of time and temperature factors during the distillation process on the C_1 and C_1+C_2 content of tars and pitches has been investigated, and it has been shown that after passage through a coil still (max. temp. 270°) the ratio of the total insoluble matter (C_1+C_2) in the treated tar to that in the original tar is substantially unity. Distillation to pitch in pot stills, whilst effecting very little change in the total C_1 content, gives rise to formation ratios for C_1+C_2 of 1.26, 1.71, and 2.31 for horizontal-retort, vertical-retort, and coke-oven tars respectively, as compared with 1.54 for coke-oven tar distilled in a continuous plant. Autoclave experiments show that time and temperature factors are increasingly important at temperatures above 300°. Small-scale distillations on various tars confirm the above figures, and further demonstrate the very low stability of chamber oven and low-temperature tars, and in the latter case it appears that the formation of C_2 during distillation is attributable to the alkali-soluble fraction. Factors favouring the reduction of “free carbon” formation during distillation are given, chief among them being rapid distillation at minimum temperatures by employment of continuous methods, combined with vacuum or free steam distillation. The formation ratio, however, is largely dependent on the history of the tar prior to distillation, particularly in regard to the temperature employed in the carbonising process. The paper is illustrated by photomicrographs.

4-Methylindole in coal tar. O. KRUBER (Ber., 1929, 62, [B], 2877–2880).—The residues from the distillation of indole fractions, b.p. 265–269°, which remain unattacked after removal of 7- and 5-methylindoles by sodium (cf. A., 1927, 157) are treated with sodium and carbon dioxide at 190°, whereby skatole remains unaffected and a mixture of sodium salts is

obtained from which 4-methylindole-3-carboxylic acid, m.p. 199° (decomp.), is isolated. Distillation of the acid affords 4-methylindole, b.p. 267°, m.p. 5°, d_4^{20} 1.062 (picrate, 194—195°). The constitution of the compound is determined as follows. 4-Methylindole-3-carboxylic acid is reduced by sodium and alcohol to 4-methyl-2 : 3-dihydroindole-3-carboxylic acid, m.p. 223°, decarboxylated to 4-methyl-2 : 3-dihydroindole, b.p. 245°, 124—126°/12 mm., d_4^{20} 1.038 (picrate, m.p. 188°; hydrochloride, m.p. 235°; benzenesulphonyl derivative, m.p. 135°; benzoyl compound, m.p. 117—118°). 1-Benzoyl-4-methyl-2 : 3-dihydroindole-3-carboxylic acid, m.p. 115°, is oxidised by potassium permanganate in the presence of magnesium sulphate to 3-benzamido-*o*-phthalic acid, m.p. 186—187°, identical with a specimen prepared from 3-nitrophthalic acid. The liquid nature of the monomethylindole fractions appears to be largely attributable to the 4-methyl compound. H. WREN.

Cracking of tars from cannel coal. J. C. MORRELL and W. F. FARAGHER (Ind. Eng. Chem., 1929, 21, 1084—1086).—A crude cannel-coal tar derived from Kentucky cannel coal was cracked under 125 lb./in.² pressure and produced without recycling 76.8% of pressure distillate and 23.2% of coke and gas. The pressure distillate contained 14% of tar acids, and yielded 42.1% of tar acid-free motor fuel of 225° end-point. The remaining distillate can be recycled and made to yield more motor fuel. Refining of the pressure distillate follows normal procedure with losses of 2.5%. The motor fuel possesses good anti-knock properties, and the pressure distillate bottoms can be used as furnace oils or distilled for special products, e.g., naphtha, solvents, turpentine substitutes.

H. S. GARLICK.

Crystallisation of paraffin. IV. Paraffin crystals from low-temperature coal tar. Y. TANAKA, R. KOBAYASHI, and I. ARAKAWA (J. Fac. Eng. Tokyo, 1929, 18, 109—115).—A low-temperature coal tar was distilled, and the fraction of b.p. 300—350° was collected and cooled to 10°. The crude paraffins which separated were purified by washing with alcohol-ether and by sweating at 58—59°; the substance finally obtained had d_4^{25} 0.7762, n_D^{25} 1.4288, and m.p. 58.7°, and consisted mainly of normal paraffins. Rhombic plates having angles of 110° and 70° were obtained by slow crystallisation from very dilute solutions, and other derived forms were prepared by using higher concentrations (cf. B., 1929, 118). The variation of the crystal shape with the nature and amount of the solvent is discussed in comparison with results obtained with other paraffins, and an equation showing the mutual effects of viscosity and concentration has been derived from that of von Weimarn.

R. H. GRIFFITH.

Solvent extraction in acetic acid production [from pyroligneous acid]. N. W. KRASE (Chem. Met. Eng., 1929, 36, 657—659).—The Suida process, as operated at one American works, effects extraction with wood-tar distillate of b.p. range 200—340°. A copper primary still and a bubbling-hood scrubbing column are used. The enriched oil containing 5% of acetic acid and a little water is passed first through a dehydrating column fitted with steam coils and then,

after heating, down a vacuum plate-column to a copper-tube boiler, where stripping is completed. The acetic acid vapours are fractionated in another column, and pass to aluminium-tube condensers. The oil becomes more viscous with use, and is changed every 3 weeks. The efficiency of acetic acid recovery is 95—96%, and the steam consumption 0.565 lb. of steam per lb. of feed-liquor.

C. IRWIN.

Refining of shale gasoline. I. Relation of oxidation to colours and gums produced in gasoline from Colorado oil shales. R. A. BAXTER (Ind. Eng. Chem., 1929, 21, 1096—1098).—Samples of gasoline derived from Colorado shale oil were exposed to sunlight, X-rays, and ultra-violet light. Samples that had been exposed to air darkened and formed gums in each case. When ozone is passed into shale gasoline a dense white cloud is evolved and the liquid gets hot, a reaction noted only for compounds with multiple linkings. Ozone removes the gum-forming constituents without greatly decreasing the concentration of desirable, stable, unsaturated compounds in the gasoline by converting them into gums which may be eliminated by centrifuging, light acid treatment, or extraction with methyl alcohol.

H. S. GARLICK.

Chemistry of gum formation by cracked gasoline. LER. G. STORY, R. W. PROVINCE, and H. T. BENNETT (Ind. Eng. Chem., 1929, 21, 1079—1084).—In the dish test for gum formation, the quantity of oxygen in the atmosphere is the determining factor in the amount of gum produced, and explains the difficulty in obtaining consistent results. Evaporation in an atmosphere of steam gives low results, since the test gives only the gum already in solution, and not the potential gum-forming ability. A copper dish gives high results due to the catalytic effect of copper oxide. A low-volatility gasoline gives a longer period for the reactions producing gum to take place. During the evaporation of untreated cracked gasoline, peroxide, aldehydes, and acids were detected, the increase in the last-named in a copper dish being considerable. Acid treatment of the gasoline causes a pronounced decrease in acidity development. An examination of a quantity of gum formed in a copper dish showed that it was completely soluble in acetone, alcohol, and chloroform, and had m.p. 98—105°, b.p. 164—169° without decomposition. It could be separated into 13% of unsaponifiable matter, 55% of water-insoluble acids, and 30.5% of water-soluble acids. The constants for each fraction were determined. Cracked gasoline rapidly absorbs oxygen when exposed to sunlight, during which time the oil becomes cloudy and deposits a yellowish gum. A theory of gum formation is offered involving primary autoxidation.

H. S. GARLICK.

Variation in the temperature of spontaneous ignition of liquid fuels containing added substances, as a function of the composition of the mixture. A. GREBEL (Compt. rend., 1929, 189, 856—858).—Data are given showing the increase in the temperature of spontaneous ignition of petrol resulting from additions of benzol, ethyl alcohol, or of both these substances. Similar data are given for additions of acetone and aniline up to 5% to a mixture containing

petrol 50%, benzol 30%, absolute alcohol 20%. The mixtures investigated are all stable down to normal winter temperatures, and the prices are not economically prohibitive. C. J. SMITHELLS.

Variation of surface tensions of lubricating oils with temperature. G. WINCHESTER and R. K. REBER (Ind. Eng. Chem., 1929, 21, 1093—1096).—The surface tensions of eight oils differing considerably in general physical characteristics were determined at intervals of 20° between 80° and 300° by the method of maximum bubble pressure (cf. Cantor, Wied. Ann., 1892, 47, 399). For each oil the surface tension varies from about 25—26 dynes at 100° to about 13—14 dynes at 300°, and although the oils differed in other properties, their surface tensions were about the same at equal temperatures. Both the density and the surface tension varied linearly with the temperature. H. S. GARLICK.

Solubilities of [lubricating] oils and waxes in organic solvents. J. W. POOLE (Ind. Eng. Chem., 1929, 21, 1098—1102).—The solubilities of a range of petroleum oils and paraffin waxes in various organic solvents were examined qualitatively, and quantitative solubility determinations were also carried out, using as solvents acetone, butyl alcohol, a mixture of these two, ethyl acetate, butyl acetate, chlorobenzene, and toluene. The results are graphed and discussed, and it is indicated that butyl alcohol offers the best possibility of application to commercial extraction processes in this connexion. S. S. WOOLF.

Alleged catalytic action of fuller's earth on colouring matter in oils. J. D. HASEMAN (J. Physical Chem., 1929, 33, 1514—1527).—Fuller's earth does not bleach oils by catalysis or by selective adsorption, but by virtue of a silicic acid which precipitates the colours; these are of an azo-humic nature. Porosity, plasticity, hardness, particle size, etc. have no direct effect on bleaching power other than to facilitate the contact of hydrogen ions from the silicic acid with the colouring matter. Fuller's earth is activated in the region of the underground water level mainly by sulphuric acid, and revivification consists in the removal of alkali. The origin of fuller's earth and the nature of the colouring matter in oils are discussed.

L. S. THEOBALD.

Recovery of volatile solvents. BODEWIG; HOCK.—See I. **Purification of anthracene.** RABEK and BOJANOVSKI.—See III. **Fuller's earth.** SCHOLZ.—See VII. **Mobility-concentration curve of lubricating oil.** BALDWIN.—See XII.

PATENTS.

Washing of coal. M. JUNG (B.P. 292,487, 18.6.28. Ger., 24.10.27).—After separation of the fine coal from the shale and dirt in a jig, the bulk of the washing water is separated from the coal by screening and the coal is then further treated in a centrifugal separator to remove the remaining water. The washing water from a coarse coal jig may be similarly treated. By keeping the water in motion during the process the suspended, finely-divided, inorganic matter is prevented from resettling on the coal. Any fine coal remaining in the washing water is recovered by pumping this to a

settling tank and treating the settled matter in a second centrifugal separator. A. B. MANNING.

Drying and pulverising of coal. F. I. BARROWS, Assr. to R. H. BARROWS (U.S.P. 1,734,992, 12.11.29. Appl., 18.7.27).—Before grinding, the coal is partially disintegrated by heating it in an enclosed chamber at reduced pressure. The grinding is then completed in a mill situated below this chamber. F. G. CLARKE.

Distillation of coal. (SIR) G. C. MARKS. From CHEM.-TECHN. GES.M.B.H. (B.P. 321,283, 26.9.28).—The coal is continuously and simultaneously forced through several distillation chambers which are separated by heating chambers or heating bars, and are only a fraction of their height in length, so that the coal has to travel only a short distance during the distillation process. The distillation chambers are subdivided in a grid-like manner by means of horizontal metal plates. The chambers may be arranged radially round a central space in which the rolling of a cylinder mounted eccentrically forces the coal under pressure through the chambers, or the coal may be forced through the chambers towards the axis by the rotation of two co-operating, drum-like, distillation-chamber systems. In another arrangement the coal is forced through the chambers by means of a plate moving to and fro.

A. B. MANNING.

Coking process. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,733,307, 29.10.29. Appl., 5.7.21. Renewed 12.3.25. Ger., 28.12.17).—The carbonisation of the charge in an externally-heated coking retort oven is continued until the greater part of the distillation is completed; during the remainder of the coking, air and steam are passed alternately over the charge. A. B. MANNING.

Treatment of carbonaceous substances under pressure for production of valuable liquid products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,918, 21.5.28).—Carbonaceous substances which are to be extracted, hydrogenated, or otherwise treated under pressure are first dehydrated by distilling off the water in the presence of an organic liquid, e.g., a high-boiling mineral or other oil. The dehydration may be carried out under reduced or increased pressure, or in a current of scavenging gases. A. B. MANNING.

Manufacture of highly-activated adsorptive carbons. N. V. NORIT-VEREENIGING VERKOOP CENTRALE (B.P. 309,855, 25.7.28. Ger., 16.4.28).—The product formed by the carbonisation of the raw material is finely divided, agglomerated, e.g., by compression after the addition of a binding agent, and activated, first by steam or other activating gases, and finally by ignition after impregnation with activating substances, e.g., phosphoric acid, phosphates, zinc chloride. The carbon may be extracted with solvents before or after its impregnation and ignition. By suitably controlling the activating processes the adsorptive capacity and retentivity can be varied as desired. A. B. MANNING.

Gas producer. R. DAAE (U.S.P. 1,735,380, 12.11.29. Appl., 24.5.24).—The cover and ash-pan of a cylindrical gas producer are geared circumferentially and each has a unit driving mechanism. Each mechanism is supplied

with a reservoir for lubricating fluid. An operative connexion is provided between the two unit mechanisms, which can be bodily removed from driving relationship with the cover and ash-pan when desired.

A. B. MANNING.

Vertical retorts for the production of gases. BAMAG-MEGUIN A.-G. (B.P. 307,478, 31.7.28. Ger., 10.3.28).—A vertical retort has a lower seal through which the degasified material can be discharged, and is provided with an upper and a lower gas outlet. The latter leads from a channel passing round the retort within the lower rim of the base-plate supporting the structure. This channel is provided with a circumferential opening facing the retort; the gas passes up through this into the channel and thence to the outlet. The channel may be cooled by periodically admitting steam through it to the retort. In addition to the direct-sealing cover at the lower end of the retort a supplementary liquid seal may be provided.

A. B. MANNING.

Manufacture of carburetted water-gas. HUMPHREYS & GLASGOW, LTD., and L. D. CARROLL (B.P. 320,852, 22.6.28. Cf. B.P. 240,665; B., 1925, 949).—The gas leaving the generator is divided into two streams, one of which passes through the carburetter and is enriched with oil before entering the holder, while the other passes directly to the holder. A. B. MANNING.

Manufacture of and apparatus for using fuel rich in methane for internal-combustion engines. J. I. BRONN, and CONCORDIA BERGBAU A.-G. (B.P. 320,863, 21.7.28).—A fuel gas of high calorific value, and permitting the use of a high compression ratio in the engine, consists principally of methane, with at least 10% of other hydrocarbons gaseous at the normal temperature and pressure, but with not more than 5% of other components. The methane may be derived from coke-oven gas, and the other components from gases obtained by the low-temperature carbonisation of coal or by the cracking of crude oil. If the fuel is stored under high pressure a separate pressure container is interposed between the storage vessel and the engine, with pressure regulators before and after the intermediate container, in order to expand the gas in stages and ensure a uniform supply to the engine. The fuel may also be stored in the liquid form under lower pressures and at low temperatures in heat-insulated containers; these are provided with a safety valve which automatically opens when the pressure becomes unduly high and conveys the out-flowing gas to a safety lamp.

A. B. MANNING.

Manufacture of acetylene [from calcium carbide] and apparatus therefor. A.-G. F. STICKSTOFFDÜNGER (B.P. 306,042, 13.2.29. Ger., 14.2.28).—Acetylene is prepared from calcium carbide and water under such conditions that the heat evolved during the reaction is absorbed by the evaporation of such a quantity of water that the temperature in the generator does not rise above 100°, whilst a substantially dry calcium hydroxide is produced. The materials are kept continually in motion during the process so that the carbide is mixed thoroughly and uniformly with the hydroxide produced. The apparatus comprises a number of plates fixed in steps one above the other, stirring arms

for conveying and mixing the materials, and inlet nozzles for spraying them with water.

A. B. MANNING.

Manufacture of asphalt. L. KIRSCHBRAUN, Assr. to F. L. BELKNAP (U.S.P. 1,735,503, 12.11.29. Appl., 27.1.23).—Petroleum residuum is mixed with steam and passed through a heating device of narrow cross-section wherein the temperature of the mixture is gradually raised and the superheated steam is brought into intimate contact with the residuum. The mixture is continuously introduced into a bulk supply of the end-product through a number of jets arranged below the surface of the liquid. The volatile constituents are led away and separately condensed, whilst the finished product is withdrawn from the bulk supply as required.

A. B. MANNING.

Bituminous emulsions. COLAS PRODUCTS, LTD., and L. G. GABRIEL (B.P. 320,847, 22.5.28).—Bitumen is emulsified in water with so small a proportion of emulsifier, e.g., oleate soap equivalent to 0.2–0.45% of oleic acid calculated on the bitumen, that the size of a substantial proportion of the dispersed bitumen particles is considerably greater than that of the particles in a stable emulsion; a solution of a stabilising agent, e.g., gelatin, is then added. Such an emulsion remains stable during storage and transport, but breaks readily when applied to a mineral aggregate or road surface.

A. B. MANNING.

Production of a bituminous composition. J. A. MONTGOMERIE (B.P. 321,334, 17.11.28; cf. B.P. 105,202; B., 1917, 552).—Bitumen is emulsified in water containing colloidal clay and magnesium oxide in amounts equal, respectively, to 5% and $\frac{1}{2}$ % by wt. of the bitumen.

A. B. MANNING.

Treatment of oil wells to enhance their productivity. A. H. ACKERMAN, Assr. to CATALYTIC CHEM. Co. (U.S.P. 1,734,990, 12.11.29. Appl., 31.12.26. Renewed 15.4.29).—The reaction products obtained by adding anthracene to sulphuric acid, nitrobenzene, naphthalene, and caustic soda are introduced into the wells.

F. G. CLARKE.

Oil still. C. L. PARMELEE (U.S.P. 1,736,325, 19.11.29. Appl., 19.7.23).—The apparatus consists of a vaporising chamber, the lower part of which is divided into two separate compartments, and the upper portion contains means for fractionating vapours from both compartments and is connected to condensers. From one of these compartments oil is pumped through a conduit surrounded by a furnace and back to the second compartment; overflow from this is conveyed directly to the first and recycled.

W. S. E. CLARKE.

Apparatus for clarifying and decolorising petroleum oil. J. C. BLACK, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,734,959, 12.11.29. Appl., 2.8.27).—Regulated quantities of oil and decolorising agent are introduced into a treating tank, circulated from the tank through a heater, thence to a separate cooler selectively, and are returned to the treating tank. The oil and the agent are separated by means of a filter-press.

W. S. E. CLARKE.

[Tubes for] treatment of hydrocarbons. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 292,535,

16.6.28. U.S., 21.6.27).—Carbonaceous materials and metals, when wet with water, will take a liquid hydrocarbon coating on treatment with oil. Earthy materials such as porcelain and non-metallic paints have a greater affinity for water, and the coating of water cannot be displaced by oil. Condenser and oil-cracking tubes are therefore lined with porcelain or earthy material to prevent the formation of carbonised coatings (cf. B.P. 159,497; B., 1922, 243 A). Better heat transfer is obtained through the porcelain and water or water-vapour cushion than would be obtained from a metal tube with a carbonised lining, and the tubes keep cleaner.

T. A. SMITH.

Refining of hydrocarbons. F. A. APGAR, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,735,988, 19.11.29. Appl., 10.3.28).—In refining hydrocarbon vapours by means of an adsorptive catalyst, the vapours are separately condensed while the catalyst is being renewed, and are subsequently passed through the latter during the high-activity stage on re-commencing the normal flow of the vapours.

F. G. CLARKE.

Refining of hydrocarbons. A. C. VOBACH, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,736,234, 19.11.29. Appl., 29.2.28).—In operations where cracking and vapour-phase refining by means of adsorptive catalysts are combined, the vapours from the cracking operation are submitted to a double fractional condensation before treatment with the contact material. The heaviest fractions are not returned to the cracking operation, but the lighter fractions which are still too heavy to be included in the gasoline are added to the cracking stock. The high-boiling polymerides produced from the vapours in contact with the catalyst are separately removed and fractionated. Only the lighter constituents of the polymerised fraction are returned to the cracking operation.

T. A. SMITH.

Refining of hydrocarbons. T. DE C. TIFFT and A. C. VOBACH, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,736,022, 19.11.29. Appl., 10.2.28).—Hydrocarbon vapours, before being passed over adsorptive catalysts, are subjected to fractional condensation, whereby the heavier fractions are removed. Polymerised products formed by the reaction condense on the catalyst, and the purified vapours are passed to a condenser. Light products contained in the polymerised portion are recovered by passing through a heat exchanger, where they are heated by the condensate from the preliminary fractionating column.

T. A. SMITH.

Treatment [oxidation] of hydrocarbons. P. L. YOUNG, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,735,486, 12.11.29. Appl., 4.8.28).—A relatively cool mixture of a hydrocarbon and oxygen is passed into a reaction zone, and the oxidation initiated by introducing a heated hydrocarbon. Liquid oxidation products are withdrawn.

F. G. CLARKE.

Treating hydrocarbon distillates and apparatus therefor. C. W. STRATFORD (U.S.P. 1,736,018, 19.11.29. Appl., 26.11.26).—Oil is circulated in a continuous stream through and around a hollow cylinder in a closed container. It is introduced into the vortex of the circulating fluid and an approximately equal amount is withdrawn by uniformly stripping the liquid at the peripheral

upper surface of the circulating stream. A treating liquid is added to the circulating stream.

W. S. E. CLARKE.

Reclaiming lubricating oils. O. J. NELSON, ASSR. to BRYTE-NELSON REFINING Co. (U.S.P. 1,735,440, 12.11.29. Appl., 8.1.23).—The used mineral lubricating oil, partially emulsified and polymerised, after defecation to remove solid matter and entrained water, is simultaneously heated in a still gradually to the vaporising point of the mineral lubricant base and blown first with air and then with steam until the remaining moisture and lighter hydrocarbons are volatilised. The latter are collected and condensed and the residue is filtered.

W. S. E. CLARKE.

Treatment of petrogen-containing substances. J. B. JENSON (U.S.P. 1,734,970, 12.11.29. Appl., 22.7.21).—Shale is heated in a bath of molten metal in the presence of hydrogen, whereby fully-saturated petroleum products are obtained. The depth of the bath is such that the shale and gases are subjected to pressure, the maximum pressure being exerted at the bottom of the bath until the shale is decomposed. The spent materials are then released and allowed to ascend quickly to the top of the bath; the resultant saturated hydrocarbon gases are collected and condensable constituents condensed.

W. S. E. CLARKE.

Manufacture of non-knocking engine fuels of the benzene type by destructive hydrogenation. I. G. FARBERIND. A.-G. (B.P. 296,984, 22.8.28. Ger., 10.9.27).—The properties of the products of destructive hydrogenation vary considerably with the initial material used. The hydrogenation of coals and tars yields spirits of high anti-knock value, but deficient in constituents boiling before 100°. Similarly, many petroleum products on hydrogenation yield products of low b.p., but also of low anti-knock value. Good engine fuels having good b.p. curves, high anti-knock value, and containing naphthenes and olefines are therefore best prepared by the hydrogenation of suitable mixtures of raw material. A 3:1 mixture of American crude oil and coal tar on hydrogenation at 200 atm. and 460° yields a non-knocking fuel, whilst the crude oil alone, similarly treated, yields a spirit to which anti-knock material has to be added.

T. A. SMITH.

Motor fuel. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES, Assees. of DEUTS. BERGIN-A.-G. F. KOHLE U. ERDÖLCHEMIE (F.P. 630,326, 5.3.27. Ger., 20.11.26).—Crude anthracene or anthracene residues are heated at a high temperature under pressure by the Bergius process and the volatile products so obtained are mixed with pure benzene to obtain an anti-knock motor fuel.

A. R. POWELL.

Retort for carbonising bituminous fuels. J. PLASSMANN, ASSR. to CHEM.-TECHN. GES.M.B.H. (U.S.P. 1,737,681, 3.12.29. Appl., 14.6.26. Ger., 30.6.25).—See B.P. 254,697; B., 1927, 644.

Production, from hydrocarbon material, of gases or liquids of changed mol. wt. I. W. HENRY (B.P. 321,910, 17.5.28).—See U.S.P. 1,709,815; B., 1929, 506.

Apparatus for continuously expelling the sulphur dioxide from mixtures of sulphur dioxide and oil.

P. JODECK, Assr. to ALLGEM. GES. F. CHEM. IND. M.B.H. (U.S.P. 1,738,070, 3.12.29. Appl., 16.6.26. Ger., 12.4.26).—See B.P. 269,118; B., 1927, 722.

Treatment of montan wax. W. PUNGS and M. JAHRSTORFER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,737,975, 3.12.29. Appl., 4.2.28. Ger., 19.8.27).—See B.P. 308,996; B., 1929, 425.

Burners for liquid fuels. ETABL. CAUVET-LAMBERT, Assees. of U. MAGINI (B.P. 304,149, 14.1.29. It., 14.1.28).

Treatment of liquids with liquids (B.P. 321,200). **Fractional condensation** (U.S.P. 1,735,455). **Condensers and scrubbers** (B.P. 321,204).—See I. **Recovery of diolefines** (B.P. 319,025).—See III. **Hydrogen from mixed gases** (B.P. 291,409). **Less-inflammable hydrogen** (B.P. 294,958).—See VII. **Emulsions** (U.S.P. 1,734,975).—See XI.

III.—ORGANIC INTERMEDIATES.

Synthesis and free energy of methane. M. RANDALL and A. MOHAMMAD (Ind. Eng. Chem., 1929, 21, 1048—1052).—The synthesis of methane from carbon and hydrogen has been studied over the temperature range 1052—1237° Abs., also the decomposition between 1024° and 1180° Abs. Nickel activated by the addition of ceria was used as a catalyst. The free energy of formation of methane was found to be $-11,573$ g.-cal., a value in good agreement with that previously found by Randall and Gerard (B., 1929, 82). Equilibrium data obtained by previous workers are tabulated.

H. INGLESON.

Vapour-pressure chart for lower aliphatic hydrocarbons. R. L. COPSON and P. K. FROLICH (Ind. Eng. Chem., 1929, 21, 1116—1117).—Vapour-pressure curves of methane, ethylene, ethane, propane, isobutane, *n*-butane, isopentane, and *n*-pentane have been plotted from data published by numerous authors.

H. INGLESON.

Catalysts for formation of alcohols from carbon monoxide and hydrogen. V. **Decomposition and synthesis of methyl alcohol with a zinc-copper-chromium oxide catalyst.** M. R. FENSKE and P. K. FROLICH (Ind. Eng. Chem., 1929, 21, 1052—1055; cf. B., 1929, 934).—A study of the properties of a catalyst composed of the oxides of zinc, copper, and chromium, in which the metals are present in the molecular ratio 43 : 49 : 8, showed that this ternary mixture was far more active in promoting the decomposition and in the synthesis of methyl alcohol than any of the binary systems copper-zinc and chromium-zinc previously examined. The temperature range of the decomposition experiments was 220—285°. It was found that at temperatures above 250° the alcohol decomposed according to the equation $\text{CH}_3\cdot\text{OH} = \text{CO} + 2\text{H}_2$ with very little production of formaldehyde or methyl formate, whilst at lower temperatures these two substances were found in somewhat larger amount. The activity of the catalyst in promoting the decomposition falls off after a time, but may be restored by oxidation and subsequent reduction. When methyl alcohol was passed over the catalyst at 180° important quantities of carbon dioxide

appeared in the exit gas due to the reduction by carbon monoxide of the oxides; the hydrogen also present did not appear to reduce the oxides at temperatures up to 250°. The results obtained in the decomposition experiments are compared with those previously found (B., 1928, 359) in the synthesis of methyl alcohol under high pressure.

H. INGLESON.

Decomposition of methyl alcohol by catalysts composed of oxides of zinc and chromium. J. R. HUFFMANN and B. F. DODGE (Ind. Eng. Chem., 1929, 21, 1056—1061).—The experiments were carried out at 350° with catalysts varying in composition from pure zinc oxide to pure chromic oxide. The catalysts were prepared by precipitation of the hydroxides from solutions of the mixed nitrates by addition of ammonia solution. After washing and drying at 110°, the catalysts were reduced by hydrogen up to a temperature of 300° before being used in the decomposition of methyl alcohol. During this reduction it was noticed that a considerable shrinkage took place, especially when the chromium content exceeded about 25%. The bearing of this observation on the comparison of catalytic activities is discussed. Comparison of results obtained in unpublished synthesis experiments with those obtained in the study of the decomposition show that agreement is good, but not complete. The addition of a small amount of chromium oxide to zinc oxide caused a decrease in the total decomposition of methyl alcohol, contrary to its effect in the synthesis. If only the decomposition into carbon monoxide and hydrogen is considered the effect is in accord with that observed in the synthesis. In all the decomposition experiments the activity of the catalysts was found to decrease. No such decrease was observed in the synthesis experiments. In addition to carbon monoxide and hydrogen the following substances were detected during the decomposition: carbon dioxide, methane, methyl formate, formaldehyde, dimethyl ether, unsaturated compounds, together with traces of unidentified substances. Curves are given in which the percentage of methyl alcohol decomposed into various products is expressed as a function of the composition of the catalyst. Although the results obtained differ in some respects from those of Frolich, the catalyst having the maximum activity was found to have the composition $\text{Zn/Cr} = 4$, in close agreement with this author's observation (cf. preceding abstract).

H. INGLESON.

Recovery of acetone vapour from the air. W. R. ORMANDY (Inst. Chem. Eng., Dec. 5, 1929, 10—13. Advance copy).—Sulphuric acid and phosphoric acids at high concentrations are almost perfect absorption media for acetone, whilst in the diluted state they are little superior to water. Phosphoric acid is in many respects superior to sulphuric acid, but no material is available which can be employed in large-scale construction and will resist the hot, concentrated acid. It is therefore proposed to use sulphuric acid (*d* 1.70) as washing agent. With air containing 24 g./cub. m. of acetone, the acid leaving the scrubbing tower will consist of 2 vols. of acid (*d* 1.64) and 1 vol. of acetone. This is diluted with water to *d* 1.45 and the acetone is distilled off. The acid during the washing process should

be kept below 25° by water-cooling. The partial pressure of acetone 4.77% by wt. in admixture with sulphuric acid of increasing concentration, increases up to about 20% acid and then falls; it is negligible in 80% acid. Vapour pressure determinations suggest that in 98% acid a compound $(\text{CH}_3)_2\text{CO} \cdot 2\text{H}_2\text{SO}_4$ is formed, but that this is largely decomposed by small quantities of water. C. IRWIN.

Determination of hexamethylenetetramine. E. E. REBAGLIATI (Rev. farm., Buenos Aires, 1929, 2, 340—349).—The solution of hexamethylenetetramine (0.025—0.07 g.) is hydrolysed with sulphuric acid at 100° during 5 min. in a stoppered flask; after cooling, 4% copper sulphate solution (20 c.c.) and 20 c.c. of a solution of sodium potassium tartrate (200 g.) and sodium hydroxide (200 g.) in water (1000 c.c.) are added. The ammonia is removed by distillation and determined, and the cuprous oxide is dissolved in standard ferric sulphate solution in presence of sulphuric acid, the ferrous iron being titrated with permanganate.

CHEMICAL ABSTRACTS.

Purification of anthracene. T. I. RABEK and J. BOJANOVSKI (Przenysl Chem., 1929, 13, 508—510).—Jaeger's view that the most rational methods of purification of anthracene are those in which the impurities are destroyed by oxidation (B., 1928, 740) is questioned, on the grounds of the commercial value of these impurities. Oil (b.p. 150—240°) obtained from crude gas tar affords an efficient and cheap solvent for the fractional crystallisation of crude anthracene; a 95% yield of 68% anthracene is obtained by one recrystallisation of 20% anthracene from this solvent. The tar oil is regenerated by distillation of the mother-liquor, whilst the residue makes a valuable admixture to impregnation oil or to carbolineum. The 68% anthracene obtained in the above way may be further purified either by Jaeger's method or by recrystallisation from high-boiling pyridine bases; the fraction of b.p. 160—190° is particularly suitable for the purpose, having the same solvent power as low-b.p. pyridine bases, being cheaper, and, as a result of the smaller losses due to evaporation, more economical in use. Two recrystallisations of the 68% product from this solvent yield 96% anthracene.

R. TRUSZKOWSKI.

Solvent recovery. BODEWIG; HOCK.—See I. Acetic acid from pyroligneous acid. KRASE.—See II.

PATENTS.

Chlorination of hydrocarbons. E. GREMLI (Aust. P. 108,421 and 108,424, [A] 13.2.24, [B] 29.12.24).—Methane, ethane, or benzene is treated with chlorine at 300—650° in the presence of oxygen with the addition, if desired, of hydrogen chloride, of diluents, e.g., steam, carbon dioxide, nitrogen, and of catalysts, e.g., iron, thallium, alkaline- or rare-earth chlorides, or charcoal.

L. A. COLES.

Recovery of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,025, 12.3.28).—Ethylene chlorohydrin, glycol acetates, glycol mono-methyl ether, glycerol di- or tri-acetate, lactonitrile (acetaldehyde cyanohydrin), ethyl tartrate, furfuraldehyde, or arylamines (aniline, toluidine, phenylhydr-

azine), or mixtures of these, are used as solvents for extraction of diolefines (butadiene, isoprene) from less saturated hydrocarbons. The scrubbing is effective at 0—10°, and the diolefines are recovered in 80—85% purity on warming the solution. C. HOLLINS.

Production of alcohols by the oxidation of methane. COMP. DE BÉTHUNE (F.P. 630,680, 7.6.26).—Methane is converted into methyl alcohol by heating it below 500°, and under raised pressure with the correct quantity of oxygen in the presence of non-reacting material, e.g., sand. L. A. COLES.

Manufacture of acetone from acetylene. I. G. FARBENIND. A.-G. (B.P. 299,048, 16.10.28. Ger., 21.10.27).—A mixture, preferably preheated, of acetylene and steam, and, if desired, air, is passed over a catalyst consisting of a metal or metal compound such that the acetate of the metal on heating decomposes with the formation of acetone (excluding ferric and thorium oxides and thorium and alkali or alkaline-earth double salts), at a rate lower than that at which acetaldehyde is the main product. E.g., a mixture of 30 pts. by vol. of acetylene and 90 pts. of steam preheated to 300° is passed per hr. over 1 pt. by vol. of zinc oxide at 450°.

L. A. COLES.

Manufacture of condensation products [acetonitrile] from acetylene and ammonia. I. G. FARBENIND. A.-G. (B.P. 295,276, 13.7.28. Ger., 8.8.27).—Acetylene and ammonia are passed at 450° over active silica or alumina gel impregnated with metal oxides not reducible under these conditions (e.g., a mixture of zinc oxide and thoria). Acetonitrile is formed in 75% yield.

C. HOLLINS.

Purification of carbon disulphide. P. L. F. NICOLARDOT (F.P. 631,033, 9.6.26).—The vapour of crude carbon disulphide is absorbed in oil, silica gel, or charcoal activated with calcium chloride or glycerin, and is subsequently expelled by heating and then condensed.

L. A. COLES.

Manufacture of concentrated volatile aliphatic acids. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 306,105, 27.4.28. Ger., 16.2.28).—Dilute (50%) acetic acid is mixed with sodium and potassium acetates and distilled up to 170° to remove water (as 10—12% acid). The acid salts are further distilled up to 300° to give 95% acid. The use of mixed salts ensures that during the whole distillation the mass remains completely liquid. Butyric acid may be extracted from its solution in acetone oil by means of concentrated aqueous potassium and zinc butyrates, and the extract distilled as before. The process is similarly applicable to formic, propionic, and other aliphatic acids. [Stat. ref.] C. HOLLINS.

Manufacture of aliphatic and cycloaliphatic saturated sulphinic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,843, 26.11.28).—Open-chain or cycloaliphatic saturated hydrocarbons, or mixtures containing them, are treated with sulphur dioxide at raised temperatures and pressures in the presence of anhydrous aluminium or boron halides.

L. A. COLES.

Manufacture of carbonic acid esters of the glycols. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,894, 18.2.29).—Vicinal glycol chlorohydrins,

e.g., ethylene chlorohydrin, are treated with alkali carbonates or bicarbonates, preferably at a raised temperature (*e.g.*, 60–70°) and, if desired, at raised pressure, and with removal by the addition of a liquid, *e.g.*, ethylene chloride, benzene, which forms a low-boiling binary mixture with water, of the water formed during the reaction. The preparation of ethylene and propylene glycol carbonates is described. L. A. COLES.

Preparation of β -hydroxy- α -phenylacraldehyde. H. RUPE (Swiss P. 120,517, 5.2.26).—The compound is produced by the catalytic reduction of β -hydroxy- α -phenylacrylonitrile and subsequent hydrolysis of the aldimine obtained thereby (*cf.* Rupe and Knup, A., 1927, 564). L. A. COLES.

Manufacture of aliphatic compounds [acetic anhydride]. H. DREYFUS (U.S.P. 1,735,959 and 1,735,962, 19.11.29. Appl., [A] 19.1.27, [B] 2.5.27. U.K., [A] 1.6.26, [B] 26.5.26).—See B.P. 279,916 and 280,972; B., 1928, 45, 82.

Manufacture of acetic anhydride. H. DREYFUS (U.S.P. 1,735,956—8, 19.11.29. Appl., 3.4.26. U.K., 9.4.25).—See B.P. 256,663—4; B., 1926, 897.

Manufacture of ethylene glycol. K. H. SAUNDERS and H. WIGNALL, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,737,545, 26.11.29. Appl., 2.2.28. U.K., 8.2.27).—See B.P. 286,850; B., 1928, 360.

Manufacture of [alkyl β -halogenoethyl] ketones. W. SCHOELLER and C. ZÖLLNER, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,737,203, 26.11.29. Appl., 16.12.27. Ger., 15.12.26).—See B.P. 282,412; B., 1928, 398.

Manufacture of quaternary ammonium compounds. M. HARTMANN and J. KÄGI, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,737,458, 26.11.29. Appl., 19.7.28. Ger., 26.7.27).—See B.P. 294,582; B., 1929, 807.

Manufacture of alkylated aromatic sulphonic acids. F. GÜNTHER and J. HETZER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,737,792, 3.12.29. Appl., 28.1.26. Ger., 30.1.25).—See B.P. 246,817; B., 1926, 850.

Manufacture of chloroperylene quinones. A. ZINKE, Assr. to F. BENSA (U.S.P. 1,738,343, 3.12.29. Appl., 7.6.27. Austr., 26.11.26).—See B.P. 281,281; B., 1928, 666.

Glutamic acid (B.P. 320,589).—See XVII.

IV.—DYESTUFFS.

Fastness to light of lake colours. S. T. KINSMAN (J. Oil and Colour Chem. Assoc., 1929, 12, 274—283).—Lakemakers are unable to guarantee their products as can dyers, since dyestuffs are faster to light on fibre than in the form of a lake, and vat dyes which form the bulk of textile colours have very limited use for lakes. The course and cause of fading, the effects of different media, comparison between daylight and lamp tests are all discussed. For a particular class of dyestuff, chemical stability increases fastness, whilst increase in amount of extender decreases it. It is suggested that lakes be classified into five arbitrary groups, accord-

ing to the various effects of sunlight exposures on them under specified conditions in a standard cabinet. Thus the lakes show (i) no perceptible alteration after 1 month of summer sunshine, (ii) slight loss, or (iii) distinct loss, in depth or alteration in shade after 1 month's exposure, (iv) distinct loss in depth etc. after 14 days' exposure, and (v) practically complete bleaching out after 1 week's exposure. It is pointed out that the actinic value of the light in any particular month is an important factor to be considered. For practical testing purposes it is necessary to reproduce these grades of fading by an accelerated test. The mercury-vapour lamp is rejected for this purpose owing to production of ozone and absence of heating effect. Further, some colours change more slowly in ultra-violet light than in sunlight. The spectral curve of the carbon arc is more comparable with that of sunlight, and gives a rough figure of 64 hrs.' exposure as equivalent to 1 month's summer sunlight, although there are anomalies, some of which are discussed. The following lakes are proposed as representatives of the five classes of fastness:—Alizarin-red, aluminium calcium phosphate lake; Permanent-red 4B, calcium salt; Lithol-red R, barium salt; Eosine YS, lead salt, barium sulphate base; lakes of fastness less than that of Eosine. The carbon-arc readings of these types are: unbleached after 240 hrs., practically unbleached after 120 hrs., bleached after 60 hrs., bleached after 34 hrs., and bleached in less than 34 hrs., respectively. S. S. WOOLF.

PATENTS.

Manufacture of nitrogenous vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,180, 24.2.28).—Homocyclic compounds containing two or more replaceable negative substituents (halogens etc.) are condensed simultaneously or successively with two or more different amines or amides; or, conversely, polyamino-derivatives are condensed with two or more different halogenated compounds; in either case one or more of the reactants must be a vattable compound. Preferably a diluent and an acid-fixing substance are present. Amongst the 66 examples are: tetrabromopyranthrone with aminodibenzanthrone and 1-aminoanthraquinone; dibromanthranthrone, tetrabromo-*ms*-benzidanthrone, dichloro-*allo*-*ms*-naphthodianthrone, 1:5-dinitroanthraquinone, and others with the same two amines; diamino-*allo*-*ms*-naphthodianthrone with 1-chloroanthraquinone and bromobenzanthrone-pyrazolanthrone. C. HOLLINS.

Manufacture of azo dyes [for lakes and wool]. I. G. FARBENIND. A.-G. (B.P. 293,352, 31.5.28. Ger., 4.7.27. Cf. B.P. 9252 of 1914; B., 1915, 623).—A sulphonated arylamine is used in place of the amine in the prior process. 4-Chloro-2-nitrobenzeneazacetoyl chloride, m.p. 154°, is condensed with *o*-anisidine-4-sulphonic acid in pyridine at 30–35° to give a light-fast yellow wool dye, which in the form of lake pigment is fast to water and light. 3-Nitrotoluene-4-azacetoyl chloride is similarly condensed with 4-chloro-metanilic acid. C. HOLLINS.

Condensation product of the anthraquinone series and its manufacture. G. KRÄNZLEIN and R. SEDLMAYR, Assrs. to GEN. ANILINE WORKS, INC.

(U.S.P. 1,736,084, 19.11.29. Appl., 23.11.25. Ger., 27.12.24).—See B.P. 245,165; B., 1927, 470.

Trisazo dyes. E. FELLNER, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,736,905, 26.11.29. Appl., 20.9.26. Ger., 14.10.25).—See B.P. 259,970; B., 1928, 226.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Theory of the sulphite cooking process. M. STEINSCHNEIDER and E. STOLZ (Papier-Fabr., 1929, 27, 750—755).—Lignin obtained from waste liquors is insoluble in water and in cold calcium bisulphite solution; it only swells or dissolves to a slight extent in water on long keeping or on heating. When precipitated from waste liquor with alcohol, and freed from inorganic and organic impurities, it contains 8—9% S. Dissolution of lignin only occurs in hot solutions under the working conditions employed in the sulphite-cellulose industry, and, independent of the nature of the cooking, these lignins contain 6—6.5% S. The difference in sulphur content and in the water-solubility of lignins containing sulphur leads to the conclusion that there is a relation between these two factors, and theories are discussed which attempt to explain the facts observed. Results of experiments in which the absorption of sulphur by wood or cellulose was examined throughout the cooking process, and in which samples of different lignin content were submitted to hydrolysis with 1% hydrochloric acid, indicate that prior to, or simultaneously with, the first absorption of sulphur a cleavage of the lignin complex occurs with the production of a lignin which may be converted by the action of hot hydrochloric acid into a water-soluble product of low sulphur content. The absorption of sulphur by lignin in the cooker proceeds at first until 6% content is reached; in this condition the lignin is insoluble in water, and it is only as a consequence of the subsequent addition of sulphur dioxide to its carbonyl groups that it is converted into the so-called water-soluble ligninsulphonic acid. Owing to the structure of the wood these processes may follow one another in the different layers, so that the different stages of decomposition are present simultaneously in the same sample. This affords an explanation of black-cooking. If insufficient sulphur dioxide is present to promote the formation of the ligninsulphonic acid, pulp is obtained which has a dark interior, but if after the formation of this acid the loosely combined sulphur dioxide is again split off owing to the higher p_H value of the liquor caused by the organic acids present, a uniformly cooked pulp is obtained which, however, is dark brown in colour and is only bleached with difficulty.

B. P. RIDGE.

Hydration of pulp in beating. A. S. KLEIN (Papier-Fabr., 1929, 27, 755—757).—During beating the fibres are crushed, stretched, burst, cut, and split, and the resulting properties of the pulp and of the paper made from it depend to a great extent on which of these effects predominates. If transverse cutting predominates, the external surface of the fibre lengths is less than if the fibres are split lengthwise, and in the latter case the material retains more water and yields it up more slowly during the formation of the paper sheet. If pulp formed from crushed rather than from cut fibres

is examined microscopically, a certain amount of mucilage is always found. The action of water on the fibres may be due to saturation or swelling, or to both, and the term "hydration" is applied in a general sense to effects characterised by increased retention of water, occlusion, absorption, or mucilage formation, without attempt at more precise definition. Hydration is influenced by mechanical processes such as occur in beating, by temperature and time of action, and may also be partly chemical in nature. The views of several authors on effects related to hydration, such as capillary action, hysteresis, chemical combination, swelling, etc., are quoted, all of which show that the mechanism of pulp hydration is little understood.

B. P. RIDGE.

Differential action of the blowpipe flame on the red and white components of wood: its uses and behaviour. H. WISLICENUS [with, in part, A. KRAESS] (Cellulosechem., 1929, 10, 206—211).—The red component (*i.e.*, heartwood, knots, and the annual rings) of wood is differentiated from the white not only by its colour, but also by its chemical and physical properties. In freshly cut wood the red portion is dense and dry and contains a larger proportion of easily oxidisable aromatic constituents. If, therefore, a freshly cut, planed cross-section is taken and a hot blowpipe flame passed over it, there follows a development (in the photographic sense) of the red wood, which is darkened, while the white portion remains unchanged. With air-dried or completely dried wood the process is not so successful, but here the same development may be brought about by ammonia gas, which causes differential darkening of the red-wood, tannin-containing portion. By means of this process the existence of heartwood may be demonstrated even in sapwood in a more or less rudimentary form. The action of the blowpipe on longitudinal sections has also been investigated. If a smooth, planed surface is taken and a very hot blowpipe flame passed over it, there ensues a development of the natural graining even in the case of thoroughly dried wood. A smooth, matte surface is obtained which has enhanced resistant properties, and which renders the wood immune to wood-destroying insects. The process is applicable to oak, birch, lime, maple, and coniferous woods generally, but in the case of beech, alder, and elm the production of irregular coloration is likely.

T. H. MORTON.

Solvent recovery. BODEWIG.—See I. **Active carbon.** EVANS and others.—See II. **Waterproofing medium.** BRAUN.—See XII.

PATENTS.

Preparing cereal-straw fibre for the manufacture of relatively thick fibrous sheets therefrom. E. S. SHEPHERD, ASSR. to A. D. STEWART (U.S.P. 1,728,258, 17.9.29. Appl., 23.11.28).—Separated cereal-straw fibres are cooked with an amount of water sufficient to loosen the gum-like material, but so regulated as to avoid softening them to a point where they would be slimy and slow. After subjection to a tearing action to reduce their cross-section, while preserving their greatest possible length, they are mixed with water, beaten with a brushing action to preserve their length, and cleansed with water.

F. R. ENNOS.

Composition for impregnating balloon fabrics.

E. and B. TRENCKMANN, Assrs. to LUFTSCHIFFBAU ZEPPELIN GES.M.B.H. (U.S.P. 1,730,544, 8.10.29. Appl., 27.8.21. Ger., 3.3.16).—To render cotton fabrics gas-tight without unduly increasing their weight or reducing their flexibility or elasticity, they are coated with a mixture of 10 pts. each of glue or gelatin and Turkey-red oil in 100 pts. of water, together with 5 pts. of glycerin and a little potassium dichromate, if desired, and afterwards dried. F. R. ENNOS.

Stiffening of fabric [containing cellulose derivatives]. BRIT. CELANESE, LTD. (B.P. 302,361, 13.12.28. U.S., 15.12.27).—The fabrics are treated with solvents, softening agents, or swelling agents, *e.g.*, ethylene dichloride, with or without a diluent, which are afterwards removed by evaporation. F. R. ENNOS.

Treatment of yarns or threads [containing cellulose derivatives] in textile operations. BRIT. CELANESE, LTD. (B.P. 295,054, 4.8.28. U.S., 6.8.27).—The threads are lubricated, before knitting or weaving, by being thoroughly wetted with water or aqueous solutions of materials, such as soaps, sulphonated oils, or polyhydric alcohols, which do not require to be subsequently removed. F. R. ENNOS.

Production of bleached chemical wood pulp. L. BRADLEY and E. P. MCKEEFE, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,730,315, 1.10.29. Appl., 19.5.21).—The fluid pulp, containing only a small amount of cellulose in a large amount of water, is sprayed into a chamber made of acid-resisting material where it gravitates through an atmosphere of chlorine gas, the operation being repeated one or more times to complete the bleaching; if desired, an acid solution containing a fluoride is added to the pulp to remove silica. F. R. ENNOS.

Manufacture of cellulose esters. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 321,575, 30.11.28).—Cellulose which has been treated according to the process of B.P. 237,567 (B., 1926, 49) is esterified with *n*- or *iso*-butyric anhydride in the presence of a catalyst and with or without a diluent, whereby mixed esters of acetic and butyric acids are obtained which are soluble in benzene at ordinary temperature. F. R. ENNOS.

Esterification of cellulose. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 303,136, 3.8.28. Fr., 30.12.27).—Cellulose is esterified by means of an organic acid and its anhydride in the presence of hydrofluoric acid as catalyst, with or without the addition of a very small amount of an oxidising agent or nitric acid and of a non-solvent such as benzene or carbon tetrachloride. F. R. ENNOS.

Saponification of primary solutions of acetylcellulose. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 303,098, 3.8.28. Fr., 28.12.27).—The solution of acetylcellulose obtained by acetylation in the presence of sulphuric acid as catalyst is hydrolysed in two stages; in the first an aqueous solution of acetic or formic acid is added in sufficient quantity to decompose the excess of acetic anhydride and to prevent incipient flocculation, whilst in the second there is added an aqueous solution of acetic or formic acid containing an

amount of hydrochloric acid mixed with hydrofluoric acid which is at least chemically equivalent to the sulphuric acid used as catalyst, the amount of water in the entire mass being 5–15% of the total organic acids present. F. R. ENNOS.

Apparatus for production of artificial filaments or threads. H. DREYFUS (B.P. 320,632, 11.7.28).—The spinning solution, consisting of organic derivatives of cellulose in volatile solvents, is brought to a predetermined temperature and maintained thereat by insulating it while it passes from the attemperating means to the spinning nozzles. F. R. ENNOS.

Manufacture of very fine viscose silk threads in an acid spinning bath. VEREIN. GLANZSTOFF-FABR. A.-G. (G.P. 454,680, 16.11.20. Addn. to G.P. 444,113; B., 1928, 187).—In addition to the constituents claimed in the chief patent, the bath contains sulphur dioxide, a bisulphite, or a sulphite. A. R. POWELL.

Compositions and materials comprising cellulose esters or ethers. BRIT. CELANESE, LTD., and A. J. DALY (B.P. 319,073, 19.6.28).—Cellulose esters or ethers and plastics, films, lacquers, fibres, etc. made from them are rendered less inflammable by incorporation with 7–10% of mono- or di-acetylated tribromoaniline. Other brominated arylamides may be used. C. HOLLINS.

Production of pressure-mouldable materials. G. TRÜMLER (B.P. 294,251, 18.7.28. Ger., 21.7.27).—Mixtures of finely-divided cellulose esters or ethers with one or more volatile liquids which are non-solvents, but plasticisers for the cellulose derivatives, together with fillers, resins, etc., if desired, are subjected to the action of hot, preferably boiling, water, hot salt solutions, heated or superheated aqueous vapour, or a combination or sequence of these; or mixtures of the esters with volatile solvents are subjected to heated or superheated water vapour. In each case the volatile liquids are driven off and the residue is converted into a readily disintegrable condition suitable for further treatment in the manufacture of pressed articles. F. R. ENNOS.

Manufacture of impregnated paper sacks. A. HOLTER (B.P. 307,005, 12.6.28. Norw., 1.3.28).—Bags for cement, fertilisers, etc. are made from a number of superimposed paper webs, which are impregnated by moistening adjacent sheets with substances reacting to form insoluble products, *e.g.*, soaps or glue on the one hand, and aluminium salts on the other, or with a single solution comprising a mixture of these substances. F. R. ENNOS.

Production of textile material. L. LILIENFELD (U.S.P. 1,736,714, 19.11.29. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,803; B., 1925, 709.

Moistureproof [cellulosic] material. W. H. CHURCH and K. E. PRINDLE, Assrs. to DU PONT CELLOPHANE CO., INC. (U.S.P. 1,737,187, 26.11.29. Appl., 3.1.27).—See B.P. 283,109; B., 1929, 715.

Opening-up materials containing cellulose. H. HEIMANN, H. SEEFRIED, I. PETERSEN, and A. BAYERL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,736,080, 19.11.29. Appl., 7.7.27. Ger., 21.7.26).—See B.P. 274,892; B., 1928, 154.

Reducing the swelling capacity of hydrated cellulose. C. HEUCK and P. ESSELMANN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,737,760, 3.12.29. Appl., 20.12.28. Ger., 25.7.27).—See B.P. 294,551; B., 1929, 892.

Wet spinning of flax and similar fibres and apparatus therefor. LINEN INDUSTRY RES. ASSOC., and J. A. MATTHEW (B.P. 321,816, 7.11.28).

Filter [candles] for use in artificial silk apparatus. H. WADE. From SYNTHETA A.-G. (B.P. 321,717, 18.8.28).

Filtration of solutions (B.P. 308,278).—See I. Chromic compounds (B.P. 294,965).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Artificial soiling of cotton fabrics preparatory to laundering studies. A. E. HILL (J. Agric. Res., 1929, 39, 539—550).—A suitable soiling mixture for laundering studies contains Oildag, olive oil, tallow, and mineral oil; these represent all the constituents of natural soiling except albuminous matter, which latter if included would limit temperature ranges for experimental work, and stains. E. HOLMES.

Fastness to light of lake colours. KINSMAN.—See IV.

PATENTS.

Bleaching of animal fibres and substances [e.g., furs, feathers]. H. GOLDARBEITER (B.P. 291,743, 31.5.28. Austr., 7.6.27).—The deleterious action on skins and furs of slow bleaching liquors containing hydrogen peroxide is avoided by adding to such solutions of relatively low concentration and small alkalinity oxygen transmitters which promote a rapid decomposition of the hydrogen peroxide. For this purpose addition of potassium ferricyanide (5—10 g. per litre of bleaching liquor), or a salt of copper or vanadium, is suitable. A. J. HALL.

Dyeing of cellulose acetate silk in fast [oxidation] black shades. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 321,034, 2.10.28).—Dyeing is effected by treatment with an aqueous suspension of α -naphthylamine followed by oxidation with nitrous acid at 35—40°. A. J. HALL.

Production or treatment of fabrics [cellulose effects on cellulose ester fabrics for cross-dyeing]. BRIT. CELANESE, LTD. (B.P. 295,582, 11.8.28. U.S., 13.8.27).—An acetate silk fabric is printed with cellulose xanthate (viscose), which is then converted into cellulose by means of dilute sulphuric acid. The material is then cross-dyed, e.g., with S.R.A. Fast Blue IV and Chlorazol Yellow GX or Chloramine Scarlet 4BX. C. HOLLINS.

Coloration of cellulose [ester and ether] derivatives. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 321,401, 1.8.28).—Materials containing cellulose esters or ethers, particularly cellulose acetate, are dyed by means of aqueous dispersions of amino-, alkylamino-, or aralkylamino-substituted derivatives (in their unreduced state) of benzo- or naphthaquinones or substitution derivatives of these compounds. Suitable dispersing agents are those claimed in B.P.

219,349, 224,925, 242,393, 242,711, 269,960, 273,819, and 273,820 (B., 1924, 906; 1925, 39; 1926, 87, 50; 1927, 475, 650). E.g., 2:5-diaminobenzoquinone dispersed by means of Turkey-red oil yields a yellow shade.

A. J. HALL.

Enhancing the action of treatment liquids employed in the textile industry. H. T. BÖHME A.-G. (B.P. 297,382, 20.8.28. Ger., 20.9.27).—Wetting-out agents suitable for addition to mercerising liquors are prepared by mixing a highly-sulphonated oil (cf. B.P. 284,280; B., 1928, 935) with a phenol other than a hydrogenated aromatic phenol. E.g., 1 pt. of tricresol is added to the product obtained by sulphonating 1 pt. of oleic acid in the presence of acetic anhydride, caustic soda being added to the mixture so as to form a clear solution. A. J. HALL.

Removal of caustic soda-soluble substances from materials containing cellulose. H. P. BASSETT (U.S.P. 1,733,393, 29.10.29. Appl., 22.12.25).—The materials are treated with sodium hydroxide solution of mercerising strength in which they are allowed to remain for 1—4 days, excess hydroxide is then removed, the product diluted with water, and boiled to remove soluble substances. H. ROYAL-DAWSON.

Mordanting and dyeing of wool. C. S. BEDFORD (U.S.P. 1,735,844, 19.11.29. Appl., 15.1.26. U.K., 23.4.25).—See B.P. 253,662; B., 1926, 706.

Dyeing of materials made of or containing cellulose derivatives. H. DREYFUS (U.S.P. 1,735,960—1 and 1,735,963, 19.11.29. Appl., [A, B] 12.3.27, [C] 30.1.28. U.K., 14.12.26).—See B.P. 292,452, 292,180, and 291,816; B., 1928, 639.

Increasing the fastness to light of basic dyes. P. RABE, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,736,835, 26.11.29. Appl., 20.9.27. Ger., 12.8.26).—See B.P. 294,286; B., 1926, 708.

Production of fast printings. A. ZITSCHER and F. MURIS, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,737,905, 3.12.29. Appl., 8.12.25. Ger., 24.12.24).—See G.P. 433,276; B., 1927, 216.

Treatment of animal and vegetable fibres. M. BERGMANN, E. IMMENDÖRFER, and H. LOEWE (U.S.P. 1,737,104, 26.11.29. Appl., 12.12.23. Ger., 18.12.22).—See B.P. 208,563; B., 1924, 978.

Improvement of cotton. L. LILIENFELD (U.S.P. 1,736,712—3 and 1,738,190, [A, B] 19.11.29, [C] 3.12.29. Appl., [A, B] 10.5.24, [C] 3.2.27. Austr., [A—C] 23.5.23).—See B.P. 216,476; B., 1925, 69.

Dye jiggers. J. POOLE, and WHITEHEAD & POOLE, LTD. (B.P. 322,042, 6.10.28).

Bleaching of wood pulp (U.S.P. 1,730,315).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Alumina production in its present-day aspects. J. D. EDWARDS and R. B. MASON (Chem. Met. Eng., 1929, 36, 674—677).—Alkaline processes are most suitable for ores low in silica and acid processes for ores low in iron. In the Bayer process 1 pt. of silica causes the

loss of 1.1—2 pts. of alumina and 1—3 pts. of sodium carbonate. The effect of titanium is not quite certain. The Bayer process is described. The Pedersen process is another practical alkaline process. A mixture of iron ore, coke, lime, and bauxite is smelted, producing a calcium aluminate slag containing 30—50% Al_2O_3 and high-grade iron as a by-product. A 3—8% solution of sodium carbonate containing 0.3—0.8% NaOH is used for extracting the slag. C. IRWIN.

Determination of silica in presence of fluorspar.

W. T. SCHRENK and W. H. ODE (Ind. Eng. Chem. [Anal.], 1929, 1, 201—202).—The method is based on the fact that fluorspar in presence of silica can be completely decomposed by heating with perchloric and boric acids without loss of silica, the fluorine being expelled as boron trifluoride. A sample (0.5 g.) is evaporated with 15 c.c. of 20% perchloric acid, which has been previously saturated at 50° with boric acid, until fumes of perchloric acid are evolved. The mixture is kept at the fuming point for 5 min., water is then added, and the evaporation repeated. The solution is diluted and filtered, the paper being washed first with dilute perchloric acid and then with water until free from calcium. The silica in the residue is determined in the usual way. Test analyses on mixtures of known composition yielded excellent results. H. F. HARWOOD.

Preparation and determination of hyposulphites.

A. MCGLYNN and O. W. BROWN (J. Physical Chem., 1929, 33, 1665—1681).—The influence of various factors in the preparation of sodium hyposulphites by electrolytic and chemical methods has been determined. In the electrolytic method sulphuric acid, d 1.224, is a better anolyte than are 10% solutions of anhydrous sodium sulphate, sodium hydroxide, or sodium chloride, whilst cathodes of amalgamated zinc and pure zinc give better yields than those of platinum gauze, tin, tinned iron, or mercury. The yield is lowered by an increase in current concentration, current density, and temperature. The highest yield is obtained when 125% of the theoretical amount of electricity has been passed. The addition of solid sodium hydrogen sulphite during electrolysis does not increase the yield, but makes possible the preparation of a more concentrated solution of the hyposulphite. The highest percentage current yield is obtained during the first amp.-hr. and with the more concentrated solutions of hydrogen sulphite (25—35%), but the decomposition of the hyposulphite soon outweighs its formation. In the chemical method of preparation a greater yield of hyposulphite is obtained by passing sulphur dioxide through a mixture of sodium hydrogen sulphite and zinc dust than through distilled water and zinc dust. The yield increases with an increase in concentration of the hydrogen sulphite, but is unchanged by an increase in the rate of flow of sulphur dioxide from 3.5 to 20 litres per hr. Existing methods for the determination of hyposulphites have been compared; those of Seyewetz and Bloch (A., 1906, ii, 578) and of Orloff (A., 1905, ii, 200) give the most satisfactory results for laboratory practice. L. S. THEOBALD.

Test for thiosulphates. E. E. JELLY and W. CLARK (Brit. J. Phot., 1929, 76, 714—716).—Feigl's original test for sulphides and thiosulphates (A., 1928, 1106), which

was shown by Metz to apply also to polythionates, has been modified so as to be specific for thiosulphates. A slight excess of 0.0005*N*-iodine-starch solution is added to the liquid under test, so that a faint blue colour persists. One drop of a 2% solution of sodium azide is then added, when the blue colour is immediately discharged if thiosulphates are present; 0.000007 mg. of sodium thiosulphate in 0.04 c.c. of solution can thus be detected. Sodium trithionate also gives a feeble reaction under the above conditions, but tetra- and pentathionates, sulphides, sulphites, and sulphates are without effect. In utilising the above test for the detection of retained thiosulphate in photographic negatives and prints, it is recommended to use a stock solution containing 0.6% of soluble starch and 0.13% each of potassium iodide, iodine, and sodium azide. 1—2 c.c. of this solution are then diluted to 200 c.c. and the wash water to be tested is added. H. F. HARWOOD.

Electrothermal production of phosphoric acid.

B. G. KLUGH (Chem. Met. Eng., 1929, 36, 666—669).—One large-scale plant using this process has been in successful operation in America since 1921, and another has just been started in Southern France. The furnaces are fed with phosphate rock, siliceous flux, and carbonaceous reducing agent continuously and slag and ferrophosphorus are tapped off periodically. The phosphorus vapour and carbon monoxide are burned by controlled admission of air and preheat the furnace feed. The reaction is said to be $(\text{CaO})_3\text{P}_2\text{O}_5 + 5\text{C} + \text{SiO}_2 = \text{P}_2 + 5\text{CO} + (\text{CaO})_3(\text{SiO}_2)_2$. The correct proportioning of silica and carbon is vital and the hearth temperature should not be so high as to volatilise lime, silica, etc. from the charge. The phosphorus pentoxide is partly cooled, sprayed with atomised water, and the acid (of 85% concentration) recovered by a Cottrell precipitator. The heat losses through the furnace walls do not exceed 5%. The efficiency of phosphorus recovery as acid in an example given is 87%. The power consumption appears to be about 2.5 kw.-hrs./lb. P_2O_5 , and a theoretical calculation shows a greater economy in the electric furnace than with coke firing. Whilst the ordinary sulphuric acid process may produce a somewhat cheaper phosphoric acid, the product is much less pure.

C. IRWIN.

Determination of fluorine in phosphorite. S. N. ROSANOV (Z. anal. Chem., 1929, 78, 321—325).—The presence of organic matter in phosphorites leads to high results in the determination of fluorine by Penfield's method, and its removal by a preliminary ignition is unsatisfactory, as 10% or more of the fluorine present may be lost in the process. A modification of Penfield's original method is recommended, 3—5 g. of chromic acid being added to the mixture of the phosphorite with silica before treatment with sulphuric acid to volatilise the silicon tetrafluoride. The carbon dioxide produced does not interfere with the subsequent titration.

H. F. HARWOOD.

Titrimetric determination of calcium in phosphorites. F. KAMIŃSKI (Przemysł Chem., 1929, 13, 505—507).—The finely-powdered phosphorite (5 g.) is digested for 10 min. at 100° with 12—15 c.c. of 25% hydrochloric acid, the resulting solution is made up to

500 c.c., and the whole is filtered. To 25 c.c. of the filtrate are added 10 c.c. of 10% ferric chloride solution and 10 c.c. of 20% sodium acetate solution, and to the red solution thus obtained, which should be clear, 120 c.c. of 4% ammonium oxalate solution are added and the mixture is boiled for 5–10 min. A voluminous precipitate separates, which on cooling is collected on a filter, washed seven times with water at 45°, and transferred to a flask. The residue on the filter-paper and the precipitate in the flask are dissolved in hot, dilute sulphuric acid, and the solution is titrated, using 0.1*N*-permanganate. The above method gives good results in the presence of phosphates, iron, aluminium, silica, and magnesia, and can be used for determining calcium in soil, ashes, etc. R. TRUSZKOWSKI.

Iodised salt. W. C. GEAGLEY (Amer. J. Pub. Health, 1929, 19, 991–996).—A method for mixing the iodide with calcium phosphate and then with salt is described. A colorimetric method for determining the iodine by extraction with carbon disulphide from a solution containing phosphoric acid and hydrogen peroxide is described.

CHEMICAL ABSTRACTS.

Highly active fuller's earth. A. SCHOLZ (Chem.-Ztg., 1929, 53, 899).—Clay from Southern Bavaria is made into a thin suspension with water, and this is pumped into a large boiler where it is digested with hot hydrochloric or sulphuric acid; the residue is collected in a filter-press, washed free from acid, dried, and ground. The product is very suitable for decolorising oils of all kinds, and 40,000 tons are used for this purpose in Germany annually. A. R. POWELL.

Absorption of nitrous gases. H. W. WEBB (Inst. Chem. Eng., Dec. 5, 1929, 34–42. Advance copy).—The reactions involved (cf. Partington and Parker, J.S.C.I., 1919, 38, 75 *r*) are: (A) $\text{N}_2\text{O}_4 + \text{H}_2\text{O} = \text{HNO}_3 + \text{HNO}_2$, (B) $3\text{HNO}_2 \rightleftharpoons \text{HNO}_3 + 2\text{NO} + \text{H}_2\text{O}$, (C) $2\text{NO} + \text{O}_2 \rightleftharpoons \text{N}_2\text{O}_4$. Rise of temperature has an unfavourable effect through promoting the dissociation of N_2O_4 molecules (NO_2 does not react with water) and through reducing the velocity coefficient of reaction C. Reaction B is accelerated, but as it is not the limiting factor this is of no importance. Reaction C is the slowest one, and the capacity of a system is largely dependent on this. Practical experience indicates that an inlet temperature of 15–40° is satisfactory. With concentrated gases the heat liberated by reaction C is considerable and cooling should be provided. The increase of partial pressure of N_2O_4 in the inlet gases would be advantageous, but methods, such as liquefaction, suggested to attain this are uneconomical. Increase of total pressure up to 4 atm., which should give a 16-fold increase of capacity as compared with atmospheric pressure, has been rendered possible by the introduction of acid-proof steels, and is said to have been used. Time-composition curves are given showing the time necessary for varying degrees of oxidation of nitric oxide in varying concentration. These indicate that in most cases a loss is inevitable unless a final alkaline wash-tower is provided. The reaction in packed and unpacked towers is essentially different, and with low nitric oxide concentration the use of empty towers may result in a loss of total capacity. The rate of

absorption of nitrogen peroxide by nitric acid decreases with increasing concentration of the latter. It is also affected by rate of circulation, distribution, and turbulence. The cost of absorption is a considerable difficulty in nitrogen-fixation processes. Possible advances may be the use of such absorbents as silica gel, and the suppression of reaction B by the use of some suitable agent to oxidise the nascent nitrous acid. C. IRWIN.

Methods of quantitative analysis for nearly pure gases. I. M. TRAUTZ, E. LEONHARDT, and H. SCHEUERMANN. II. M. TRAUTZ and K. KIPPAN. III. M. TRAUTZ, E. LEONHARDT, and K. KIPPAN (Z. anal. Chem., 1929, 78, 341–349, 350–364, 401–410).—I. A form of gas-analysis apparatus is described which permits of the determination with an accuracy of 0.05% of the nitrogen, oxygen, and inert gases present in liquid carbon dioxide, and of the nitrogen and inert gases present in compressed oxygen. Figures are also given showing the variation in composition of the residual gas obtained from successive fractions of liquefied carbon dioxide after absorption of this constituent.

II. The absorption of nitrogen by metallic calcium, strontium, and lithium has been investigated, and a method is described for the determination of small amounts of the inert gases in nitrogen, this latter being absorbed by metallic lithium at 300°. Small amounts of oxygen in nitrogen, carbon dioxide, or the inert gases may be determined by removing the first-named constituent from a definite volume of the gas by means of an electrically-heated iron spiral, and measuring the change in pressure. The electrolytic calcium prepared for the above work (purity 99.8%) had d_{25}^{25} 1.544, m.p. 803°; the strontium contained 3% of potassium and had d_{25}^{25} 2.52, m.p. 797° ± 1°.

III. The oxygen in compressed hydrogen can be determined by absorption with pyrogallol, and the nitrogen and inert gases by the methods previously given under carbon dioxide. The hydrogen can be determined with an accuracy of 0.05% by removing this constituent from a definite volume of the gas (previously dried and freed from oxygen) by diffusion through a tube of heated palladium, and measuring the change in pressure. In the determination of small amounts of hydrogen in nitrogen or the inert gases the above method cannot be used, and the hydrogen after diffusion through palladium must be pumped off and measured directly. A correction must also be applied for the nitrogen present, as palladium is somewhat permeable to this gas at high temperatures. Special forms of apparatus for carrying out the above methods are described.

H. F. HARWOOD.

Extraction of helium from monazite sand. R. TAYLOR (J.S.C.I., 1929, 48, 369–372 *r*).—Monazite sand is heated at 1000° in special heat-resisting steel pots in a stream of carbon dioxide. The gas is passed through copper oxide heated to 500° in order to oxidise hydrogen and carbon monoxide. After removal of carbon dioxide by caustic soda solution the gas is passed over metallic magnesium heated at 600° in order to remove nitrogen, and finally over metallic calcium heated at 580°, which removes the remaining impurities. Working drawings of the plant are given.

Catalysts for methyl alcohol synthesis. FENSKE and FROLICH.—See III. **Pallas alloy.** QUADRAT and JIRISTE.—See X. **Electrolysis of alkali chlorides.** BUNET.—See XI. **Titanium white.** VON BICHOWSKY.—See XIII.

PATENTS.

Manufacture of highly concentrated nitric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,728, 21.6.28).—Dilute nitric acid and nitrogen peroxide are brought in contact with oxygen at, e.g., 10 atm. pressure in a fractionating column containing fillers and maintained at temperatures ranging from 20–25° at the top to 100–125° at the bottom of the column. Nitric acid of varying concentrations is withdrawn as desired from sections at different temperatures, e.g., 99% at 88°, 90% at 105°, or fuming acid at 86°, and residual dilute acid is removed as vapour or liquid from the bottom of the column. L. A. COLES.

Removal of silicic acid from liquids or solutions. A. ROSENHEIM (B.P. 295,317, 4.5.28. Ger., 9.8.27. Addn. to B.P. 291,435; B., 1929, 798).—Titanic, tungstic, molybdic, or stannic acid, or thorium or zirconium oxide gels and/or gels obtained from previously formed zinc, cadmium, or magnesium oxide or hydroxide, or copper or aluminium hydroxide, are used together with or instead of the gels described previously. The material may be deposited upon, or arranged as alternating layers with, non-reacting organic or inorganic material. The exhausted gels are regenerated by treatment with tungstates or molybdates, with alkali salts, with hot water or steam, or with carbon dioxide solutions. L. A. COLES.

Production of compact masses from pulverulent metal oxides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,394, 29.5.28).—Mixtures of finely-divided metal oxides, e.g., ferrosiferrous oxide, with heavy metals, e.g., iron, nickel, copper, chromium, manganese, cadmium, which are also at least partly in a finely-divided condition, are heated in a non-reducing atmosphere and at a temperature above that at which the metal is oxidised but below the m.p. of the mixture. Alternatively, a mixture of a metal with an oxide of a higher stage of oxidation than that required is heated in a non-reacting atmosphere of, e.g., nitrogen. The products are suitable for use, e.g., as filter plates, diaphragms, or electrodes for alkaline accumulators. L. A. COLES.

Production of high-percentage, unfused lead monoxide. CHEM. FABR. COSWIG-ANHALT G.M.B.H., Assees. of VON DRAHTEN (G.P. 454,862, 26.10.26).—Lead carbonate is heated *in vacuo* in rotating ovens. L. A. COLES.

Recovering borax from brine. H. D. HELLMERS, Asst. to WEST END CHEM. CO. (U.S.P. 1,733,537, 29.10.29. Appl., 26.10.27).—Brine containing sodium carbonate and sodium borates is treated with carbon dioxide in such excess that the borax remains in solution. H. ROYAL-DAWSON.

Preparation of [mixed] ammonium and potassium phosphates. SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 300,919, 19.11.28. Fr., 19.11.27).—Potass-

ium chloride is added in small portions to orthophosphoric acid heated gradually from 100° to about 270°, and the product, after cooling to 70–80°, is treated with sufficient water and ammonia to yield the desired ammonium potassium phosphate, preferably $\text{NH}_4\text{K}(\text{H}_2\text{PO}_4)_2$, the temperature, which rises by the heat of reaction, not being allowed to exceed 150°. The product is obtained in the form of dry, non-hygroscopic crystals, which do not lose ammonia on storage. L. A. COLES.

Decomposition of raw phosphates. A. MESSERSCHMITT (B.P. 300,961, 12.11.28. Ger., 21.11.27).—Mixtures comprising raw phosphates, alkali sulphates (at least 1 mol. per 1 mol. of phosphoric acid) or minerals containing them, basic and/or acid material (alkaline-earth oxides, silica, alumina, silicates, etc.), and reducing agents, e.g., carbon and/or material containing sulphur, e.g., iron pyrites, in quantity sufficient only for partial reduction of the sulphate, are heated to sintering in an atmosphere neutral or slightly oxidising at first and strongly oxidising later, and under conditions such that the product retains its porous nature. Products containing citrate-soluble phosphates are obtained. L. A. COLES.

Production of alkali hydrosulphides from barium hydrosulphide and alkali chlorides. B. REINHARDT (G.P. 454,693, 22.5.24).—Barium hydrosulphide, prepared, e.g., by treating the sulphide with sufficient hydrochloric acid to convert it into a mixture of barium chloride and hydrosulphide, is treated at first with a small quantity of an alkali chloride, and, after removal from the solution of barium chloride mixed with a little alkali chloride, the decomposition is completed by the addition of excess alkali chloride. A mixture of barium and alkali chlorides crystallises, which, after removal, is used for treating fresh barium hydrosulphide solution. Residual barium salts are removed from the solution, e.g., by treating it with carbonates or carbon dioxide. Alkali sulphides are obtained, e.g., by heating the alkali hydrosulphide solutions. L. A. COLES.

Production of normal magnesium carbonate and other magnesium compounds from magnesites. T. HUGHES (B.P. 320,937, 26.7. and 30.8.28).—Dense, hard magnesite in lumps 6–18 in. in length is treated with, e.g., 20% sulphuric acid under conditions such that mechanical disintegration is avoided; after the reaction has proceeded for some time, the liquor is circulated through a strainer to remove silica etc., and then through a series of tanks in which suspended particles of magnesium carbonate settle out, and, after the addition of some fresh acid, is returned to the magnesite. The lumps are subsequently washed with magnesium sulphate solution under pressure to remove adhering magnesite particles. The product is suitable for pharmaceutical use, for the manufacture of fire-proofing and refractory surfaces, and, after grinding in a colloid mill, for the manufacture of water- or oil-paints etc. L. A. COLES.

Production of anhydrous aluminium chloride. T. R. HAGLUND (Swed. P. 60,424, 17.4.24).—Aluminium sulphide or material containing it is heated in the presence of chlorine or, e.g., hydrogen, ammonium,

sodium, or barium chloride, at a temperature sufficiently high for the production of aluminium chloride.

L. A. COLES.

Manufacture of chromic compounds [containing hydrated chromic oxide]. E. BAUMGARTNER (B.P. 294,965, 2.8.28. Fr., 2.8.27).—Chromium sulphate solutions (about 20% Cr_2O_3 ; basicity about 66.6), prepared preferably by treating dilute sodium chromate solution with sulphur dioxide and adding solid sodium chromate in small quantities as the reaction proceeds, are treated with an alkali (sodium) compound, *e.g.*, sodium hydroxide, carbonate, bicarbonate, sulphite, formate, acetate, or chromate, or with ammonia. After the reaction has proceeded, *e.g.*, in accordance with the equation, $\text{Cr}_2\text{O}_3 \cdot \text{SO}_3$ (hydrated) + $2\text{Na}_2\text{CrO}_4 = \text{Cr}_2\text{O}_3$ (hydrated) + $\text{Na}_2\text{Cr}_2\text{O}_7$ + Na_2SO_4 , the product sets to a crystalline mass from which the sodium sulphate and part of the dichromate are removed by washing with water. The use of an excess of sodium chromate for the precipitation yields a product, approx. $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{Cr}_2\text{O}_3$ (hydrated), of use as an anti-rust paint; other products are of use in tanning, dyeing, etc.

L. A. COLES.

Production of hydrogen [from mixtures of it with carbon monoxide]. SOC. ANON. BREVETTI "CICALI" (B.P. 291,409, 31.5.28. Ital., 1.6.27).—Water-gas, coke-oven gas, etc., after preliminary removal of hydrogen sulphide, carbon dioxide, etc., is partially liquefied and scrubbed with liquid nitrogen (containing traces of liquid hydrogen) in the lower section of a high-pressure rectifying column to condense the greater part of the carbon monoxide. The residual gas is rectified in the upper section of the column by treatment with a partially-liquefied mixture of hydrogen and nitrogen having approximately the same composition as the gases in the top portion of the column; this is produced externally and introduced into the top of the column at a temperature below the solidifying point of nitrogen. Hydrogen mixed only with small quantities of nitrogen is drawn off from the top of the upper section (*cf.* B.P. 259,643; B., 1927, 43). L. A. COLES.

Reducing the inflammability of hydrogen. KOKUKENKYUJO (B.P. 294,958, 9.7.28. Jap., 2.8.27).—Hydrogen is mixed with 0.5–2% of the vapour of one or more compounds, *e.g.*, petroleum hydrocarbons, benzene, alcohols, having a flame-propagation temperature above that of hydrogen.

L. A. COLES.

Production of oxides of nitrogen from the air. A. GASCHLER (G.P. 454,694, 17.6.25).—Air is passed through an electric arc struck between mercury electrodes to which mercury oxide or other oxides may be added. Mercury volatilised during the process is condensed and recovered during the scrubbing of the gases.

L. A. COLES.

Manufacture of readily condensable nitric oxides [nitrogen peroxide]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,425, 7.7.28).—Gas mixtures containing a high concentration of nitric oxide and, if desired, nitrogen peroxide, produced, *e.g.*, by the acidification of solutions containing nitrates and nitrites, by the treatment with sulphur dioxide of mother-liquors from the manufacture of potassium nitrate, by the decomposi-

tion of nitrosyl chloride, or by heating ferrous sulphate solutions which have been used for the absorption of nitric oxide, are treated with 20–68% nitric acid, preferably in countercurrent, at 40–110°. Mixtures of nitrogen peroxide and water are obtained from which the water is removed by condensation under conditions such that the nitrogen peroxide is brought as little as possible in contact with the condensed water, after which the nitrogen peroxide is condensed at 10–15°, and the liquid is treated with water and oxygen for the direct production of concentrated nitric acid.

L. A. COLES.

Manufacture of ammonia. C. URFER, Assr. to SOC. D'ETUDES MINIÈRES & INDUSTRIELLES (U.S.P. 1,737,953, 3.12.29. Appl., 25.1.27. Fr., 6.7.26).—See B.P. 274,023; B., 1928, 522.

Purification of crude sodium sulphide. R. BOTSON (U.S.P. 1,736,741, 19.11.29. Appl., 26.11.27. It., 24.10.27).—See B.P. 289,354; B., 1928, 446.

Production of [sodium] perborate. ROESSLER & HASSLACHER CHEM. CO. (B.P. 297,777, 26.9.28. U.S., 27.9.27).—See U.S.P. 1,716,874; B., 1929, 643.

Manufacture of calcium hypochlorite. L. MELLERSH-JACKSON. From MATHIESON ALKALI WORKS (B.P. 320,182, 321,250, and 321,720, 18.8.28).—See U.S.P. 1,718,284, 1,718,287, and 1,718,285; B., 1929, 718.

Conversion of lead sulphate and lead chloride into lead carbonate. S. C. SMITH, Assr. to CHEM. & MET. CORP., LTD. (U.S.P. 1,738,081, 3.12.29. Appl., 13.6.27. U.K., 29.6.26).—See B.P. 272,053; B., 1927, 555.

Sulphur from roaster gases (U.S.P. 1,734,991).—See X.

VIII.—GLASS; CERAMICS.

Effect of boric oxide on the rate of melting and on some physical properties of colourless bottle glasses. V. DIMBLEBY, M. PARKIN, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1929, 13, 248–258).—The rate of melting of soda-lime-silica and soda-lime-alumina-silica glasses, of the type used in the automatic manufacture of bottles, is appreciably and progressively increased, whilst the rate of refining is distinctly quickened, by the addition of increasing amounts of crystalline borax to the batch. Glasses so prepared show increased resistance to the action of water, and have a lower rate of thermal expansion.

A. COUSEN.

Devitrification of glass. J. J. KITAIGORODSKY (J. Soc. Glass Tech., 1929, 13, 219–229).—Tests consisted in subjecting small samples of clear molten glass of varying composition to desired temperatures for 3–12 hrs. From the final appearance of the glasses the temperature-devitrification range of each group was determined. It is concluded that (1) the presence of alumina in glass does not always tend to prevent devitrification; (2) the effect of alkalis is to reduce, and of lime to increase, the tendency to devitrification; (3) magnesia (from magnesite) when substituted for lime in a standard glass increases the crystallising capacity; (4) glasses containing a large number of constituents have a lower tendency to crystallisation than simpler glasses.

A. COUSEN.

Homogeneity of small-scale glass meltings in platinum. V. DIMBLEBY, H. W. HOWES, and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 243—247).—Small-scale preparations of glass in platinum or its alloys required at least one remelting with an intermediate crushing and mixing to secure homogeneity, lack of which was not found to be due to volatilisation of alkali. Dissolution of platinum or rhodium by the glass was reduced by previously sintering the batch in a silica crucible. A. COUSEN.

Photo-elastic properties of glass. F. C. HARRIS (J. Soc. Glass Tech., 1929, 13, 213—219).—The presence of large proportions of lead oxide in a glass causes a reversal of normal stress-optical effects, leading to the conclusion that the glass is a colloid in which particles of one constituent are scattered amongst those of the others. Stress-optical coefficients of a number of glasses remeasured after 20 years showed apparent increases in every case. A. COUSEN.

Analysis of gases emitted by glass. S. KONDRASHEVA (J. Appl. Phys., Moscow, 1928, 5, 23—38).—Below the softening point the glasses examined evolved gases which are vigorously absorbed by alkali oxides; above this point hydrogen and carbon monoxide are liberated. CHEMICAL ABSTRACTS.

Principal structural features of raw materials for silica bricks. P. JUDINSON (Feuerfest, 1929, 5, 165—169).—In European and American practice two types of raw quartz, differing greatly in structure, are used in making silica bricks of good quality. The first group includes materials of the English ganister type, *i.e.*, quartzites consisting of closely-packed angular grains and containing no bonding material. The second group, represented by the German "findlings" quartzites, consists of irregular and frequently corroded grains cemented together, usually by a cryptocrystalline, more rarely by an amorphous siliceous, bond. Twelve typical silica raw materials were examined under the microscope in polarised light; the results are tabulated and photomicrographs are reproduced. Despite differences in structure, certain similarities are observed: the quartz grains are small, varying from 0.01 to 0.80 mm. in diam., with a preponderance of grains between 0.10 and 0.25 mm.; foreign materials are rare, and are finely distributed throughout the mass; all the materials show the effects of powerful dynamometamorphic processes, in which lateral pressures have altered the optical properties of the materials, so that the quartz grains show a decidedly wavy extinction. Special emphasis is laid on this last point. The silica in raw materials containing such metamorphosed quartz grains is more readily inverted to its high-temperature modifications. A good silica brick is structurally composed of a lattice or skeleton of tridymite, containing an amorphous glass and crystals of cristobalite, quartz, and silicates. Quartzites used as raw materials for silica bricks should be such as consist of quartz grains bonded together by an amorphous or cryptocrystalline cement, or such as have undergone dynamic metamorphosis, in which the quartz grains show a wavy extinction. The structure should be dense and the grain size uniform, and the greater portion of the

grains should have an average diameter of 0.3—0.4 mm. The material should contain at least 95—96% SiO_2 , not more than 1.5% Al_2O_3 , and not more than 0.4% R_2O . Foreign matter and accessory minerals should be evenly distributed, and should not exceed a few per cent. of the whole. F. SALT.

Thermal insulation of brick, tile, and pipe kilns. C. PRESSWOOD (Trans. Ceram. Soc., 1919, 28, 436—444).—The advantages of thermal insulation are pointed out, and data on furnace wall construction for different working temperatures are presented. F. SALT.

New apparatus for determining degrees of fineness, and some applications of this apparatus to various ceramic materials. S. BERG (Trans. Ceram. Soc., 1929, 28, 427—435).—The methods suggested by Andreasen (B., 1928, 915) have been applied to ceramic problems. The apparatus consists of a cylindrical, glass sedimentation vessel about 50 cm. deep and 10 cm. diam. The tube of a pipette passes through the cover of this vessel and fits into a small groove at the bottom. The lower end of the pipette tube is closed by a rubber hood, and 4 cm. from this end the tube has two oppositely arranged lateral openings. Between the tube and the bulb of the pipette is fitted a two-way tap, which communicates both with the tube and with an outlet. A rubber brush with a hollow glass handle is used for agitating the suspension. The glass vessel is filled to a given mark with the suspension under test, the suspension is thoroughly agitated, air being simultaneously blown in through the brush handle, a pipetteful is quickly drawn off, run into a dish, evaporated, and weighed, to determine the dry contents of the suspension. The suspension is then thoroughly agitated again. At given intervals from the time agitation ceased pipettefuls are withdrawn, and their dry contents indicate the quantity of material having a smaller grain-size than that corresponding to the falling velocity h/t , h being the mean height of the liquid above the two lateral openings in the tube, and t the sedimentation time. The effects of coagulation are shown, and the means adopted to avoid this phenomenon are described. F. SALT.

Measurement of the viscosity of glass at high temperatures by the rotating-cylinder viscosimeter. R. F. PROCTOR and R. W. DOUGLAS (J. Soc. Glass Tech., 1929, 13, 194—213).—See B., 1929, 851.

Progress report on investigation of fireclay bricks and the clays used in their preparation. R. A. HEINDL and W. L. PENDERGAST (Bur. Stand. J. Res., 1929, 3, 691—729).—See B., 1929, 979.

PATENTS.

Polishing of glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 292,104, 11.6.28. Fr., 13.6.27).—Hard abrasives, such as are used for grinding or smoothing glass, are rendered suitable for use as polishing media by moist grinding to extreme fineness, converting into an aqueous suspension of suitable p_H value, and, after allowing coarser particles to settle, siphoning or decanting and finally flocculating the fine-grained material. A. COUSEN.

Manufacture of refractory materials having a base of bauxite. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 294,179, 18.7.28. Fr., 19.7.27).—Mixtures containing bauxite, refractory clay, etc. with not more than 2% of an alkaline-earth or alkali fluoride or fluosilicate as catalyst are moulded, dried, and calcined at a temperature not lower than that to which the product is to be subjected subsequently. L. A. COLES.

Refractory insulating brick. W. H. RAMAGE (B.P. 321,188, 1.8.28).—A plastic mixture of equal weights of fireclay and sawdust, preferably from hard wood, is moulded under pressure and burned. C. A. KING.

Abrasives and method of treating the same. CARBORUNDUM CO., LTD. FROM CARBORUNDUM CO. (B.P. 321,240, 8.8.28).—An agglomeration of abrasive particles held together by means of a binder is impregnated or covered with a lubricant which possesses the property of readily spreading over the surfaces of the abrasive particles. Suitable lubricants are organic fatty acids, their hydrolysable compounds, and sulphonated oils, e.g., sulphonated pine oil. C. A. KING.

IX.—BUILDING MATERIALS.

Compression and bending strengths, contraction and expansion, resistance to abrasion, permeability to water, and resistance to chemical attack of cement mortars and concrete with varying grain size and water content of the mortars. O. GRAF (Mitt. Materialprüf., 1929, 70—72).—The highest strength of sand mortars is obtained when 25% passes a 0.2-mm. screen, 40% a 1-mm. screen, and 65% a 3-mm. screen. The usual method of grading the sand by adding to the coarse material sufficient of the fine sand to fill the hollow spaces is not recommended as it frequently produces poor mortars. The compression and bending strengths of concrete depend on the strength of the mortar, and are scarcely affected by the grain size of the ballast (7—40 mm.). During drying the compression strength of concrete increases whilst its bending strength diminishes; the relation of the former to the latter is 1:3—12 in mortar and 1:5—12 in concrete. Addition of calcium chloride increases the contraction of cement mortars in drying. The resistance to wear decreases with an increase in the content of fine sand, and the relation between the water/cement ratio and the resistance to abrasion in a sand blast is independent of the cement content of the mixture. The grain size of the sand has a considerable influence on the permeability of cement and mortar, the most impermeable mixture being that mentioned above. Concrete is more permeable to water in a direction at right angles to the direction of stamping than in a direction parallel thereto. Addition of trass retards the action of magnesium salts on mortars.

A. R. POWELL.

Relation between mortar strength and concrete strength. BURCHARTZ (Mitt. Materialprüf., 1929, 72).—The volume, density, and compression strength after 28 days of 20-cm. cubes of mixtures of cement (1 vol.), sand (1—3 vols.), and gravel (0—5 vols.) have been determined. With slightly moistened mixtures the

addition of a small proportion of gravel increases the strength, but further quantities reduce it again; with wetter mixtures addition of even a small proportion of gravel decreases the strength, the reduction being proportional to the amount of gravel in the mixture. No definite relation between the density and compression strength of concrete could be established.

A. R. POWELL.

Influence of artificial ageing on the mechanical properties of woods. R. LYON, G. FRON, and FOURNIER (Compt. rend., 1929, 189, 992—995).—The action of weakly ozonised air on green wood for a month produces all the microscopical and chemical characters of old wood. The mechanical properties of the wood as expressed in terms of its unit resilience, maximum unit tension, and variation in volume with 15% of water (shrinkage) were of the same order for woods (beech) lightly ozonised or artificially aged for 4 years. Woods strongly ozonised for a week to a month are classed as of medium strength, though shrinkage is not substantially modified by ageing. The suitability of such woods for aircraft purposes is discussed. J. GRANT.

Waterproofing medium. BRAUN.—See XII.

PATENTS.

Fusing or calcining rock materials for production of cements, limes, or like substances. W. FAWCETT (B.P. 321,205, 31.7.28).—Component materials of cement are fed through a stationary, electrically heated zone by means of a forced draught. A number of resistance elements situated between the inner and outer walls of a kiln comprise the heating element.

C. A. KING.

Manufacture of cement. K. BALTHASAR, R. KRAUSE, A. ZINGL, K. DERFLINGER, and J. ORTIS (B.P. 309,069, 31.1.29. Austr., 4.4.28).—Portland cement of high initial setting strength is made from materials containing magnesia, e.g., blast-furnace slag, by intensifying the calcination to above 1450° for at least 20 min., to produce a clinker containing 5—15% MgO.

C. A. KING.

Cementitious material [for manufacture of glazed bricks, building blocks, etc.]. A. METHERELL, Assee. of G. E. BARNHART (B.P. 299,427, 22.10.28. U.S., 26.10.27).—The use is claimed of solutions containing magnesium and calcium chlorides, sodium or potassium silicate, and aluminium sulphate or an alkali aluminium sulphate, with or without the addition of magnesium sulphate.

L. A. COLES.

Hardening cement or binder. BINDPHAST PRODUCTS, LTD., and J. E. HACKFORD (B.P. 321,646, 6.6.28).—As binder a mixture of calcined dolomite (35 pts.) and magnesium chloride (12.5 pts.) is claimed.

C. A. KING.

Production of oxychloride cements. J. BAXTER and H. P. FLETCHER (B.P. 314,004, 14.8.28. U.S., 21.6.28).—Magnesium or zinc chloride is treated in aqueous solution with an oxidising agent, e.g., an alkali manganate or permanganate, hydrogen peroxide, before admixture with magnesium or zinc oxide.

L. A. COLES.

Acid-resisting cement. SCHMELZBASALT-A.-G., and C. TRENZEN (G.P. 453,896, 28.8.26. Addn. to G.P. 435,913; B., 1927, 412).—Finely-divided carbon, such as soot, is added to the mixture specified in the chief patent. A. R. POWELL.

Concretes or plastic masses comprising cement and sawdust. B. J. KOPELOWICZ and L. KOPELOWICZOWA (B.P. 321,744, 23.8.28).—Sawdust to be mixed with cement for concrete is boiled in lime water to remove fats and resins, which are skimmed off. In order to counteract differential contractions during the setting period, nettings of long straw are introduced into the cast mass at intervals. C. A. KING.

Manufacture of [artificially weathered] bricks. CAFFERATA & Co., LTD., and B. J. CAFFERATA (B.P. 321,593, 22.12.28).—Green bricks, after a preliminary drying, are immersed for 2–3 min. in a dilute mineral acid before they are burned. L. A. COLES.

Fireproofing of wood and like material. A. EICHENGRÜN (B.P. 321,070, 14.11.28).—The material is impregnated with alcoholic ammonium bromide solution, the solvent subsequently being expelled by heating. L. A. COLES.

Impregnation of wood with rubber. J. R. COOLIDGE, Assr. to MONTAN, INC. (U.S.P. 1,733,483, 29.10.29. Appl., 23.1.26).—Rubber latex is forced into the pores and tracheids of the wood, sufficient alkali being mixed with the latex to neutralise any acid present in the wood at those points. H. ROYAL-DAWSON.

Producing imitations of valuable kinds of wood, tarsia work, etc. J. WERNER, and F. SCHLOBACH GES. M.B.H. (B.P. 303,836, 10.1.29. Ger., 10.1.28).—Transfer pictures are applied under a pressure of at least 20 kg./cm.², and without heating, to wood surfaces which have been smoothed by hot compression or rolling and coated with a thin layer of a paste comprising zinc oxide, a binder, and, if desired, a diluent, the paste being dried before application of the transfer. L. A. COLES.

Transferring veining or graining of wood on to printing plates and the like. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 321,591, 22.12.28).—The pores of mahogany etc. are filled, e.g., by treating the wood with a mixture of alcohol, turpentine, shellac, and coloured pumice meal, and the surface is, if desired, impregnated with oil and polished before the wood is photographed for the production of printing plates etc. L. A. COLES.

Manufacture of stable, aqueous [bituminous] emulsions. C. A. BRAUN, Assr. to AMER. BITUMULS Co. (U.S.P. 1,737,491, 26.11.29. Appl., 5.12.25. Ger., 21.3.25).—See B.P. 254,012; B., 1926, 748.

Bituminous emulsions (B.P. 320,847 and 321,334).—See II. Paper sacks (B.P. 307,005).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Corrosion testing apparatus. D. F. OTHMER (Ind. Eng. Chem. [Anal.], 1929, 1, 209).—The apparatus consists of a glass column, packed with glass beads and fitted with a condenser, which is inserted into an electri-

cally heated flask containing the corroding liquid, the weighed pieces of metal under test being inserted at various heights in the column. It is claimed that the rate of corrosion can thus be studied under reproducible conditions, and that comparative tests on different metals have a greater value in determining their relative usefulness; in the latter case care must be taken to insert the samples at the same height in the column.

H. F. HARWOOD.

Corrosion of cemented or nitrated steels. L. GUILLET and M. BALLAY (Compt. rend., 1929, 189, 961–963).—Nitridation increased the corrodibility of steels by 5% (by vol.) sulphuric or hydrochloric acid with the exception of a steel containing 4.08% Ni and 0.91% Cr, the resistance of which towards the former acid was increased. For the chromium-aluminium steels cementation had the same effect. The resistance to sea-water containing 5% of 12-vol. hydrogen peroxide and to a mist from 20% sodium chloride solution was increased by nitridation more than by cementation followed by tempering, especially in the cases of the chromium and aluminium steels. This effect was even more marked with Seine water, especially for nickel steels. Cementation slightly increased the resistance towards 10% copper sulphate solution, but the nitrated steels were hardly attacked. The control steels were all either tempered in oil at 900° with recovery at 700°, or annealed at 750–800°.

J. GRANT.

Heat treatment of complex ferro-nickels containing two constituents. P. CHEVENARD (Compt. rend., 1929, 189, 846–849).—The addition of another element such as copper or aluminium to ferro-nickel alloys results in the formation of two phases, the mutual solubility of which increases with temperature. These alloys show, after quenching, the same ageing properties as duralumin, the slow precipitation of a hard constituent from the supercooled solid solution resulting in improved mechanical properties. The alloys show a hysteresis loop in the dilatation-temperature curve.

C. J. SMITHIELLS.

Determination of small amounts of nickel in steel. B. JONES (Analyst, 1929, 54, 582–589).—The method used depends on the red-brown colour given by the action of an oxidant on the dimethylglyoxime complex of nickel, and will detect 1 pt. of nickel in 10⁷. For plain carbon steels 1 g. is dissolved in hydrochloric acid, the solution is oxidised with nitric acid, and, after dilution, ammonia solution (1:1) is added until the precipitate just fails to redissolve. 2 c.c. of a 1% solution of potassium cyanide are added, and, after shaking, 10 c.c. of 1:1 ammonia solution. The liquid is made up to 200 c.c., mixed, the precipitate collected on a filter, and 100 c.c. of the filtrate are used for the determination, 50 c.c. being also required for an approximate quantitative test made by adding to 2 c.c. of dimethylglyoxime in alcohol 1 c.c. of sodium hypochlorite solution and matching the colour developed with a standard. This is made by adding to the water in the Nessler glass 5 drops of 1:1 ammonia solution, 2 c.c. of dimethylglyoxime solution, and 1 c.c. of sodium hypochlorite solution. Three drops of a standard nickel solution containing 0.00005 g. Ni per c.c. are run in, the

mixture is stirred, and more nickel solution added till the colour is matched. Meanwhile any copper present in 100 c.c. of filtrate is removed, and the test is repeated with a suitable volume of filtrate containing not more than 0.1 mg. Ni. Manganese up to 2% does not interfere; chromium, molybdenum, aluminium, and vanadium are removed by the ammonia; tungsten is quantitatively removed in the iron precipitate; and copper gives an interfering colour which is overcome by addition of 1 c.c. of the potassium cyanide solution, whereby the colour is nullified temporarily. Certain modifications in the process are described for high-manganese and -cobalt steels. D. G. HEWER.

Influence of a third metal on the constitution of brasses. II. **Nickel.** O. BAUER and M. HANSEN (*Z. Metallk.*, 1929, **21**, 357—367, 406—411).—Previous work on the ternary system zinc-copper-nickel is reviewed and the results are recorded in a series of equilibrium diagrams of thermal and micrographical investigations on alloys containing 30–60% Cu and 20–0% Ni. In alloys with more than 2.5% Ni, the α -($\alpha + \beta$) boundary is displaced towards the zinc end of the equilibrium diagram, and above 450° it becomes concave to the temperature axis, the degree of displacement increasing with increasing nickel content. Hence these alloys after quenching from 600–800° age-harden on tempering above 100°. As in the binary zinc-copper system, the β -field of the ternary system becomes broader with rise of temperature; at 0–400° the coefficient of equivalence of nickel in the β -phase is -1.1 , but between 0 and 10% Ni this coefficient is only -0.15 . The α -phases in the nickel-zinc and nickel-copper systems are isomorphous, so also are the β -phases; as, however, the β -phase in the former system is stable only above 640° the β -phase of the ternary system in certain ranges of concentration below 30% Cu is stable only at temperatures above the normal, and hence the ternary β -field becomes smaller at 20° with decreasing copper content. With less than about 20% Cu the β -phase decomposes into the eutectoidal ($\alpha + \gamma$) at the ordinary temperature, so that there must be a region of ($\alpha + \beta + \gamma$) in the ternary system. In all probability the γ -phases in the two binary systems are isomorphous. Numerous photomicrographs showing the characteristic structures of α , ($\alpha + \beta$), β , ($\alpha + \beta + \gamma$), and ($\alpha + \gamma$) are reproduced and the effect of heat treatment on these structures is shown. Nickel affects the hardness of α -alloys only slightly, but it increases the hardness of ($\alpha + \beta$)- and β -alloys and to a much greater extent the hardness of alloys containing the γ -constituent. A. R. POWELL.

Sampling and analysis of bronzes and other alloys which tend to segregate. O. BAUER and E. DEISS (*Z. anal. Chem.*, 1929, **79**, 47—53).—A rectangular bronze ingot of the approximate composition Cu 85%, Sn 5%, Zn 7%, Pb 3%, was cut into vertical and horizontal slices, and each section was analysed. The results showed that whilst the percentage of zinc remained practically constant throughout the ingot, variations up to 1.69% occurred in the copper content, 1.19% in the tin, and 0.74% in the lead. The content of copper was least in the outer layers and increased towards the centre of the block, whereas with the tin and

lead the reverse was the case. Of the minor constituents of the alloy, antimony was present in the largest amount in the outer layers, iron, nickel, and sulphur being almost uniformly distributed throughout. It is concluded that no satisfactory representative sample can be obtained by any method of boring from ingots of alloys which tend to segregate. It is recommended that the ingot should be sawn in two perpendicular to its long axis, and that a thin layer of shavings should be removed from one of the surfaces thus exposed; the whole of these shavings must be dissolved subsequently for the analysis.

H. F. HARWOOD.

Corrosion tests with aluminium and duralumin in sea-water. CORROSION COMMITTEE OF THE DEUTSCHE GES. F. METALLKUNDE (*Z. Metallk.*, 1929, **21**, 378—386).—The appearance and loss in weight of sheets of aluminium and duralumin, with and without welds and with and without protective coatings of other metals or oxide films, after exposure for 10 months to North Sea water and 8 months to Baltic Sea water are recorded. Duralumin, owing to its greater tendency to local pitting, is much less resistant to sea-water than is aluminium, but its resistance can be considerably increased by careful heat treatment. Both aluminium and duralumin are protected for a long time by chromate films or by oxide films electrolytically produced, but there is a tendency for bubbles to develop in the films owing to penetration of the salt water through minute imperfections. Welding has little effect on the resistance to sea-water of pure aluminium, but it decreases considerably that of duralumin unless the welded sheet is subjected to the usual heat treatment and ageing. A. R. POWELL.

Brinell hardness, elasticity, and tensile strength of ageable aluminium alloys. H. BOHNER (*Z. Metallk.*, 1929, **21**, 387—389).—The hardness of aluminium alloys increases with the load and remains constant for a given load with a time of application between 5 and 300 sec. except when the load is very high, in which case the hardness decreases with the time of application to a constant value. Under a load equal to $5D^2$, where D is the diameter of the ball, the Brinell hardness of these alloys is equal, irrespective of the thermal treatment, to twice the sum of the elastic limit and the tensile strength, provided that the alloy has been rolled or drawn and that it has an elongation of 11–24%.

A. R. POWELL.

High-temperature allotropes of manganese. M. L. V. GAYLER (*Nature*, 1929, **124**, 840).—Photomicrographs show that manganese heated in hydrogen for 2 hrs. at 975° or 1075° is unchanged, whilst that heated in hydrogen for 2 hrs. at 1220° suffers a change in structure (cf. Persson and Öhman, *B.*, 1929, 899).

A. A. ELDRIDGE.

Pallas alloy as a substitute for platinum. O. QUADRAT and J. JIRÍŠTĚ (*Chem. Listy*, 1929, **23**, 493—496).—Cathodes of pallas alloy (palladium, platinum, and gold) gauze can advantageously be employed in place of those of platinum; in nitric acid a slight corrosion takes place initially, but the metal soon acquires passivity. Crucibles made of this alloy are more resistant to the action of fused alkali carbonates and of hot concentrated sulphuric acid than are those of

platinum, and the alloy is, further, completely unattacked by fused borax and by hydrofluoric acid. The greatest corrosion is caused by fused potassium hydrogen sulphate. The alloy is harder, cheaper, and lighter than platinum. R. TRUSZKOWSKI.

Chromium plating of metals. W. BIRET (Z. Metallk., 1929, 21, 372—377).—A lecture dealing with the methods used in chromium plating, preparation of the material, and properties of chromium-plated articles, e.g., resistance to heat, corrosion, and wear.

A. R. POWELL.

Electrodeposition of tin. C. H. PROCTOR (Metal Ind., N.Y., 1929, 27, 267—268).—The bath, containing sodium stannate (339.5 g.), acetate (56.7 g.), hydroxide (28.3 g.), and borate (3.5—7 g.), in water (3.79 litres), is used at 71—91° with 4—6 volts.

CHEMICAL ABSTRACTS.

Corrosion of metals by milk. MCKAY and others. —See XIX.

PATENTS.

Regenerative [open-hearth] furnace. F. E. KLING (U.S.P. 1,735,687, 12.11.29. Appl., 28.2.22).—At each end of the hearth are three ports, and when working as inlets the outer ones are used for air alone and the central one for the air-gas mixture. The supply uptakes for air communicate mainly with the outer ports, and that for gas only with the central port. The air uptakes have a constriction causing a rise of pressure, and from that zone auxiliary passages lead to the central port. B. M. VENABLES.

Oil-burning [metallurgical] furnace. G. NIEMKOFF, Assr. to SOC. ANON. DES APPAREILS DE MANUTENTION ET FOURS STEIN (U.S.P. 1,736,415, 19.11.29. Appl., 19.8.21).—A vertical duct provided with a cap covering leads into the intake passage of the furnace. Means are provided for feeding liquid fuel (by gravity) through the cap, the supply pipe having a liquid seal and sighting device. C. A. KING.

Cleaning of blast-furnace gases or the like. H. A. BRASSERT & Co. (B.P. 307,904, 21.11.28. U.S., 17.3.28).—The gas from the usual cyclone dust-catcher is passed successively through a vortex cleaner, a vertical tower with water-spray jets, a disintegrator which agitates the gas with water, and a vortex moisture eliminator. A. R. POWELL.

Utilisation of sulphur-bearing gases from ore-roasting or -sintering operations. NAT. PROCESSES, LTD., and T. B. GYLES (B.P. 321,390, 27.7.28).—Gases poor in sulphur, evolved towards the end of a sulphide-roasting operation, are conducted over or through a moistened sinter obtained from the roasting of ores; e.g., zinc sulphide may be roasted and the gases of low sulphur content passed over the residue from a previous roasting operation. The sintered material may then be mixed with fresh batches of ore and returned to the desulphuriser, or may be leached with water or dilute acid to recover the soluble salts. C. A. KING.

Recovery of sulphur from roaster gases. R. F. BACON (U.S.P. 1,734,991, 12.11.29. Appl., 6.5.27).—The gases are treated under pressure with hot reducing agents to liberate free sulphur. F. G. CLARKE.

Froth flotation concentration of ores etc. P. T. WILLIAMS, and MINERALS SEPARATION, LTD. (B.P. 321,927, 18.8.28).—The end cells of a series are provided with auxiliary outlets for dilute pulp just below the froth level, and the effluents from these are returned to an earlier cell in the series. Flow of slimes towards cells lower in the series is prevented by the introduction of fresh water into one or more of the lower cells either into the auxiliary outlets or at points near them.

A. R. POWELL.

Binder for foundry cores. A. STAHN, Assce. of K. MICHAELIS (B.P. 303,858, 7.1.29. Ger., 11.1.28).—A binder for foundry cores of pure quartz sand comprises 2—4% of powdered boric acid with or without a small quantity of phosphoric acid and the usual organic binder. The cores are dried at 150—250° so that a glassy mass is formed round the sand grains; after casting, the core may readily be broken out.

A. R. POWELL.

Moulds for casting metals. METALLGES. A.-G., V. M. GOLDSCHMIDT, and K. STENVIK (B.P. 321,609, 24.1.29).—Magnesium orthosilicate is an excellent moulding material for metals of high m.p. or which are chemically reactive. Natural olivine, peridotite, or dunite in the form of powder is mixed with a binder, shaped, and heated to form a mould. C. A. KING.

Manufacturing, from by-products, ferrous bricks for use as iron ore in various metal furnaces. SOC. LORRAINE DES ACIÉRIES DE ROMBAS (B.P. 295,051, 3.8.28. Fr., 6.8.27).—A mixture for making into bricks consists of the dust from the mouth of a blast furnace, coke dust, a ferrous flux (fuel ash), and water. The bricks are fritted in an ordinary brick kiln, chiefly by combustion of the coke in the briquettes.

C. A. KING.

Manufacture of steel. F. W. DAVIS, Assr. to S. G. ALLEN (U.S.P. 1,735,074, 12.11.29. Appl., 2.6.24).—In the open-hearth process operations are normal until the slag is removed. The process is then completed under non-oxidising conditions.

C. A. KING.

Hardening iron and steel articles by nitrogenisation. P. F. M. AUBERT, A. J. P. and H. A. M. DUVAL (AUBERT & DUVAL FRÈRES) (B.P. 300,633, 24.10.28. Ger., 17.11.27).—Parts of iron and steel articles which are not to be hardened in the nitrogenising process are painted with a solution of sodium silicate, then with (a) potassium chloride, (b) a mixture of 2 pts. of barium chloride, 1.5 pts. of potassium chloride, 1 pt. of calcium chloride, and 1.25 pts. of sodium chloride, or (c) a finely-ground flux made by melting a mixture of 70% of lead oxide, 20% of silica sand, and 10% of borax.

A. R. POWELL.

Rustproofing of iron and steel articles. W. H. ALLEN, Assr. to PARKER RUST PROOF CO. (U.S.P. 1,735,842, 19.11.29. Appl., 11.8.28).—The articles are dipped into a solution containing chromium and phosphate radicals, and are then heated sufficiently to convert the chromium phosphate into the pyrophosphate.

H. ROYAL-DAWSON.

Manufacture of tinned metal products. Annealing or coating of metals. Manufacture of metal

products covered with a coating of metal (e.g., zinc) or alloy. W. HANBY (B.P. 321,638, 321,642—3, [A—C] 3.8.28, [B, C] 19.12.28).—(A) Sheet iron or steel is annealed and cleaned prior to tinning by immersion at 500—1200° in a bath comprising (a) 60% of sodium chloride, 20% of sodium cyanide, and 20% of sodium carbonate, or (b) 80% of barium chloride, 15% of calcium chloride, and 5% of potassium chloride. (B) Apparatus for carrying out the annealing and tinning operations comprises a bath of the fused salts, a water-bath or series of sprays for washing the annealed goods, and the usual tinning bath, sinuous guides being provided to carry the goods continuously through the three baths. (C) The operation described in (A) is claimed for the pretreatment of iron and steel sheets for galvanising. A. R. POWELL.

Manufacture of finely-divided metal powders obtained from carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,039, 28.9.28).—The flocculent metal powder obtained by thermal decomposition of, e.g., iron carbonyl at 320° is ground for a short time in a ball-mill filled with nitrogen to obtain a fine, loose powder. A. R. POWELL.

Removal from metal powders of the impurities which form volatile compounds with oxygen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,082, 17.11.28).—Finely-divided iron containing carbon and produced by the thermal decomposition of iron carbonyl is moistened with water and dried at 100° to effect superficial oxidation of the particles. The mass is then heated at 800° in hydrogen, whereby all the carbon is removed as carbon monoxide. A. R. POWELL.

Regeneration of spent metals which have been used for the production of metal carbonyls. CHEM. FABR. VON HEYDEN A.-G., Asses. of K. BUCHHEIM (G.P. 454,861, 5.12.25).—Surface contaminants are removed by treating the metals successively with oxidising gases and with hydrogen. L. A. COLES.

Recovery of copper and other metals from copper-bearing ores. F. L. DUFFIELD (B.P. 321,685, 15.8.28).—Copper sulphide concentrates mixed with coal and limestone are introduced into the bottom of a heated, vertical retort, and the mixture is caused to move upwards through progressively hotter zones, discharged at the top of the retort, and treated for the separation of iron and copper by mechanical means. The gases produced in the retort are freed from dust and burnt to provide the necessary heat. A. R. POWELL.

Refining nickel-copper matte. F. E. LATHE (B.P. 303,066, 24.12.28. Can., 27.12.27).—In the treatment of nickel-copper matte in the Bessemer converter, the blow is stopped when 2—10% Fe is still retained, the matte is cooled rapidly, e.g., by granulation in water, and the product is ground and boiled with sulphuric acid, which dissolves 98% of the nickel and leaves a residue of copper sulphide and any precious metals. A. R. POWELL.

Copper coating [of metal objects]. J. G. DELY, Assr. to CHEM. RES. & DESIGNING CORP. (U.S.P. 1,735,000, 12.11.29. Appl., 19.4.28).—A silicate vehicle, containing copper particles and a sodium halide, is applied to the object and then baked. F. G. CLARKE.

Coating metallic or non-metallic bodies with (A) vanadium, (B) chromium. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 321,391 and 321,914, [A] 27.7.28, [B] 9.7.28).—The body is heated to incandescence in a quartz container filled with the vapour of iodine and (A) vanadium iodide or (B) chromium iodide and containing a heated mass of vanadium or chromium to form fresh quantities of iodide. In each case the deposited metal is ductile.

A. R. POWELL.

Coating of metals [iron, steel, and copper] with metals [lead alloys]. E. C. R. MARKS. From AMER. MACHINE & FOUNDRY CO. (B.P. 321,407, 29.6.28).—The metal is first coated at 470° with an alloy of lead with 4.5% Zn and 0.5% Al, then at 400° with an alloy of lead with 2% of phosphor-tin and 0.1% of lead phosphide. A. R. POWELL.

Jointing of aluminium by solder. H. JACKSON (B.P. 321,554, 10.11.28).—The parts to be joined and the solder are cleaned and coated with petroleum jelly which volatilises at the temperature at which the solder melts. A suitable solder, m.p. 230—240°, contains Sn 50%, Zn 12.5%, Al 12.5%, Pb 25%.

C. A. KING.

Beneficiation of manganese ores. L. B. MILLER and W. G. RINEHART, Assrs. to L. B. MILLER (U.S.P. 1,736,615, 19.11.29. Appl., 22.4.27).—The ore is washed and graded, clay and dirt being removed by washing. The fines are agitated in water in a series of steps and the larger ore is crushed to uniform size; foreign materials are removed mechanically. C. A. KING.

Hard solder particularly for grey pig iron, cast steel, iron, and the like. E. LAY and C. MATTICK, Assrs. to AMER. LURGI CORP. (U.S.P. 1,736,654, 19.11.29. Appl., 15.2.28. Ger., 18.2.27).—See B.P. 285,485; B., 1928, 931.

Recovery of tin and its associated metals from alloys, mechanical mixtures, and ores. F. BISCHITZKY (U.S.P. 1,737,140, 26.11.29. Appl., 25.5.26. Ger., 5.7.24).—See B.P. 288,049; B., 1928, 412.

Galvanising bath. N. K. TURNBULL (U.S.P. 1,736,948, 26.11.29. Appl., 13.6.28. U.K., 14.6.27).—See B.P. 291,961; B., 1928, 609.

Electrochemical process for extraction of copper and zinc from ores. H. S. MACKAY (U.S.P. 1,737,425, 26.11.29. Appl., 28.1.26. U.K., 24.9.25).—See B.P. 262,546; B., 1927, 115.

XI.—ELECTROTECHNICS.

Present position of the industrial electrolysis of alkali chlorides. P. BUNET (Chim. et Ind., 1929, 22, 659—675).—Cells for the electrolysis of solutions of sodium or potassium chloride may be divided into two main classes: (A) diaphragm cells, in which the diaphragm may be immersed (Outhenin-Chalandre, Griesheim) or work with the cathode chamber practically empty (Nelson, Gibbs, Allen-Moore, and Krebs). In these cells the diaphragm forms a filter of increasing thickness downward. The cathodic liquor in this type may contain 120 g./litre of soda, and the current efficiency may be 90—95%. In some designs the anodes

are in pairs and the cell so shaped that both sides of the anodes are at least partly effective. With the Krebs cell the diaphragms should last 6—9 months and the anodes up to 2 years. (b) This type of cell works with a mercury cathode and requires a higher minimum *E.M.F.* Their internal resistance is less, but owing to the high first cost of the cell it is essential to work with a high current density. In spite of the advantage of the direct production of caustic soda of high concentration, the cost of mercury has limited the development of this type. A large French plant having 72 cell-pairs and working with a mercury depth of 6 mm. (which corresponds to 1½ kg. per cell-pair) loses 40 kg. of mercury per year. Whilst the electrolytic manufacture of hypochlorites has practically ceased, that of chlorates is now on a scale comparable with that of chlorine. Cells of simple design and made by the user are employed; sodium dichromate is added to improve the yield, which with the addition may be 80%. The hydrogen produced in these cells contains 8% or more of oxygen. Sodium chlorate is prepared in solution by batch-working, freed from dissolved chlorine, hypochlorites, and sulphates by addition of barium sulphide, and the solution evaporated or, better, refrigerated. Details of yields are given. Potassium chlorate is best made by double decomposition, as electrolysis at 80° (necessary owing to the insolubility of the salt) involves heavy wear on the anodes. The production of sodium chlorate is not adversely affected by great variations in the current supplied, and it is thus suitable as an outlet for power at off-peak periods. C. IRWIN.

Treatment of gases. PETERS.—See II. **Hypo-sulphites.** MCGLYNN and BROWN. **Phosphoric acid.** KLUGH.—See VII. **Chromium plating.** BIRETT. **Tin.** PROCTOR.—See X.

PATENTS.

Electrical heating body [resistor] and its manufacture. H. LOHMANN (U.S.P. 1,736,745, 19.11.29. Appl., 15.3.28. Ger., 19.3.27).—A plastic mixture of carbon and metal oxides is moulded under pressure and burned so as superficially to coat the product with carbonate; the whole is then encased in metal.

F. G. CLARKE.

Manufacture of incandescence bodies. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 309,573, 12.3.29. Holl., 13.4.28).—A mixture of a non-crystallised metal of high m.p., preferably tungsten, with a light- or electron-emitting metallic compound is heated at a temperature below that of recrystallisation of the metal.

J. S. G. THOMAS.

Electric incandescence lamp. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 310,523, 15.4.29. Holl., 27.4.28).—A resistance consisting of powdered cadmium oxide is introduced into a series lamp in parallel with the filament.

J. S. G. THOMAS.

Metal-vapour lamp. G. P. BEAUDRY (U.S.P. 1,736,642, 19.11.29. Appl., 25.3.2).—The casing of the lamp consists of a receptacle made of transparent material containing equal amounts of sodium aluminate and sodium and calcium borate. H. ROYAL-DAWSON.

Formation of emulsions and the like. A. L. LOOMIS and R. W. WOOD (U.S.P. 1,734,975, 12.11.29.

Appl., 2.9.27).—Emulsions or colloidal suspensions are formed from two adjacent, immiscible substances, one at least of which is liquid, by subjecting them to a radio-frequency electric current of high voltage. The two substances, in a container, may be immersed in a bath of oil in a vessel made of insulating material, the electrodes being formed by two conductors separated by a crystal. F. G. CLARKE.

Manufacture of articles from molten carbon. S. MÜNCH, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,736,832, 26.11.29. Appl., 24.2.26. Ger., 30.6.25).—See B.P. 254,679; B., 1927, 370.

Electrolytic cell. R. EDGEWORTH-JOHNSTONE (U.S.P. 1,738,372, 3.12.29. Appl., 10.1.29. U.K., 4.11.27).—See B.P. 307,093; B., 1929, 362.

Galvanic batteries and the like. G. REDMAYNE (B.P. 322,048, 13.10.28).

Compact masses [for accumulators] (B.P. 321,394). **Oxides of nitrogen from air** (G.P. 454,694).—See VII.

XII.—FATS; OILS; WAXES.

Fluorescence of cacao fat. J. D. VAN ROON (Chem. Weekblad, 1929, 26, 576—577. Cf. van Raalte, B., 1928, 792; Carrière, B., 1929, 26).—It is concluded that fluorescence is a property of one or more of the glycerides present in the pure fat. Pressed unrefined fat shows no fluorescence by transmitted light, and little by reflected light, unless the fat has been exposed to sunlight for some time. The test is regarded as of little value.

S. I. LEVY.

Cadmium oleate. A new impregnating [water-proofing] medium. H. J. BRAUN (Chem.-Ztg., 1929, 53, 913—914).—Cadmium oleate, prepared by saponifying olein with moist, precipitated cadmium hydroxide at 60°, separates as a yellow waxy product retaining 30% of water; by drying with stirring on a sand-bath at 120—130° it is obtained as an anhydrous, pale brown solid which is soluble in most organic liquids (benzine, xylene, toluene, amyl acetate, etc. with increasing viscosity to form almost colourless gels at suitable concentrations), but is insoluble in acetone and ethyl acetate, and only sparingly soluble in ethyl or butyl alcohol or trichloroethylene. A milky, stable, colloidal dispersion is obtained by saponifying in the presence of ammonia (cadmium oleate 70, 25% ammonia 400, water 780 pts.), which is very suitable for waterproofing stone, fabrics, etc. The anhydrous salt is compatible with ethyl- and benzyl-cellulose lacquers, but these are not improved by the addition. Cadmium palmitate and stearate are less soluble, hard, whitish solids. E. LEWKOWITZ.

Head and blubber oils of the sperm whale. II. **Component wax esters and general structure of the oils.** III. **Determination of the higher fatty alcohols present.** T. P. HILDITCH and J. A. LOVERN (J.S.C.I., 1929, 48, 359—368 r).—II. The head and blubber oils of the sperm whale have been submitted to oxidation by potassium permanganate in acetone solution in order to investigate their general structure: the fully-saturated esters left unattacked by the oxidant, and also the fully-saturated alcohols and acids isolated from the acidic products of oxidation,

have been examined in detail by quantitative fractional distillation in the case of sperm head oil. The head oil was found to contain 29% of fully-saturated components, whilst the blubber oil contained 1–2%. The semi-quantitative examination of the saturated fragments isolated from the head oil revealed that in 100 pts. of the original oil there were present about 2 pts. of fully-saturated glycerides, together with about 27 pts. of wax esters, built up entirely from saturated alcohols and acids: there was a marked tendency for the fatty acids of lower molecular weight (capric, lauric) to associate predominantly with the three saturated alcohols (tetradecyl, octadecyl, and cetyl, the latter in much greater proportion than the two former). Further, about 24 pts. of the oil were composed of esters of saturated acids with unsaturated alcohols, and about 18 pts. of esters of unsaturated acids with saturated alcohols; the remainder of the oil (about 29 pts.) consisted of esters of unsaturated acids and alcohols and of mixed saturated-unsaturated glycerides. The oil is thus of the heterogeneous character frequently observed in animal fats, with no evidence of any one component being preferentially formed. In particular, there can be no marked proportion of cetyl palmitate present, since although cetyl alcohol is the major alcoholic component of the wax, palmitic acid is combined with it to a far less degree than lauric, myristic, and capric acids; the chief saturated wax esters present are undoubtedly cetyl laurate and myristate. Although the blubber oil was not amenable to similar detailed treatment, the data obtained indicate that its general structure is not dissimilar from that of the head oil, and that it is a heterogeneous mixture of various wax esters (mainly liquid, owing to the more unsaturated character of both the alcohols and the acids present) and mixed triglycerides. Since oleyl alcohol is the chief alcoholic component and oleic and palmitoleic acids comprise the greater part of the fatty acids present, it is clear that both oleyl oleate and palmitoleate are present in abundance, whilst appreciable amounts of cetyl oleate, palmitoleate, and perhaps palmitate may also be expected.

III. An attempt has been made to arrive at the quantitative composition of the higher fatty alcohols present in these oils by methods analogous to those employed for the determination of fatty acid compositions. The process is more complicated and less certain than in the latter case, but by fractionation of the crude alcohols under reduced pressure together with removal of the unsaturated portions from acetylated fractions by disruptive oxidation figures were obtained which are probably accurate to within a few units per cent. The values obtained were: sperm head oil alcohols, cetyl (ca. 45%), oleyl (27–30%), tetradecyl (ca. 8%), hexadecenyl (ca. 4%), octadecyl (ca. 6%), and unsaturated C₂₀ alcohols (ca. (?) 10%); sperm blubber oil alcohols, oleyl (66–70%), cetyl (25–27%), octadecyl (1%), and unsaturated C₂₀ alcohols (ca. 8%). There is a general similarity between the higher alcohols and the fatty acids present in each oil; the head oil alcohols are of lower average mol wt. and also more saturated in character than the blubber oil alcohols. The former oil contains some tetradecyl and a little hexadecenyl alcohol,

whilst the blubber oil alcohols are almost entirely made up of oleyl and cetyl alcohols. The biological origin of the higher fatty alcohols in these oils remains obscure.

Detection of linseed oil in soya-bean oil. J. F. CARRIÈRE (Chem. Weekblad, 1929, 26, 575).—Precautions necessary in the determination of the hexabromide value, from which the proportion of linseed oil is calculated, are detailed. S. I. LEVY.

Decompositions occurring on heating oils and fatty acids with active nickel on carriers. H. I. WATERMAN and M. J. VAN TUSSENBROEK (Chem. Weekblad, 1929, 26, 566–567; cf. B., 1929, 861).—Variations in the contents of linoleic and stearic acids, and of their ethyl esters, when these are heated alone and with nickel, lead to the conclusion that the changes in the constants of soya-bean oil when heated with nickel arise chiefly from changes in the glycerides of linoleic acid. S. I. LEVY.

The mobility-concentration intercept [of fatty and lubricating oils]. J. T. BALDWIN (Ind. Eng. Chem., 1929, 21, 1121–1124).—The mobility-concentration curves of castor oil, tung oil, linseed oil, and lubricating oil with silica and “drop black” were studied. It is shown that the mobility-concentration intercept (point of zero mobility) is independent of the wetting power of the liquid used, but is dependent on grinding conditions. Yield point and oil absorption, on the other hand, are measures of degree of dispersion, dependent on wetting power or adhesion tension of the liquid. The mobility-concentration relationship is linear except in regions of pigment concentration approximating to the “oil absorption,” where the cohesion forces residual in the pigment probably become more active. S. S. WOOLF.

Action of fuller's earth on oils. HASEMAN.—See II. **Highly active fuller's earth.** SCHOLZ.—See VII. **Soaps in paints.** BALDWIN.—See XIII. **Beet fat.** PAVLAS.—See XVII. **Fluorescence of cacao butter.** RIPERT. **Determination of sulphurous acid in foods.** ROTHENFUSSER.—See XIX.

PATENTS.

Manufacture of lubricating grease. W. V. ATKINSON, ASSR. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,736,302, 19.11.29. Appl., 14.7.23).—The soap stock is saponified under pressure at the corresponding temperature; the pressure is then rapidly reduced to cause partial evaporation of the water from the mass, and the requisite mineral oils are added during agitation. E. LEWKOWITSCH.

Production of stable foams and emulsions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 321,650, 9.8.28).—Fish glands, especially soft roe (milt), or dry preparations therefrom (e.g., those obtained by degreasing and drying the raw material after alkaline extraction) are added (e.g., 0.5%) to soaps or saponaceous materials (saponins, sulphonic acids, sulphite-cellulose waste liquors, etc.) capable of foaming, in order to stabilise the foam; such foams are stable in hard water or acid liquors. E. LEWKOWITSCH.

Ozonation of [animal and vegetable] oils. J. McKEE (B.P. 321,633, 10.5.28).—Edible and medicinal

oils of which the sp. gr. may be increased to 0.970—0.980 by heating (*e.g.*, castor, sardine, rape, linseed oils) are heated at reduced pressure in order to deodorise them and to raise their sp. gr. and are ozonated at about 65.5° by blowing with ozone, yielding products containing 2—15% O₃. Vegetable oils (*d* 0.913—0.932) may, prior to ozonation, be blown or heated at the flash point to about *d* 0.975. Suitable apparatus is described. E. LEWKOWITSCH.

Hardening or solidification of organic isocolloids containing unsaturated high-molecular acids. L. AUER (B.P. 321,690, 8.5.28).—Such isocolloid materials, *e.g.*, fatty oils, resins, are hardened by heating, preferably at reduced pressure and at temperatures between 250° and 300° with 2—10% of metallic peroxides (except those of lead and manganese) until dissolution of the latter occurs. The reaction mixture may then be treated with gases, *e.g.*, alternately with sulphur dioxide and hydrogen sulphide, or with organic metal-free compounds, *e.g.*, formaldehyde, hexamethylene, glycerin, phenol. The products are suitable for varnishes, hard resins, rubber substitutes, etc. E. LEWKOWITSCH.

Apparatus for pressing out oil-containing seeds or fruit. DUCHSCHER & CIE., SOC. EN COMMANDITE (B.P. 303,498, 27.10.28. Ger., 6.1.28).

Emulsions for boiler-scale removal (B.P. 321,337). **Fluid for refrigeration** (U.S.P. 1,735,170).—See I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Preliminary report on the investigation of soaps [in paints]. J. T. BALDWIN (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 858—881).—A number of typical paint soaps (resinates, linoleates, etc. of lead, manganese, zinc, cobalt) were dissolved separately in benzene, triolein, and oleic acid, representing the three classes of material present in paint vehicles. Lowering of f.p. was taken as the criterion of dissolution, and it was found that metallic soaps are in no case dissolved in paint vehicles, but are colloiddally dispersed. Standard white pigments were ground in 18 dispersions of soaps in oil and the properties of paints prepared from these pastes were observed. Full details are tabulated of changes in consistency, degree of settling, ease of reincorporation, etc. These results are discussed in general terms. S. S. WOOLF.

Settling of pigments in house paints. II. H. L. BEAKES (Ind. Eng. Chem., 1929, 21, 1118—1121; cf. B., 1928, 647).—Pigments differing in sp. gr. were not found to separate from one another to any great extent in the normal settling of mixed paints. On applying an accelerated test (cf. Werthan and Wien, B., 1928, 647), the pigment ratio is not necessarily maintained. The effect of charging pigments by grinding on a charged mill was studied. With a positive charge, the settling of red lead was hastened whereas that of silica was retarded. Indications are obtained that single-pigment suspensions of various concentrations settle to a definite concentration characteristic of the components; thus silica (40—65%) in linseed oil (60—35%) produces on settling for 17 days a paste containing 71% of silica, and a similar figure is obtained on centrifuging a sus-

pension, although the sediment in the latter case is more closely packed. On long-continued settling this figure increases, but the "critical" concentration is maintained in samples drawn from the middle of the paint for periods exceeding 100 days. It is suggested that an ideal figure for the oil necessary to grind a pigment may thus be obtained. Further points treated are the stratification of paints on settling, changes in consistency on mixing paints, effects of sp. gr. of pigment and composition of vehicle on settling rate etc. S. S. WOOLF.

Suspension agents in flat wall paints. J. L. CROWLEY, F. W. CORKERY, and I. R. MESSER (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 882—886).—A number of flat wall paints were made up on a standard formula with the addition of small amounts of various suspension agents, *e.g.*, aluminium stearate, gum tragacanth, East India gum, water, linseed oil soap, sulphonated castor oil, etc. In a preliminary examination after 2 months' storage at 21°, the paints are rated in 5 groups. No definite conclusions can yet be arrived at, but the paints containing sulphonated castor oil appear to tend towards hard settling. A series of accelerated settling tests is being run in conjunction with the normal settling tests.

S. S. WOOLF.

Causes of floating of pigments in paints and enamels. EVERMAN (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 887—893).—With a view of studying "floating," a number of enamels containing a proportion of black pigment were prepared, the nature of the vehicle and the grinding process being varied. It is shown that free fatty acids aggravate floating, the sp. gr. of a pigment is not a major cause in its floating, fine or protracted grinding reduces the tendency to floating, and rosin is an aid in reducing floating by virtue of its superior wetting action rather than of its acidity. The conclusion is drawn that floating is mainly due to the lack of pigment wetting or dispersion.

S. S. WOOLF.

Estimation of brushing and flowing properties of paints from plasticity data. R. V. WILLIAMSON, G. D. PATTERSON, and J. K. HUNT (Ind. Eng. Chem., 1929, 21, 1111—1115).—The viscosity of some paints varies greatly with rate of flow, whereas that of others is practically independent thereof. Thus paints of similar brushing properties differ widely in viscosity. Since the resistance to shear of a plastic substance varies with the rate of shear, the flowing properties of a plastic under any given practical conditions depend on its resistance to shear at the particular rate of shear characteristic of these conditions. Further, paints at brushing consistency do not flow in accordance with the law of ideal plastic flow. It is shown that the equation for pseudoplastic flow (cf. B., 1930, 39) can be applied to the estimation of the relative brushing and flowing properties of paints. Results obtained in this manner are correlated with practical tests by a painter, good agreement being found in general order of classification, whilst the plasticity data indicate small differences that the practical tests fail to establish. S. S. WOOLF.

Evaluation of plastic or texture paint. F. C. ATWOOD (Amer. Paint & Varnish Manufs.' Assoc.,

Nov., 1929, Circ. 356, 833—842).—Plastic paints are dry powder compositions of suitable fillers and water-soluble binding agents that are made ready for use by mixing with water, and undergo irreversible change when applied. Their essentials are texturing or stippling properties enabling various decorative finishes to be obtained, ease of application without loss of plasticity, bonding and hardening qualities, and durability. Among other desiderata are ease of mixing (non-lumping) freedom from objectionable odour, rapidity of drying while permitting reasonable "working." All these points are discussed in detail.

S. S. WOOLF.

Material testing in the service of paint technology. A. V. BLOM (Farben-Ztg., 1929, 35, 493—496).—A series of brief notes describing the study of the drying of films in various stages. The settling of pigments is shown to vary with the dielectric constant of the vehicle; thus the order of relative settling of two samples of Victoria-green in ethyl alcohol was reversed in carbon tetrachloride. Retention of solvent in a film is bound up with the determination of volatile content of a varnish. The normal loss of weight method is unreliable owing to variations in film thickness, and a satisfactory method is to brush the varnish (weighed by diff.) on a plate and observe the increase of weight on drying, thoroughly reliable figures being obtained after 1 hr. at 110°. Correlation of tackiness (measured by a new apparatus recording the force necessary to pull a leather pad out of the surface) with time offers a convenient method of studying drying under various conditions of humidity, temperature, light, and film thickness. Determination of resistance to scratching and of breaking tension are also discussed as affording methods of following the hardening of a film after it is set, and typical problems clarified by these means are instanced.

S. S. WOOLF.

Titanium white. F. VON BICHOWSKY (Ind. Eng. Chem., 1929, 21, 1061—1063).—A new method of preparing chemically pure titanium oxide is described. Crude titanium cyanonitride suspended in water is treated with nitric acid formed *in situ* from sodium nitrate and sulphuric acid, sodium phosphate being added as negative catalyst to inhibit violence of reaction. After 24 hrs. more water is added and the titanium white is allowed to settle and washed, 47% of the combined nitride-nitrogen being recovered from the wash waters as ammonium sulphate. The dry titanium oxide obtained has d 2.72, which is extremely low for this material and renders it very suitable for use as a paint pigment.

S. S. WOOLF.

Exposure tests of white pigments in various vehicles. F. LECHLIDER, H. MILLER, and J. F. HARTLEY (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 894—896).—White lead, zinc oxide, lithopone, and Titanox were each ground separately in the following liquids: raw linseed oil, kettle-bodied linseed oil, "20-gallon" varnish, and "35-gallon" varnish. Two paints were made from each base, one containing 10% more pigment than the other, and all were exposed at an angle of 45° facing south. After 6 months the lead panels were somewhat darkened and showed considerable chalking, the lithopone paints were yellowed

considerably, those containing varnish had checked, whilst the Titanox series were all chalking. The paints containing the higher percentage of pigment were better than the others throughout. Further paints were prepared with mixtures of the above pigments. In both series of tests the zinc paints were by far the best.

S. S. WOOLF.

Effect of ageing on the drying of printing inks. L. M. LARSEN and A. GRUNDER (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 897—900).—The drying of printing inks that had been stored for 2 months was compared with that of freshly prepared inks of the same composition. The pigments used are classified as (a) those not affecting the drying; (b) those leading to retardation. It is shown that printing-ink pigments containing aluminium hydroxide are retarders when common driers are used, probably owing to precipitation of one or more metals in the driers. This is overcome by the use of a proprietary drier, believed to consist of metallic naphthenates.

S. S. WOOLF.

Effect of plasticisers in clear and pigmented [tung oil] varnishes. ANON. (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 900—904).—Various quantities of butyl phthalate, tolyl phosphate, phenyl phosphate, and ethyl abietate were added to a series of tung oil varnishes containing ester gum, Amberol, limed rosin, and Cumar, respectively, as resin component. The varnishes were pigmented with zinc oxide, lithopone, and Titanox, and these enamels and the varnishes were exposed on wood and iron. The results of these tests are fully tabulated and discussed. It is considered that plasticisers in either short- or long-oil varnishes or enamels made therefrom are of little or no practical value, although there is some evidence that plasticisers retard chalking. Any increase in durability is, however, at the expense of drying and hardening, and the ideal plasticiser in this connexion is one which will decompose, forming anti-oxidants after the film has dried.

S. S. WOOLF.

Use of synthetic resins in lacquer and varnish. A. E. STAUDERMAN (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356, 815—822).—Cellulose lacquers were prepared containing various proprietary synthetic resins ("glyptals" and "rezyls"), and submitted to exposure tests etc. The suitability of these products for various classes of work is indicated. It is considered that glyptal undercoats for cellulose lacquers are inferior to oil undercoats in durability. Oil varnishes containing ester gum, Congo ester, and Amberols were also prepared and exposed. Amberol varnishes were much superior to the others, whilst Congo ester is rated higher than ester gum. From the behaviour of differently-cooked varnishes of similar composition it is concluded that the correct way of making this type of oil-varnish is to cook resin and oil together rather than to cook the oil alone and check it with the resin.

S. S. WOOLF.

Effect of method of application and type of wood on durability of spar varnish. R. W. FRIESE, J. H. MEYER, A. ZINZER, and A. MURPHY (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1929, Circ. 356,

810—814).—Factors influencing the durability of exterior varnishes on wood are discussed. A series of accelerated exposure tests was undertaken to investigate the variable factors. Sitka spruce, Alaskan spruce, cypress, yellow pine, and oak were coated with an ester gum-tung oil varnish over primers consisting, respectively, of boiled oil, thinned varnish, normal varnish, and wood filler. The anticipated superiority of boiled oil priming was not evinced. Thinned varnish was found to be a suitable primer for wood of resinous nature, *e.g.*, yellow pine and cypress, whilst wood filler did not increase the life of the film except on oak. In "stepped" exposures of 3 coats of varnish without any priming, 1 and 2 coats failed quickly, but 3 coats gave excellent results.

S. S. WOOLF.

Adhesion tension of carbon black. BARTELL and SMITH.—See II. **Fastness of lake colours.** KINSMAN.—See IV.

PATENTS.

Manufacture of printers' ink. L. C. NEALE (B.P. 321,558, 12.11.28).—Vulcanised rubber is heated with residual crude hydrocarbon oil to 127°, the liquid is strained if necessary, up to 25% of carbon black or other suitable pigment and up to 10% of a commercial varnish, *e.g.*, a rosin-tung oil varnish, are added consecutively with agitation, and the whole mass is ground. Waste rubber may be used, *e.g.*, old rubber tyres, and if these contain fabric or other similar material they are cut up into small pieces, immersed in concentrated sulphuric acid to destroy the fabric, and neutralised by immersion in caustic soda solution. This treatment is unnecessary if granulated rubber be used.

S. S. WOOLF.

Varnish. H. E. BUC, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,735,493, 12.11.29. Appl., 15.9.27).—The film-forming constituents consist of 50—90% of drying oil, 5—25% of resins, and 5—25% of the calcium salt of an oil-soluble sulphonic acid derived from sulphonated petroleum oil.

H. ROYAL-DAWSON.

Production of artificial [water-soluble] resins. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,915, 27.4.28).—Methylol-uræas or -thioureas, or their esters or polymerides, are heated with at least 1 mol. of a polyhydric alcohol (other than a carbohydrate) or of an ether of the same containing at least two free hydroxyl groups, in the presence of a buffer substance maintaining the solution at pH 4—7, *e.g.*, acid potassium oxalate or tartaric acid (cf. B.P. 261,029, 266,752, 266,361, 301,696; B., 1928, 532, 761, 826; 1929, 138).

L. A. COLES.

Condensation products of polyhydric phenols and formaldehyde. SOC. DES USINES CHIM. RHÔNE-POULENC, N. B. GRILLET, and E. C. COTTER (B.P. 321,697, 17.5. and 16.6.28).—Polyhydric phenols (particularly resorcinol) are condensed with formaldehyde below 80°, preferably in the absence of catalysts, and, if desired, in the presence of retarding agents, *e.g.*, alcohol, glycerin, sugars. Water is eliminated from the products by distillation *in vacuo* below 50°, or by pouring the reaction mixture into cold water or salt solution, while it is still viscous, but when the condensation has reached a stage sufficiently advanced for the precipitation of a

suitable amount of resin, the precipitate being removed, washed, and air-dried at a low temperature. The products may be used for the manufacture of varnishes or, with or without the addition of fillers, pigments, etc. before or after the condensation, of moulded articles.

L. A. COLES.

Hardening of condensation products from phenols and aldehydes. F. SCHMIDT (B.P. 304,648, 28.11.28. Ger., 23.1.28).—Phenol-aldehyde condensation products containing little or no filling materials are hardened very rapidly by the addition of as small quantities as possible of acid or acid-acting substances, *e.g.*, ethylene chlorhydrin, magnesium chloride, in kneading or other suitable apparatus where thorough and continually renewed contact of particles is assured. The mass is heated, *e.g.*, for 5—6 hrs. at 40—50°, until it becomes almost non-adhesive, and is then subjected to further treatment, *e.g.*, rolling, hot or cold pressing, and subsequent heating.

S. S. WOOLF.

Moulding of synthetic resins. F. H. BENGE and R. R. TITUS, Assrs. to CONTINENTAL-DIAMOND FIBRE Co. (U.S.P. 1,735,668, 12.11.29. Appl., 2.8.28).—The initial condensation product of a synthetic resin is heated at a temperature and for a time which will cause the resin to be converted into the final stage after the pressing operation, the hot plastic mass is placed in a cold press, and, while the mass is still hot, sufficient pressure is exerted to obtain the moulded form desired.

S. S. WOOLF.

Manufacture of ester gum. B. F. WILLIAMSON and W. H. BEISLER (U.S.P. 1,734,987, 12.11.29. Appl., 23.5.27. Renewed 13.9.29).—A mixture of glycerin and gum theus is heated to drive off undesirable volatile substances and to cause the glycerin to combine with the resin content of the gum.

F. G. CLARKE.

Production of higher aldehyde derivatives of reaction products of aldehydes and amines. C. O. NORTH, Assr. to RUBBER SERVICE LABS. Co. (Re-issue 17,511, 3.12.29, of U.S.P. 1,619,953, 8.3.27).—See B., 1927, 392.

Azo-dye lakes (B.P. 293,352).—See IV. **Cellulose compositions** (B.P. 319,073).—See V. **Chromic compounds** (B.P. 294,965).—See VII. **Isocolloids** (B.P. 321,690).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Influence of pigments on some physical properties of unvulcanised rubber. H. A. DEPEW (Ind. Eng. Chem., 1929, 21, 1027—1030).—Unvulcanised rubber containing fine powders disperses very slowly in solvents, but particle size is not the only influencing factor. It is suggested that the reduction in solubility is caused by the powder becoming flocculated and forming a structure which sets as a semi-permeable membrane, thus retaining the rubber aggregates. If the powder be zinc oxide the addition of stearic acid breaks down the flocculated structure and increases the solubility of the rubber. Mathematical calculation as to the probable order of magnitude of the openings in a structure of flocculated zinc oxide and of the size of the

rubber micelle accords satisfactorily with the above theory. As is well known, an increase in the percentage of powder, which would reduce the size of the openings in the semi-permeable membrane, further reduces the solubility of the rubber, whilst mastication of the rubber, which reduces the size of the rubber aggregates, increases the solubility. Magnesium carbonate gives a dry, hard mixture with rubber, thereby indicating its flocculated character; in accordance with this the rubber containing it is decidedly resistant to dissolution in benzene.

D. F. TWISS.

Relation between artificial ageing tests and natural ageing [of rubber]. J. M. BIERER and C. C. DAVIS (Ind. Eng. Chem., 1929, 21, 1008—1009).—Most rubber goods deteriorate from a combination of influences rather than from one; hence artificial tests are unlikely to duplicate exactly the results of natural ageing. On exposure at 60° to oxygen under a pressure of 300 lb. the behaviour of under-vulcanised samples or those containing a high proportion of sulphur may deviate seriously from behaviour in natural ageing, and a lower temperature and pressure are better for the "bomb" test, selection of the actual conditions being a matter of judgment. From a practical point of view it is not possible to designate a certain number of hours in the oxygen bomb as the equivalent of so many years of natural ageing.

D. F. TWISS.

Accelerated ageing v. shelf ageing [of rubber]. E. M. FOLLANSBEE (Ind. Eng. Chem., 1929, 21, 1012).—Average results on a large number of samples of rubber-insulated wire kept in air at ordinary temperature and 70°, respectively, and in oxygen at 300 lb. at 60° show that a period of 24 hrs. under the last conditions is roughly equivalent to one year and 96 hrs., respectively, under the former conditions. The oxygen test represents a much shorter period of natural ageing for lower-grade compounds. In both types of artificial test evidence of deterioration, relative to the results of natural conditions, occurs more rapidly in the values of tensile strength than in those of elongation.

D. F. TWISS.

Natural v. artificial ageing [of rubber]. S. KRALL (Ind. Eng. Chem., 1929, 21, 1009—1012).—Experiments with a "pure rubber" mixing and two motor-tread rubbers, with and without reclaimed rubber, respectively, show that there is no general correlation between artificial ageing either at 70° in air or at 70° in oxygen at 300 lb., and natural ageing. It is impossible to quote generally equivalent periods for natural and artificial ageing, and it is important to select that method of artificial ageing which will most closely simulate the conditions of service to which the rubber in question is to be subjected.

D. F. TWISS.

Ageing of mechanical rubber goods stocks by various methods. K. J. SOULE (Ind. Eng. Chem., 1929, 21, 1013—1015).—From a comparison of 30 mechanical rubber stocks kept for one year in the dark, for 24 hrs. and 48 hrs. at 60° in oxygen at 300 lb., and for 96 hrs. at 70°, respectively, it is found that none of these artificial conditions shows satisfactory correlation with natural ageing for all types of "compound" or for all the physical properties of any one class of "com-

pound." As an approximation to natural ageing there is little choice between the "bomb" and the "oven" test.

D. F. TWISS.

Artificial ageing for rubber. W. W. VOGT (Ind. Eng. Chem., 1929, 21, 1015—1016).—In using natural, oven, and bomb ageing tests even under ideal conditions, widely different results are obtained for the equivalence factor according to the physical property selected as the basis of comparison; e.g., with one and the same "compound," the effect of 16 hrs. in oxygen at 400 lb. and 50° was comparable with either 7 or 3 months of natural ageing, using the tensile product and modulus, respectively, for comparison. Various accelerators also influence greatly the course of artificial ageing. The "bomb" test is regarded as a sensitive and trustworthy index to the oxidisability of rubber stocks. In mixtures relatively resistant to oxidation the alteration in an accelerated test is mainly a heat effect. No single test can reproduce quantitatively all the phenomena associated with natural ageing, and it is better to apply independent tests to accentuate each particular phase; e.g., resistance to oxidation may be tested by heating sheets in oxygen at 150 lb. and 50° for 6—12 days, and freedom from after-vulcanisation or stiffening can be tested by heating for 6 and 12 days in nitrogen at 70°.

D. F. TWISS.

Correlation of various ageing tests with natural shelf ageing [for rubber]. S. M. CADWELL (Ind. Eng. Chem., 1929, 21, 1017—1019).—Bomb and oven ageing tests are qualitatively trustworthy, particularly for stocks containing an antioxidant. No simple relation exists, however, between such artificial ageing tests and the behaviour of rubber under tension in the sun. The oven test is inferior to the bomb test when applied to stocks containing manganese or copper. To ensure good ageing careful compounding and vulcanisation are important, as is also the use of an antioxidant.

D. F. TWISS.

Correlation between Geer oven and natural ageing of selected [rubber] tyre compounds. A. II. NELLEN and II. M. SELLERS (Ind. Eng. Chem., 1929, 21, 1019—1020).—Test results obtained with typical rubber compounds as used for the tread and carcass of a motor tyre show that periods of 4 and 8 days, respectively, at 70° in a Geer oven are equivalent approximately to 1 and 2 years of natural ageing. The tensile product of the rubber forms the best basis of comparison.

D. F. TWISS.

PATENTS.

Removal of water from deposits formed from aqueous dispersions of rubber or similar vegetable resins. ANODE RUBBER CO., LTD., Assees. of A. SZEGVARI (B.P. 298,483, 8.10.28. U.S., 6.10.27).—Tubular articles, freshly formed from aqueous dispersions of rubber compositions, are partially dehydrated, while still on their deposition bases, by applying pervious, helical wrappings under tension so as to subject the deposits progressively from end to end to compacting pressure.

D. F. TWISS.

Continuous removal of agglomerates from rubber dispersions and the like. ANODE RUBBER CO., LTD., Assees. of A. SZEGVARI (B.P. 294,153, 18.7.28.

U.S., 18.7.27).—The homogeneity of aqueous dispersions, *e.g.*, of rubber, whether compounded or otherwise, is maintained by withdrawing the dispersion at or near the surface through a conduit by which it is reintroduced into the bottom of the chamber with a smooth, stream-line flow. Particles larger than a predetermined size are removed at some point of the circulatory system.

D. F. TWISS.

Manufacture of a latex mixture having high adhesive properties. NEDERL. GUTTA-PERCHA MAATSCHAPPIJ (B.P. 307,731, 23.7.28. Holl., 29.3.28).—*Hevea* latex or latex of similar composition is mixed with latex or latices, *e.g.*, jelutong latex, relatively poor in rubber and containing a high proportion of cholesterol-like substances.

D. F. TWISS.

Manufacture of rubber conversion products. GOODYEAR TIRE & RUBBER Co., Assees. of H. A. BRUSON (B.P. 306,390, 5.12.28. U.S., 18.2.28).—Thermoplastic products are obtained by heating masticated or dissolved rubber with an acid, such as chlorostannic acid or bromostannous acid, the negative radical of which contains tin and a halogen. The products may be subsequently worked on a hot mill for several hours.

D. F. TWISS.

Treatment of rubber latex compositions and articles formed thereby. E. HOPKINSON and M. C. TEAGUE, Assrs. to AMER. RUBBER Co. (U.S.P. 1,736,404, 19.11.29. Appl., 22.9.25).—A cold-water paint, comprising a non-oily aqueous suspension, is prepared from latex, a protective colloid, a binder, and a pigment.

F. G. CLARKE.

Manufacture of rubber tyres. I. G. FARBENIND. A.-G. (B.P. 299,037, 30.8.28. Ger., 20.10.27).—Plastic polymerisation products obtained from butadiene, preferably with the aid of sodium, are used for the production of elastic tyres, *e.g.*, by applying sheets of a mixture with sulphur and compounding ingredients to textile fabric by means of an adhesive, and then vulcanising to form a tyre.

D. F. TWISS.

Tyre mould. A. BOERDER (U.S.P. 1,735,916, 19.11.29. Appl., 30.6.28).—A mould for curing rubber has a coating of zirconium upon its inner surface.

H. ROYAL-DAWSON.

Coating of metal articles with rubber or similar material. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 321,398, 6.7.28).—Articles with corrugated or ribbed surfaces are given a substantially uniform coating of rubber or similar material from aqueous dispersion thereof by applying successive coats by electrophoretic deposition and dipping or *vice versa*.

D. F. TWISS.

Impregnation of wood (U.S.P. 1,733,483).—See IX. **Isocolloids** (B.P. 321,690).—See XII. **Printers' ink** (B.P. 321,558).—See XIII.

XV.—LEATHER; GLUE.

Oiling of leather. T. G. ROCHOW (J. Physical Chem., 1929, 33, 1528—1542).—A soft and pliable leather can be obtained by displacing water from wet cow-hide leather by alcohol and introducing cod oil as a solution in xylene. Untanned skin swollen by water and kept swollen by means of solutions which removed

it remain flexible on drying when immersed in a xylene solution of oil, whilst skin which is allowed to dry before immersion in oil remains hard and stiff. The function of water in the oiling process appears to lie in the swelling and separation of fibres, so that oil can penetrate universally. Cod oil wets leather in preference to water, but the displacement of water by the oil is slow. In general, more surface of oil should be offered to leather fibres in order to increase penetration. Usually the water already present is sufficient to reduce the surface tension of the oil, but with mineral oil this must be accomplished by the addition of suitable substances. Theories of tanning are discussed. L. S. THEOBALD.

PATENTS.

Manufacture of artificial [casein] compositions and articles made therefrom. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,423, 30.6.28).—Mixtures comprising casein, pigments, etc., and substances which generate gases at raised temperatures, *e.g.*, ammonium and sodium bicarbonate, are forced through an extrusion press at a temperature, *e.g.*, 60—70°, such that the gases are generated and render the products porous. The products may be dried or hardened, *e.g.*, by treatment with formaldehyde.

L. A. COLES.

Manufacture of a casein adhesive. E. STERN (G.P. 451,308, 6.9.24).—A mixture of dry, powdered casein and dry starch compounds of the alkaline earths or magnesium is made into a paste with cold water.

A. R. POWELL.

Manufacture of dry adhesives soluble in cold water. M. HEIM (G.P. 453,501, 25.11.24).—A mixture of 100 pts. of starch, 50 pts. or more of powdered glue, and 50—100 pts. of saturated calcium chloride solution is set aside in a warm place until a dry mass is obtained which can be ground to a powder readily soluble in a small quantity of cold water.

A. R. POWELL.

Treatment of hides. O. RÖHM (U.S.P. 1,735,977, 19.11.29. Appl., 5.2.25. Ger., 1.4.24).—See B.P. 236,483; B., 1925, 730.

Chromic compounds (B.P. 294,965).—See VII.

XVI.—AGRICULTURE.

Geology and mineralogy of soils. II. **Soils of South-East Scotland.** R. HART (J. Agric. Sci., 1929, 19, 802—813; cf. A., 1929, 289).—Mineralogical examination of the soils in this area suggests an appreciable supply of potash, although this may not be readily available, an indefinite amount of phosphate-bearing minerals, chiefly apatite, and a fair proportion of lime-bearing minerals, notably plagioclase, augite, and hornblende. One soil from Innerwick is peculiar in that the ferrosilicate minerals are mainly iron oxides, possibly due to continued cultivation with its attendant aeration, and to the use of artificial fertilisers.

E. HOLMES.

Composite character of the soil profile, its relation to soil classification. C. G. T. MORISON (J. Agric. Sci., 1929, 19, 677—683).—A discussion of the modifications in the Russian scheme of soil classification necessary to fit British conditions. Examination of

soil profiles near Oxford has shown that in addition to the *A* zone, or zone of leaching, and the *B* zone, or zone of accumulation, there can be distinguished a solubility profile, in which the total content of exchangeable bases is low, but increases in depth, especially in the *B* horizon; a skeletal profile, in which iron and aluminium are low in the *A* horizon, but high in the *B*; and an organic profile showing normal vertical distribution in *A*₀ and *A*₁, very low in the rest of *A*, showing accumulation in the *B* horizon and normal diminution due to oxidation throughout *B*. Soil types can then be indicated by the dominance of one profile; thus in saline and alkaline soils the solubility profile is dominant, in podsol types the humus profile is dominant (e.g., in the north and west of Great Britain), whilst in laterite, skeletal and solubility profiles build up the typical section. The chief agricultural lands of Great Britain are discussed on the basis of these classifications. **E. HOLMES.**

Laws of soil colloidal behaviour. II. Cataphoresis, flocculation, and dispersion. S. MATTSO (Soil Sci., 1929, 28, 373—409).—Bentonite and several soil colloids are used in an examination of the phenomena of flocculation and cataphoresis in solutions of various electrolytes. Experimental results do not confirm the theory that the particle charge and stability are associated with an absorption of ions from the added electrolyte, but tend rather to show their relationship with the degree of dissociation of the exchangeable ions. The proportion and nature of these latter control the osmotic hydration, sensitivity to electrolytes, and dispersibility of the colloid. The size of colloidal particles represents an equilibrium between cohesive and dispersive forces as governed by ionic density. For each particular colloid there is thus a particle size of maximum stability. Flocculation results from the linking together of micelles (not of particles) by electrostatic attraction. There is no critical cataphoretic potential at which soil colloids flocculate. Stability is largely controlled by hydration. The dispersion and size of colloidal particles are intimately connected with the charge they carry, but it is not established that the stability of the micelle results from mutual repulsion. The osmotic hydration of the micelle is sufficient to explain the stability of a suspension.

A. G. POLLARD.

Microflora of leached alkali soils. I. Synthetic alkali soil. J. D. GREAVES (Soil Sci., 1929, 28, 341—346).—An artificial alkali soil was prepared, subsequently leached with water, and cropped. Leaching resulted in an increase in the number of micro-organisms, notably among the ammonifying, nitrifying, and nitrogen-fixing bacteria. The morphological characteristics of the flora of the leached and cropped soil are recorded.

A. G. POLLARD.

Composition of some Turkish soil-improving "earths." F. GIESECKE (J. Landw., 1929, 77, 201—217).—The efficiency of various "earths" used in Turkish agriculture as soil improvers depends more on their contents of chalk or marl than on the presence of notable amounts of plant nutrients.

A. G. POLLARD.

Nutrient content of Turkish soils. F. GIESECKE (J. Landw., 1929, 77, 223—250).—Chemical and me-

chanical analysis of numerous soils are recorded. Generally speaking, these soils are not so highly fertile as earlier work suggests.

A. G. POLLARD.

Oxidation and reduction processes in podsol soils. N. P. REMESOV (Z. Pflanz. Düng., 1929, 15A, 34—44).—Changes in the oxidation-reduction potential (E_h) of soils under various conditions are recorded. A rapid decrease in E_h is associated with the transition from the humus sub-horizon to the podsol horizon, and this becomes still more marked in the illuvial horizon. Among soils in different stages of peat formation E_h declines as the process advances. Maximum E_h values are attained in early summer. In humus horizons seasonal variations in E_h are greater than in any other sub-horizon. Values of E_h in pasture soils are always greater than those of the same soil under any other treatment. Farmyard manure reduces the E_h of soils, not only by adding organic matter, but also by increasing the numbers of reducing bacteria such as *B. coli*. Lime increases the E_h of soils when applied alone or with farmyard manure, and such increase is due to improved soil structure and aeration leading to more intense oxidation.

A. G. POLLARD.

Quinhydrone-electrode and indicator methods for measuring the p_H of soils; their individual application and comparison. S. KÜHN (Z. Pflanz. Düng., 1929, 15A, 13—33).—The use of the quinhydrone electrode with certain soils leads to indefinite p_H measurements, and the changes in recorded p_H values with time are irregular. After removal of all colloidal matter from aqueous soil extracts by mixing with barium sulphate ("pro Röntgen") and centrifuging, the recorded p_H values show a strong tendency to drift towards the acid area. Carefully recrystallised quinhydrone in aqueous solution has p_H 3.4 approx., and imparts a definite acid reaction to a soil suspension. The barium sulphate treatment did not significantly affect the p_H values of soil extracts determined by the indicator method. Aqueous soil extracts are much more weakly buffered than the corresponding soil suspensions. The indicator method is considered far more exact and reliable than the quinhydrone-electrode method.

A. G. POLLARD.

Determination of the buffer capacity of soils. B. TACKE and T. ARND [with W. SIEMERS and W. HOFFMANN] (Z. Pflanz. Düng., 1929, 15A, 44—51).—In the application to moor soils of the Jensen method for determining lime requirements, it was found that the p_H value of the limed soil, after the specified treatment for removing excess of free lime, did not rise above 7.5 however great was the quantity of lime added; irregularities in the form of the p_H -lime curve for buffer-free sand also appeared. By passing air of normal carbon dioxide content through lime water, an equilibrium condition was produced, after several hours, having p_H 7.4—7.5. The same value was reached when equilibrium was attained by other processes. It is considered that Jensen's aspiration method for removal of excess of free lime prior to determinations of p_H values fails to ensure equilibrium between lime, water, and carbon dioxide, and results are correspondingly unreliable. By prolonged aspiration it is shown that

both in mineral and moorland soils the true lime titration- p_H curve intersects the control buffer-free curve in the alkaline area. The buffer-free sand yields a titration curve which, on the alkaline side, lies nearer the neutral zone than Jensen suggests.

A. G. POLLARD.

Determination of the lime requirement of soils on the basis of their hydrolytic acidity. J. VON CRIKY and G. VON EFERJESSY (*Z. Pflanz. Düng.*, 1929, 15A, 5—12).—Well-known methods for determining the lime requirement of soils are compared. Kappen's process gives satisfactory results when an empirical factor is used to calculate from the hydrolytic acidity the lime required to bring the soil reaction to a definite p_H value. The method is applicable to soils of neutral reaction and considerable "latent" acidity to which the Christensen-Jensen method is unsuited. In the Hungarian soils examined the lime requirement, calculated as $5 \times$ hydrolytic acidity (Kutchinsky), agrees with values obtained in the Christensen-Jensen process if the latter is calculated to p_H 8.0. The hydrolytic acidity values vary considerably with the ratio soil: solution used in the determination. Hissink's value ($T - S$) as a measure of the lime necessary to neutralise hydrolytic acidity is preferable to those obtained by other adsorption methods.

A. G. POLLARD.

Mechanical analysis of soils. C. S. PIPER and H. G. POOLE (Council Sci. Ind. Res. Australia, 1929, Pamphlet No. 13, 19 pp.).—A comparison of methods and results of mechanical soil analysis by the method developed at the Waite Institute and by the International method. A description is given of modifications necessary to make the former conform to, and of methods of interpolating results by the Waite method in terms of, International standards.

E. HOLMES.

Hot fermentation of manure in practice. WECKE (*Forts. Landw.*, 1929, 4, 68; *Bied. Zentr.*, 1929, 58, 501—504).—The general practice of the "making" of farmyard manure is discussed with special reference to the advantages of the Krantz hot-fermentation process. Apart from the mechanical advantages in stacking and the improved physical condition of the hot-fermented product in handling, field trials show an enhanced fertiliser value. Comparative crop yields with ordinary yard manure and hot-fermented manure averaged 1:1.5—2.7 in three successive years.

A. G. POLLARD.

Cold- and hot-fermented dung. GERLACH (*Z. Pflanz. Düng.*, 1929, 8B, 529—545).—In field trials with hot- and cold-fermented dung recorded by Löhnis and his co-workers, the superiority of the former product is not always convincingly shown. Hot-fermented manure is not necessarily the practically sterile mass suggested, but contains many organisms and enzymes which continue to decompose organic matter and produce carbon dioxide and ammonia. After the preliminary heating of the loose-layered stack to 60° (Krantz) cooling may be fairly rapid, and unless compression of the stack occurs before the temperature falls, subsequent losses of carbon and nitrogen are not avoided. Initial loose-stacking to promote heating in the stack is unnecessary, and does not always bring about

the effects claimed. Farmyard manure is most economically produced by immediate compacting of fresh stall manure and preventing leaching by rain. Building of stacks on loose stone drainage bottoms and the banking up of the sides with soil as the stack grows is suggested.

A. G. POLLARD.

How may nitrogen losses in the making of farmyard manure be avoided? H. VON RATHLEF (*Forts. Landw.*, 1928, 3; *Bied. Zentr.*, 1929, 58, 491—492).—Nitrogen losses from manure stacks are greatest in warm, dry, windy weather. Similar losses occur when the manure is spread but ploughing-in is delayed. As much as 50% loss of efficiency occurred in field trials when four days elapsed between spreading and ploughing.

A. G. POLLARD.

Nitrification of farmyard manure in soil. C. BARTHEL (*Z. Pflanz. Düng.*, 1929, 8B, 557—561).—The biological effects of the addition of farmyard manure to soil are due not to the addition of large numbers of micro-organisms, but to the stimulation of organisms already in the soil following added nutrients. The urinary nitrogen is the principal material concerned. Other nitrogen in the dung is practically unnitrified after 12 months. The mechanism of the transformation of urinary nitrogen into nitrate is discussed. It is suggested that the increased efficiency of hot-fermented manure (Krantz) may be due to the partial decomposition of the more stable nitrogen compounds in the heating stack leading to their more easy nitrification in the soil.

A. G. POLLARD.

Nitrogen manuring of low-moor soils. L. RINNE (*Z. Pflanz. Düng.*, 1929, 8B, 545—556).—In manurial trials for hay on low-moor soils, nitrogen fertilisers when used in addition to potash and phosphates were practically non-effective in dry seasons, particularly with a low rainfall in June. Comparative yields are recorded on horizons exhibiting various stages of peat decomposition. Generally speaking, nitrogen fertilisers were only profitable in seasons when the market price of hay was high.

A. G. POLLARD.

Rational manuring of hops with special reference to phosphates. L. R. GROB (Superphosphat, 1929, 5, 124; *Bied. Zentr.*, 1929, 58, 490—491).—Farmyard manure forms the basis of hop manuring, but unless used with complementary artificial fertilisers leaf production may be excessive and cone formation limited. Phosphatic fertilisers are most important as correctives in this respect, and tend also to improve the quality of the hops.

A. G. POLLARD.

Influence of lime in phosphoric acid mobilisation of soil. O. K. KEDROV-ZIKHMAN (*Trans. Sci. Inst. Fertilisers, Moscow*, 1929, No. 61, 107—108).—Lime releases soluble phosphates by forming organic phosphorus compounds which are readily attacked by micro-organisms. The calcium also replaces iron and aluminium in phosphates. Calcium oxide is more effective than the carbonate.

CHEMICAL ABSTRACTS.

Influence of lime and raw phosphates on podsolised soil and crop yields. D. V. DRUZHININ (*Trans. Sci. Inst. Fertilisers, Moscow*, 1927, No. 45, 5—51).—Liming of podsolised soils decreases their acidity and increases the nitrate content; the water-soluble

phosphorus decreases, whilst the plant-phosphorus increases. Raw phosphate does not change the active acidity, but affects nitrate formation and increases the yields. The active acidity of podsolised soils is that reaction of water cultures at which the phosphoric oxide from raw phosphate becomes available to plants.

CHEMICAL ABSTRACTS.

Influence of liming on the composition of oats. F. T. PERITURIN (Trans. Sci. Inst. Fertilisers, Moscow, 1927, No. 45, 55—63).—Lime (up to 0.5%) increased the nitrogen content from 170.9 to 448.1 mg., the phosphoric oxide content from 125.6 to 245 mg., the calcium oxide content from 69.2 to 161.0 mg., and the dry matter from 17.87 to 27.22 mg.

CHEMICAL ABSTRACTS.

Liming experiments [on soils]. O. NOLTE and M. RAUTERBENG (Mitt. deut. Landw.-Ges., 1929, 555; Bied. Zentr., 1929, 58, 486—487).—The importance of liming in conjunction with the increasing use of artificial fertilisers is emphasised. Field trials show that the effect of lime on crop yields can be closely correlated with the soil's content of exchange acidity and calcium carbonate, but not with the total calcium content and hydrogen-ion concentration.

A. G. POLLARD.

Ammonia in fertilisers and its relation to the life of plants. D. N. PRIANISHENKOV (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 61, 99—103).—Ammonia is taken up by the plant more rapidly, and is assimilated more easily, than are nitrates, although excessive amounts of ammonia are the more injurious. Nitrification is not desirable from the point of view of plant nutrition: nitrification regulates the concentration of ammonia.

CHEMICAL ABSTRACTS.

Field and vegetation experiments with nitrogenous fertilisers in 1927. A. N. LEBEDIANTZEV (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 61, 35—60).—Experiments on the podsol and the northern and southern chernozem soils are described. Poppy, cotton, and rice specially needed phosphorus, whilst sugar beet and tomatoes specially needed potassium. Physiologically acid nitrogen fertilisers (including urea) on acid soils with potassium and phosphorus fertilisers which also leave acid residues decreased the yields. On neutral and alkaline soils the physiologically alkaline fertilisers proved more satisfactory.

CHEMICAL ABSTRACTS.

Effect of different nitrogenous fertilisers as influenced by soil reaction. ZIELSTORFF and BUROW (Landw. Versuchs-Stat., 1929, 109, 237—252).—Fertilisers supplying 40 kg. N, 30 kg. P_2O_5 , and 80 kg. K_2O per hectare were applied to oats and barley, the nitrogen being given as ammonium sulphate, sodium nitrate, cyanamide, and urea. In the case of oats neither the physiological nature of the nitrogenous fertiliser nor the soil reaction had any influence on the utilisation of the different nitrogen compounds, but with barley on an acid soil ammoniacal nitrogen was utilised to a smaller extent than nitrate-nitrogen. This difference disappeared as neutrality was approached. Liming produced a large increase in the yield of barley on soils with a high exchange acidity, but had no effect on the yield of oats. On soils with a high hydrolytic acidity

liming produced no increase in the yield of barley. Hydrolytic acidity appears, therefore, to have very little influence on the growth of acid-sensitive plants.

E. HOLMES.

Seven-year investigation of permanent grass-land flooded by water carrying potash effluents. H. IMENDORFF and C. A. WEBER (Landw. Versuchs-Stat., 1929, 109, 127—224).—The herbage of permanent meadows and pastures in 12 different areas regularly or frequently flooded by rivers carrying effluents from potash works were examined to determine whether changes occurred in the botanical character of the flora, or in the yield and chemical composition of the hay. The results indicate no harmful effects. Allegations that observed periodic changes in the flora of permanent meadows and pastures were due to the effect of chlorides are unsupported. Chlorides are not responsible for isolated appearances of "salt plants" such as *Triglochin maritimum*, *Scirpus Tabernaemontani*, *Juncus Gerardi*, *Trifolium fragiferum*, and *Plantago maritima*, which are met with under wet conditions. Chemical examination of the herbage and of the soil showed that neither was affected by the chlorine content of the flood water, the same being true of the yield and composition of the hay.

E. HOLMES.

Experiments with Solikamsk potassium salts in 1927. D. V. DRUZHININ (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 61, 71—88).—The salt (a mixture of potassium and sodium chlorides) compared favourably with other forms of potassium salts. Experiments on sugar beet, barley, lupins, potatoes, and flax were performed.

CHEMICAL ABSTRACTS.

Physiologically acid nature of potash fertilisers. O. LOEW (Chem.-Ztg., 1929, 53, 909).—Since ripe plants contain potassium as malate, citrate, or combined with complex organic phosphoric acids even when potassium chloride or sulphate is supplied to the soil as fertiliser, the process of absorption of the potash by the plant must involve an interchange of acid radicals in which the mineral acid is set free at some stage; hence all commercial potash fertilisers must be considered physiologically as acid salts.

A. R. POWELL.

"Nitrophoska" as a fertiliser. O. NOLTE and M. RAUTERBENG (Mitt. deut. Landw.-Ges., 1929, 44, 62; Bied. Zentr., 1929, 58, 488—489).—Fertiliser trials in 1928 confirm the satisfactory nature of "Nitrophoska" as a complete fertiliser. Soils must be well limed to ensure regular and certain results from this fertiliser.

A. G. POLLARD.

Action of the complete manure "Nitrophoska" in comparison with other fertiliser combinations on the growth of sugar beet. O. ENGELS (Z. Pflanz. Düng., 1929, 88, 561—568).—In field trials with sugar beet, "Nitrophoska" produced crops of the same order as those obtained with equivalent combinations of the simpler fertilisers.

A. G. POLLARD.

Reversion of the natural alkalinity during storage of beet. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zuckerind., 1929, 79, 767—770).—Beets were examined for theoretical and practical natural alkalinity and residual lime according to the authors' methods

(B., 1928, 421). Freshly harvested roots grown under dry conditions showed a theoretical natural alkalinity varying from 0.004 to 0.026, and a practical natural alkalinity which was constant at about 0.024–0.030, the residual lime (equal to the difference between the two values) varying accordingly. Roots which previous to lifting had received plenty of rain, and were examined immediately after being harvested, showed good agreement between the two alkalinity values, the residual lime content being thus reduced almost to vanishing point. On the other hand, with roots which had been kept in a room for 5 days at 12–17° and had become soft, whereas the theoretical natural alkalinity was low, and in some cases had a negative value, the practical value was about 0.018, thus giving a high residual lime content, as has always been observed in the case of rotting roots.

J. P. OGILVIE.

Influence of thiourea on the development of apical buds and the number of shoots from potato buds. F. E. DENNY (J. Landw., 1929, 77, 219–222).—Freshly cut tubers were treated with 2% thiourea solution. More shoots per bud were thus produced and the dominance of the growth of the apical bud, when the tubers were cut lengthwise, was decreased.

A. G. POLLARD.

Composition and nitrification studies on *Crotalaria striata*. W. A. LEUCKEL, R. M. BARNETTE, and J. B. HESTER (Soil Sci., 1929, 28, 347–371).—Analyses are recorded of *Crotalaria* plants in various stages of growth, and the course of nitrate accumulation following incorporation with soil of the various parts of the plant in various growth stages has been studied. At all stages of growth the nitrogen content of the plant parts increases in the order: roots (least), stems, complete plant, leaves. The reverse order characterises the contents of cellulose and lignin. The carbohydrate content is fairly evenly distributed throughout the plant. In the complete plant there is an increase in the percentage of cellulose and lignin and decrease in the percentage of nitrogen as the plant approaches maturity. The rate of nitrification of the plant parts is greatest for leaves, followed, in descending order, by the complete plant, stems, and roots, there being a slight decrease in the case of leaves in the last stages of growth. When the complete plant is considered, nitrification declines with advancing age.

A. G. POLLARD.

Automatic watering apparatus for pot experiments. W. S. MYBURGH (Z. Pflanz. Düng., 1929, 15A, 1–5).—By means of a pivoted siphon tube water is automatically fed to culture pots to maintain these at a definite total weight.

A. G. POLLARD.

Feeding of sodium chlorate to sheep and goats. P. BRIGL and C. WINDHEUSER (Landw. Versuchs-Stat., 1929, 109, 225–235).—Quantities of sodium chlorate in the fodder such as might arise from weed-control were without effect on sheep, but experiments on goats showed that long-continued moderate doses of chlorate might be injurious.

E. HOLMES.

Field experiments with phosphate in 1927. A. N. LEBEDINTSEV (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 61, 5–21).

Determination of calcium in phosphorites. KAMINSKI.—See VII.

PATENTS.

Diminishing or preventing loss of carbohydrates in root-crops when stored. SCHERING-KAHLBAUM A.-G. (B.P. 308,820, 27.3.29. Ger., 29.3.28. Addn. to B.P. 266,695; B., 1928, 64).—The material worked up to a paste is treated with a narcotic in finely-divided liquid form.

L. A. COLES.

Powdered insecticide containing active ingredients of the root of the *Derris* species. ZAIDAN HOJIN RIKAGAKU KENKYUJO (B.P. 300,606, 17.7.28. Jap., 16.11.27).—Absorbent materials such as clay or kieselsgrub impregnated with the ether extract of *Derris* root, and to which powdered sulphur etc. have been added, are claimed as useful contact insecticides.

E. HOLMES.

Paper sacks (B.P. 307,005).—See V. **Ammonium potassium phosphate** (B.P. 300,919).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Present relation between the sugar content of the beet and the rendement. E. JELÍNEK (Z. Zuckerind. Czechoslov., 1929, 54, 87–88).—At the present time the yield calculated to the 88° rendement is inclined to be somewhat lower than corresponds to the actual sugar content of the roots. Thus with a sugar content of 18% and a total loss (including the sugar left in the molasses) of 1.7%, the difference is equal to 0.47. A table is given showing for a rendement of 88°, corresponding to a sucrose content of 93%, this difference for sugar contents of 17, 18, and 19, and losses of 1.5–2%. These figures indicate that for every 0.1 increase of polarisation the difference is increased 0.78%.

J. P. OGILVIE.

Determination of the volume and sp. gr. of the mark of the sugar beet. E. MÜLLER and J. PUCHERNA (Z. Zuckerind. Czechoslov., 1929, 54, 99–102).—Working with roots of South Moravian and Slovakian origin, the following average values were found for the mark: vol. in c.c. per normal weight 1.69, g./m.³ 1.333, hydrated mark (% by wt.) 8.61, juice factor 91.39 (cf. Spengler and Brendel, B., 1927, 234).

J. P. OGILVIE.

Use of sodium sulphite in place of [sugar] juice sulphitation. V. STANEK (Z. Zuckerind. Czechoslov., 1929, 54, 114–115).—Sulphuring being difficult, if not impossible, in the case of the juice of the present campaign, owing to the marked fall of alkalinity that is being observed, the addition of sodium sulphite is advised in its place. As small an amount as 0.01% of the roots generally suffices. An addition of soda can still be made in the normal manner before the second carbonatation.

J. P. OGILVIE.

Purifying diffusion juices by means of small quantities of lime and at temperatures above 100°. H. FRIEDRICH (Z. Zuckerind. Czechoslov., 1929, 54, 34–36).—Treatment of beet diffusion juice with 0.25–0.3% of lime (calculated on beets) precipitates almost all the removable impurities, but the precipitate cannot

be collected on a filter. Laboratory experiments showed, however, that after the limed juice had been heated for a short time in an autoclave at 1–1½ atm. (120° or over) the precipitate could be easily separated by filtering or centrifuging, yielding juice of pale yellow colour with a purity quotient about 3·5° above that of the original juice, and much greater increases in purity were obtained in the same way with very thin juices. In a factory trial with 10 hectolitres of diffusion juice of 17·5° Brix and 87·14% purity, limed and heated under pressure to 108° in an apparatus adapted for continuous working, the centrifuged juice had a purity of 93·62% and resembled thin juice from double carbonatation except that its alkalinity was 0·05% CaO, which would need to be corrected by slight carbonatation. The unwashed precipitate in the centrifuge formed a cake containing 31·5% of dry substance, representing 0·7% of the original beets. The dry substance contained 23·5% of sugar, 24·96% of ash, 8·37% of crude protein, 6·1% of fat, and 37·07% of nitrogen-free extractives. The method of treatment of juice described requires far less lime than the usual carbonatation process; it would dispense with lime kilns, for the slight carbonatation required to correct the alkalinity of the defecated juice could be operated from cylinders of gas; and the defecation cake would furnish a new and valuable fodder material.

J. H. LANE.

Causes and prevention of crystallisation in raspberry syrups. V. STANEK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1929, 54, 25–33).—Syrups made from raspberry juice and refined sugar, and containing the average proportion of moisture (about 34%), are liable to crystallisation of dextrose monohydrate if the added sugar is so far inverted that the syrups contain more than 50% of invert sugar. On the other hand, unless the ratio of uninverted sucrose to water is well below 2:1 sucrose may crystallise. With syrups of the usual acidity, corresponding to 0·5–0·6% of malic acid, heating for 2 hrs. at 90°, 1 hr. at 98°, or ½ hr. at 118° (ebullition) will invert about half the sucrose, but in presence of abnormal acidities different periods might be required. In any case heating is desirable to destroy any invertase present.

J. H. LANE.

Influence of some nitrogenous substances on the determination of invert sugar in sugar-factory products. M. HERLESOVÁ (Z. Zuckerind. Czechoslov., 1929, 54, 1–5).—In invert sugar determinations, by the gravimetric method and Schoorl's volumetric method, on 20 mg. of invert sugar in presence of 10 g. of sucrose, addition of ammonium sulphate corresponding to 40 mg. of nitrogen lowered the results by about 1 mg., i.e., about 5% of the amount of invert sugar present. Errors two to four times as great were produced by adding amounts of glutamine or asparagine containing 80 mg. of nitrogen, half of which is liberated on boiling with alkalis. Addition of 5 mg. of various purine bases occurring in beet molasses produced errors up to 25% on the low side, in absence of sucrose. A large proportion of the purine nitrogen was precipitated with the cuprous oxide. Norit will remove about 75% of the purine bases from molasses solutions, but it also removes some reducers.

J. H. LANE.

Formation of reducing substances on heating alkaline sucrose solutions, and their bearing on analysis. M. HERLESOVÁ (Z. Zuckerind. Czechoslov., 1929, 53, 749–754).—On prolonged boiling of alkaline sucrose solutions reducing substances are formed which, unlike invert sugar, are very resistant to alkalis. The author boiled 20% sucrose solutions containing sodium carbonate and sodium hydroxide equivalent to 0·05% of lime, for periods up to 38 hrs., and after 18 hrs. found reducers corresponding to 0·1% of invert sugar (calculated on sucrose) which did not increase on further heating. The solutions were then concentrated and boiled to grain *in vacuo*, yielding two crops of sugar and a dark molasses containing reducers corresponding to 0·6% of invert sugar, calculated on sucrose. None of the clarifying agents used in sugar analysis precipitated this reducing matter completely; neutral lead acetate removed only about 43%, basic lead acetate and Norit were more effective, and basic lead nitrate precipitated about 66%. It is concluded that the reducing power of normal beet molasses of alkaline reaction is not due to invert sugar.

J. H. LANE.

Cane-molasses distillery slop. E. K. NELSON and G. A. GREENLEAF (Ind. Eng. Chem., 1929, 21, 857–859).—At present the best that can be done with the distillation residue from cane-molasses distillery worts is to concentrate and burn it, recovering ammonia from the combustion gases and potassium salts from the ash. A sample from a large American distillery contained 51·22% of total solids dried at 100°, 12·65% of ash (including 5% K₂O, 1·78% CaO, 2·17% Cl, and 0·21% P₂O₅), 1·43% N, 1·11% of pentoses, 4·63% of reducers (calc. as invert sugar), and 11·67% of matter non-diffusible through a collodion membrane. About 0·6% of glycerol was extracted. The volatile acids present comprised about 0·9% of acetic and 0·3% of formic acid. The non-volatile acids identified were: about 0·5% of succinic, 1% of tricarballic, 3% of lactic, a small amount of aconitic, and a possible trace of citric acid. The original molasses from which the slop was derived contained neither succinic nor tricarballic acid and only a small quantity of lactic acid. Fermentation is responsible for the formation of succinic and lactic acids and probably for the conversion of aconitic into tricarballic acid.

J. H. LANE.

Beet fat and beet sterol. P. PAVLAS (Z. Zuckerind. Czechoslov., 1929, 54, 9–23).—According to data published by previous investigators, sugar beets contain 0·05–0·2% of fat. Technical cossettes employed by the author contained, in the fresh state, 0·126%. Ether alone does not extract all the fat, even from finely-powdered, dried cossettes; a subsequent extraction with alcohol is necessary. In the factory about half of the fat in the beets passes into the diffusion juice and half remains in the slices, only about 6% of the total being carried away in the diffusion waste waters. The pressing of the exhausted slices expels about 10% of the fat in them, with the press waters. The diffusion juice loses almost all its fat in the carbonatation precipitate, so that the filtered thin juice contains only traces. Beet fat differs considerably from ordinary fats. It is strongly contaminated with nitrogenous substances

and contains about 40% of unsaponifiable matter which, on dissolving in hot alcohol and cooling, deposits beet sterol. This is really a mixture of closely related sterols one of which, $C_{28}H_{46}O_2$, was isolated by the author. Beet fat contains about 10% of sterols, partly in the free state and partly as esters of fatty acids. J. H. LANE.

Effect of the temperature of mixing and the concentration of the potato starch mucilage on the Saare test. SPROCKHOFF (Z. Spiritusind., 1929, 52, 358—359).—The Saare test is a means of testing the viscosity of a starch and its ability to form a mucilage of good quality. For the test 9 g. of air-dried (20% of moisture) potato starch are made into 200 g. of paste with water of known temperature. Immediately after mixing, a disc of 22 mm. diam. is sunk into the paste to a fixed depth, and the determination made of the weight necessary to withdraw the disc from the cold mucilage 24 hrs. later. The Saare values reach a maximum when the temperature of mixing lies between 72—75°, and thereafter decrease with great rapidity. The values also show marked increases when the concentration of the paste is raised. With quantities of starch corresponding to 7 and 7.66 g. of dry substance, the Saare values are 58.6 and 99.2. This sensitivity to small variations in concentration necessitates the use in the test of starch weighed according to the amount of dry substance present. Even then the sources of error due to temperature and concentration are too great, and the determination of the quality of starch by viscosity measurements is considered preferable.

C. RANKEN.

Fertilisers and sugar-beet growth. ENGELS.—See XVI.

PATENTS.

Treatment of syrup. F. LAFEUILLE (B.P. 301,304, 4.10.28. Fr., 26.11.27).—Factory or refinery syrups are boiled, grained, and crystallised in a rotating vacuum vessel of cylindrical or other elongated form, provided with a jacket or internal tubes for steam or hot or cold water. In this vessel the whole of the syrup for a strike is from the outset boiled blank to a predetermined density which is such that the required amount of grain may be then formed by applying a higher vacuum or shutting off the steam or circulating cooling water for a short time. After grain has been formed boiling is resumed with the charge continuously in motion, and a very dense masseculite can thus be produced, which is cooled in motion in the same vessel; before cooling, some molasses from preceding strikes may be drawn in. It is claimed that the process requires less skill and experience than present methods of sugar boiling, and enables a higher degree of exhaustion to be attained in one boiling than is possible with a stationary pan. J. H. LANE.

Preparation of glucose. COMMERCIAL ALCOHOL Co., LTD. (B.P. 292,918—9, 22.6.28. Ger., 25.6.27).—(A) In the production of sugar from wood or the like by the action of concentrated acid followed by saccharification of the partially decomposed cellulose, the saccharification is effected, without addition of water as such, by exposing the material, after the acid treatment, to

steam or mixtures of steam and air, whereby the material is agitated, warmed, preferably not above 70°, and diluted by condensation of the steam. The primary treatment with acid may be carried out below 0° to prevent premature saccharification. (B) Wood or the like, moistened with acid or water, is exposed, preferably below 0°, to the action of strongly cooled gaseous hydrochloric acid or mixtures of this with air, so that no substantial formation of sugar occurs. The operation is arrested before the material has formed a mash, and saccharification is then effected as in (A). The sugar solution, separated from the lignin, may be decalcified and concentrated at about 70° or below by blowing air at 90—100° through it in suitable vessels. Apparatus is claimed consisting of an upright, cylindrical, jacketed vessel with an internal mixing screw for the primary acid treatment, and below this a conical saccharification vessel in which the material is kept in motion by the injection of steam and air at the lower end.

J. H. LANE.

Production of glutanic acid [in sugar factories]. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, and K. BROMIG (B.P. 320,589, 31.1.29).—Waste liquors from sugar factories or distilleries are boiled with concentrated hydrochloric acid (e.g., 100 g. of molasses waste liquor of $d_{1.35}$ —1.40 are boiled for 4—5 hrs. with 100 c.c. of acid), filtered hot, cooled, again filtered to remove potassium chloride, concentrated to two thirds of its volume, and saturated with gaseous hydrochloric acid below 20°. Crude glutamic acid hydrochloride separates and may be purified by dissolving in 10% hydrochloric acid, decolorising with active carbon, and saturating with gaseous hydrochloric acid. J. H. LANE.

XVIII.—FERMENTATION INDUSTRIES.

Distribution of the total, soluble, and coagulable nitrogen between the distal and proximal ends of barley and malt grains. W. WINDISCH and P. KOLBACH [with H. HANKE] (Woch. Brau., 1929, 46, 459—463).—Barleys and the corresponding malts are cut in a Pohl's cutter and the two ends of the grain analysed separately. In barley the proximal (embryo) end contains a higher proportion of soluble nitrogen than does the distal end. The proportion is greatly increased by malting, especially at the proximal end, the increase in which exceeds that at the distal end by 2.2—2.6 times. The increase of permanently soluble nitrogen is greater than that of coagulable nitrogen. The possible applicability of such a method to the control of malting is discussed, but an improvement on Pohl's cutter would be desirable, as this does not divide all samples of grain into portions of uniform ratio. F. E. DAY.

Extract determination in barley. C. ENGELHARD (Woch. Brau., 1929, 46, 463—465).—Barley meal (25 g.) and wheat malt meal (10 g.) of high diastatic power are mashed in the water-bath with 150 c.c. of water at 20° with continual stirring for 2 hrs. The temperature is slowly raised till the bath has boiled for 10 min. The total time from commencement of heating is 1½ hrs., including pauses of 10 min. at 70° and 5 min. at 80°. After cooling in 5 min. to 45°, a further 15 g. of wheat

malt meal and 50 c.c. of water at 45° are added and the temperature is raised to 50° in 5 min. This is maintained for 25 min., then the bath is raised to 75°, 50 c.c. of water at 70° being added when the mash reaches 70°. The bath is held at 75° for 1 hr., when the mash is cooled, made up to 450 g. with water, filtered, and the extract determined as usual. A correction is made for the wheat malt, which is similarly mashed using double the amount stated above, and for the correction halving the extract obtained. The results on five barleys of the 1928 crop differ by only 0.02–0.15% from those actually obtained by malting (Pilsener), whereas both Pavlovski's and Seibriger's methods gave too high results.

F. E. DAY.

Effect of the protein content of barley on quality of dark beer. H. STADLER (Woch. Brau., 1929, 46, 479–483).—Two malts, *A* and *B*, were made from barley with a high content of protein (15.8%). The flooring temperature of *A* was kept high and reached 25.6°, whereas that of *B* approximated to normal and never exceeded 19.3°. Comparison was made with a malt, *C*, prepared under normal conditions. The amounts of total protein in *A*, *B*, and *C* were 15.95, 15.80, and 10.9%, respectively, whilst the colours were 1.6, 1.4, and 0.8, and the p_H values 5.55, 5.54, and 5.81. The contents of the different types of nitrogenous constituents did not vary except that the quantity of glutelin nitrogen in *C* was very much lower, which explained why the worts from *A* and *B* gave higher and denser heads of yeast compared with those from *C* at the stage of fermentation. The values of the beers from *A* and *B* differed little. The beer from *C* contained 6.55% N (calc. on dry extract), whilst for *A* and *B* the values were 10.24 and 10.73%, respectively. The p_H values of the beers were 4.74, 4.75, and 4.40, whilst the surface tensions amounted to 64.0, 64.0, and 67.9. The beer from *A* far excelled those from *B* and *C*. Its flavour was immeasurably superior and the beer drank rounder and fuller. Beers from *B* and *C* had similar flavours, with *B* somewhat sweeter. The differences between the values obtained for *A* and *B* as malt, wort, and beer were insufficient to explain the marked superiority of *A*, and the factor to which the difference is due, and which is created by the high temperature of germination in conjunction with the high protein content of the barley, remains unidentified. C. RANKEN.

Boiling of brewery wort. E. JALOWETZ (Woch. Brau., 1929, 46, 483–486).—Since diminishing the content of protein in beer lessens the risk of infection and allows the curtailment of the length of time the beer is retained in the cellars to ensure ripening, it is desirable that, when the wort is boiled, the coagulation and separation of the proteins should be efficient. The chief factor which influences the degree of flocculation is the energy of the boiling action. The wort should be agitated and kept in turbulent motion during the boiling, which should be at as high a temperature as possible. The area of the heating surface should be extensive, and the evaporation should amount to 8–10% per hr. The duration of boiling is usually 1½–2 hrs., and is dependent on the type of boiling. A wort may be as efficiently boiled by steam-heating

as by direct firing, provided the steam-heated coppers have a sufficiently large heating surface. To ensure a greater turbulency of the worts during boiling, many types of apparatus, which are described in the paper, can be used in conjunction with the wort copper.

C. RANKEN.

Pure fermentation of wines by the so-called "fermentation superquatre" method of [yeast] selection. L. SEMICHON (Ann. Falsif., 1929, 22, 466–471).—Diseases in wines are caused in the early stages of fermentation by moulds and wild yeasts, and it is not until the alcohol content has risen above 4% that the true wine-yeast cells (*Saccharomyces ellipticus*) begin to predominate. If 4–5% of alcohol be added to the must, pure alcoholic fermentation will proceed from the commencement, but such addition is prohibited by law. To obviate this difficulty in the manufacture of white wine the fresh must is added to an equal volume of new wine containing about 8% of alcohol, so that with an initial alcohol concentration of about 4%, fermentation will be effected solely by the elliptical cells contained in the wine. If new wine is not available at the commencement, some of the previous year's vintage may be used. For red wines, after being well trodden, the bottom half of a vat is drawn off and added to new wine, whilst fresh must is added to the residue in the vat. The advantages of this method of alcoholic fermentation are numerous: the alcohol content is often 1% higher, the wine is more fruity and of better bouquet, red wines are of better colour, and the keeping qualities are greatly improved. H. J. DOWDEN.

Use of potassium ferrocyanide in wine-making. P. MALVEZIN (Ann. Falsif., 1929, 22, 486–491).—A polemical retort to Couerbe (cf. B., 1929, 793).

H. J. DOWDEN.

Detection of cider in wine by the sorbitol method. J. WERDER (Z. Unters. Lebensm., 1929, 58, 123–131; cf. B., 1929, 619).—The presence of unfermented sugar in the sample under examination interferes with the detection of the sorbitol. The process is equally applicable to spoilt and unspoilt wines. W. J. BOYD.

Determination of alcohol in brandy from the refraction and the density. A. BECKEL (Z. Unters. Lebensm., 1929, 58, 78–98).—The alcohol refraction tables of Wagner show errors up to 0.7° Zeiss, thus giving too high values for the alcohol content. The formula $A = 1.3(KZ/100) \times [30.07 - (KZ/100)] - 6.16$, in which A = vol.-% of alcohol and $KZ = 1000 + \text{refraction (° Zeiss)} - \text{wt. per litre}$, gives values for alcohol contents between 20 and 70 vol.-% within ± 0.05 vol.-% of those obtained from the density. The extract content of brandies and wine distillates is without appreciable influence on the values so obtained. The extract content E in g. per 100 c.c. can be calculated from the formula $E = [(B + L) - (B + L)_A]/7.62$, in which $(B + L)$ is the sum of refraction (° Zeiss) and wt. per litre of the sample, and $(B + L)_A$ is the sum of refraction and wt. per litre of an alcohol-water mixture of the same concentration as the sample. A table is given from which the alcohol content and the value $(B + L)_A$ appropriate to any value of KZ can be readily found, and also a nomograph is supplied for rapidly

ascertaining the extract content from the value $(B + L) - (B + L)_A$. W. J. BOYD.

Sulphurous acid in wine. ROTHENFUSSER.—See XIX. **Iodised malt.** MEYER.—See XX.

XIX.—FOODS.

Detection of artificially bleached flours. G. JØRGENSEN (Ann. Falsif., 1929, 22, 471—486).—The difficulties which beset the detection of artificially bleached flours are described, with special reference to the investigations of Winton (U.S. Dept. Agric. Bull. No. 137, 1911), of Kent-Jones (cf. B., 1927, 762), and of H. Jørgensen (cf. B., 1928, 585). Natural flours exhibit great variability in colour, so that a dark flour may have undergone considerable bleaching without reaching the colour value of the lighter flours. To determine the nitrogen content for the detection of flours bleached by nitrogen peroxide or nitrosyl chloride is inconclusive, since the oil extracted from natural flours has been shown to contain 0.64—1.01% N. Also flour in storage may absorb nitrogen peroxide from the air, so that the presence of nitrous acid in the flour is not conclusive proof of bleaching. Similarly, the presence of chlorine does not prove that the flour has been bleached by chlorine or chlorine compounds, since the oil of natural flours has been found to contain 0.51% Cl. Bleaching by benzoyl peroxide (Novadelox) is detected with certainty if the oil extracted by ether contains benzoic acid, whilst an abnormally low value for the colour of the carotene extract strongly suggests bleaching by oxidising agents. The influence on colour of storing flour in the dark and in light, at various temperatures, has been studied and the formation of moulds investigated. In forming an opinion on any flour showing abnormal values, all factors such as age, colour of the gasoline extract, humidity, fat content, ether-soluble chlorine content, conditions of storage, and the presence of moulds must be taken into consideration. H. J. DOWDEN.

Examination of goats' milk for unboiled milk. A. G. HOLBOROW (Analyst, 1929, 54, 658—659).—The most sensitive of the various reagents which form colorations with the enzymes of unboiled milk are "ortol" and *p*-phenylenediamine, which will detect 2.5% of unboiled milk with certainty; with guaiacol, 30% of unboiled milk escaped detection. To 5 c.c. of the milk is added 1 drop of 10% hydrogen peroxide (90 vol.) followed by 1 c.c. of freshly-made 1% aqueous solution of "ortol." (If less than 10% of unboiled milk is present 2 drops of hydrogen peroxide are used.) The deep red colour is formed almost at once in presence of unboiled milk, and only a slight pink coloration results after some time with boiled milk. *p*-Phenylenediamine, giving an indigo-violet colour, is used as a confirmatory reagent, in a 0.25% solution with a drop of hydrogen peroxide (90 vol., equally diluted) and 5 c.c. of milk; readings should be taken 1—2 min. after mixing. Unboiled milk develops a blue tint in 3—5 min. D. G. HEWER.

What molecular f.p. [depression] of water should be adopted as standard in determining the

f.p. of milk? P. WEINSTEIN (Z. Unters. Lebensm., 1929, 58, 136—139).—In order to secure uniformity the adoption of the value 1.90 for the molecular f.p. depression of water is advocated. W. J. BOYD.

Resistance of nickel and monel metal to corrosion by milk. R. J. MCKAY, O. B. J. FRASER, and H. E. SEARLE (Amer. Min. Met. Eng. Tech. Pub., 1929, No. 192, 47 pp.).—The maximal amount of nickel dissolved by milk was 12 in 10^6 ; monel metal was rather less soluble. Agitation raised the solubility at the pasteurising, but not at the ordinary, temperature; the solubility in buttermilk was increased by aeration or by rise of temperature. At low temperatures winter milk was more corrosive than summer milk. Lactic acid dissolved more nickel than did milk of equivalent acidity. The effect of milk constituents was examined. In tests with technical apparatus milk dissolved 1.3 pts. of nickel in 10^6 . CHEMICAL ABSTRACTS.

Composition of Irish winter butter. P. S. ARUP (Analyst, 1929, 54, 634—645).—A total of 310 samples of cream or butter from 30 creameries and 2 agricultural schools in the Irish Free State were taken between Oct. 27, 1927, and Mar. 31, 1928, and 270 samples from 36 creameries and 3 schools between Nov. 1, 1928, and Feb. 23, 1929. Of these 16.1 and 32.6%, respectively, had Reichert-Meissl (R.-M.) values between 24 and 20, and all these occurred between Oct. 31 and Jan. 31. The predominating factor influencing these low values is the lactation period, calving taking place almost exclusively during one season of the year; the unusual cold in Feb., 1929, had no noticeable effect in checking the upward trend of the Reichert-Meissl values. The maximum Polenske values (R.-M. values in brackets) were 3.2 (31.4) and 3.4 (29.2); and the minimum 1.15 (22.2) and 1.30 (21.1 and 24.9); Kirschner values, maximum 24.1 (32.7), minimum 14.1 (20.4), on the average, were 73.4% of the average R.-M. value, and for the second period varied from 67.3 to 81.2 as a percentage of the corresponding R.-M. value. Brownlee's conclusion (B., 1925, 609) that the Avé-Lallemant value cannot be used for distinguishing genuine butters of low R.-M. value from adulterated butters of higher R.-M. values is confirmed. As a measure of the volatile acids in pure butter, the brine-soluble and -insoluble figures are not regarded as offering any particular advantage over the R.-M. value. D. G. HEWER.

Routine determination of salt in butter and margarine. P. S. ARUP (Analyst, 1929, 54, 658).—By substituting 10 c.c. of hot water for the acetone in Stewart's method for making this determination (B., 1928, 425), similar results are obtained which are in substantial agreement with those obtained by standard methods involving the separation of the fat. D. G. HEWER.

Fluorescence phenomena and their application to investigating adulteration in cacao butter. J. R. RIPERT (Ann. Falsif., 1929, 22, 459—463).—Adulteration of cacao butter may be detected by the colour of the fluorescence exhibited by the material when exposed to ultra-violet light. The fat is extracted with

chloroform, and, after evaporation, the residue is decolourised by heating with hydrogen peroxide (100-vol.) at 90°. Pure cacao butter fluoresces with a blue-violet colour, but additions of Rotterdam butter or "beurre Radisson" produce green and pink tints, respectively. To facilitate comparison of the colours an apparatus has been devised which is essentially a combined colorimeter and differential photometer. The sample under investigation and a standard are viewed through monochromatic red, green, yellow, and blue filters, alternately, and the radiation intensities are matched by moving a diaphragm. The relative intensities in the various colours are read on an arbitrary scale.

H. J. DOWDEN.

Physics of the ice-cream mix. I. Relationship between basic viscosity and whipping capacity of ice-cream mixes. A. LEIGHTON and O. E. WILLIAMS (J. Physical Chem., 1929, 33, 1481—1484; cf. B., 1928, 33).—An inverse linear relationship exists between whipping capacity and basic viscosity, but since no simple relationship exists in a mix when the viscosity is increasing with the homogenisation temperature, basic viscosity is not the only factor which influences whipping capacity; further, no simple relationship exists when viscosity is altered by the pressure of homogenisation while the temperature remains constant.

L. S. THEOBALD.

Basic viscosity and plasticity of ice-cream mixes. A. LEIGHTON and F. E. KURTZ (J. Physical Chem., 1929, 33, 1485—1488; cf. preceding abstract).—The basic viscosity of mixes may be considered as a true measurement since no plastic properties could be detected in mixes which had been stirred. Addition of gelatin to a mix affects the viscosity, giving a higher yield value and a greater consistency to the unstirred mix.

L. S. THEOBALD.

Viscosity-plasticity measurements of the effect of gelatin on ice-cream mixes. F. E. KURTZ (J. Physical Chem., 1929, 33, 1489—1494; cf. preceding abstract).—The effects produced by equal concentrations of different commercial gelatins on the basic viscosity, consistency, and yield value of otherwise identical ice-cream mixes have been investigated, and are discussed in relation to the grading of gelatins for ice-cream manufacture.

L. S. THEOBALD.

Nutritive value of proteins in certain kinds of sausage and other meat food products. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1929, 39, 531—537).—Feeding experiments with young albino rats indicate that the nutritive value of the protein varies considerably in similar prepared-meat products made by different establishments. The nutritive value also varies according as the protein is derived from maize meal or meat.

E. HOLMES.

[Malic and tartaric] acid contents of grapes during ripening. E. HUGUES (Ann. Falsif., 1929, 22, 463—466).—The relative proportions of malic and tartaric acid in two varieties of grape have been determined by Mestrezat's method (cf. A., 1906, ii, 635). Samples of the grapes (about 1 kg.) were gathered as

uniformly as possible a few days before turning colour, when fully coloured, and when completely ripe. The berries were separated from the bunches, counted, weighed, crushed in a mortar, and mixed with aqueous alcohol at 25°. The supernatant liquid was decanted and the residue extracted twice with warm water, the total extract being diluted to 1 litre. The results expressed in g. per kg. of grapes show that with both varieties the malic and tartaric acid contents decrease as ripening proceeds, and with one variety, at maturity, the malic acid had entirely disappeared. The decrease in the tartaric acid is, however, only apparent, and is due to the increase in weight of the grapes during ripening, for when the results are expressed on the basis of 1000 berries it is found that the total tartaric acid present is the same when immature and when fully ripe.

H. J. DOWDEN.

Sugar: acid ratio of oranges. D. J. ESSELEN (Farming in S. Africa, 1929, 4, 87—88).—Wilting increases the sugar: acid ratio, but considerable loss of weight occurs.

CHEMICAL ABSTRACTS.

Pectin [in the preparation of jellies]. C. GRIEBEL and F. WEISS (Z. Unters. Lebensm., 1929, 58, 189—201).—In confirmation of the work of Lüers and Lochmüller (B., 1927, 568), it was found that an approximate direct proportionality exists between the methoxyl content and the gelatinising power of a pectin product. Commercial pectin preparations examined had, with one exception, a pectin content of 4.42—5.04% as calcium pectate and a methoxyl content of 10.55—12.65% (calc. on the calcium pectate). Only an inconsiderable loss of gelatinising power occurs in the usual boiling process of fruit preservation. Jellies were prepared by the method of Lüers and Lochmüller (*loc. cit.*), using sugar (60%), water, and tartaric acid and a boiling time of 15 min.; 0.5—0.6% of high-grade pectin gave a jelly superior in strength to some commercial jellies. Methods of evaluation of pectin preparations are given, including determinations of pectin as calcium pectate in the preparation and in the alcohol precipitate, of the methoxyl content of the alcohol precipitate, and of the gelatinising power of a jelly, prepared under definite conditions, by means of the pectinomometer.

W. J. BOYD.

Examination of honey. J. FIEHE and W. KORDATZKI (Z. Unters. Lebensm., 1929, 58, 69—77).—A solution of honey (100 g.) in water (400 c.c.) is shaken with 5 c.c. of 30% zinc acetate solution and 5 c.c. of 15% potassium ferrocyanide solution and filtered. The filtrate is thrice extracted in a 500-c.c. Maassen percolator each time for 4 hrs. with 40—50 c.c. of ether. An equal volume of light petroleum and 10 g. of anhydrous sodium sulphate are added to the ether extract and the mixture is shaken, left for 24 hrs., and filtered. The filtrate is gently evaporated, the residue dissolved in 10 c.c. or 20 c.c. of water, and the solution filtered. Both the natural and the artificial product yield a few eg. of a colourless or golden residue having the aroma of the respective product. That from artificial honey is almost entirely soluble, however, whereas from genuine honey it is only partly soluble in water. Practically no

hydroxymethylfurfuraldehyde is lost in the above process and the extract is free from sugars. A suitable portion of the aqueous solution of the ether extract is titrated with alkaline iodine solution as described by Troje (B., 1925, 1004; 1929, 146, 375) and another portion is precipitated by phloroglucinol as described by the same author (*loc. cit.*) with slight modifications. With artificial honey the two methods give values for hydroxymethylfurfuraldehyde which agree approximately, whereas genuine honey gives a negative result by the precipitation method and a considerable iodine value. The difference between the values obtained by the two methods is therefore a measure of the quantity of genuine honey present in a "honey" product.

W. J. BOYD.

Composition of Philippine coffee. A. VALENZUELA (Philippine J. Sci., 1929, 40, 349—351).—Philippine coffees appear to compare favourably with those of other origin.

Quantitative and qualitative determinations of castor seed press-cake in cattle feed cake. M. WAGENAAR (Pharm. Weekblad, 1929, 66, 1021—1025).—The characteristic appearance under the microscope of the cells of the hard, resistant, interior tissue of the seed husk reveal the presence of the poisonous castor-seed cake. The only other seed having similarly resistant husk tissue cells is that of *Aleurites*; not only are the microscopical characteristics different, however, but cake from this seed is not used in Europe. Quantitative determinations may be based on the number of husk cells counted in a given area, on the basis of 1500 sq. mm. of husk tissue per g. of oil-free castor-seed powder.

S. I. LEVY.

Detection and determination of sulphurous acid [in foods]. S. ROTHENFUSSER (Z. Unters. Lebensm., 1929, 58, 98—109).—A suitable quantity of the sample (*e.g.*, 50 c.c. of wine; 30 g. of minced flesh, fat, or oil; 20 g. of dried fruit) is mixed with 250—300 c.c. of water in a 500-c.c. flask and approximately neutralised if alkaline. Some finely-powdered pumice is added and the flask is attached by means of a delivery tube to a condenser and receiver. 5—10 c.c. of 25% phosphoric acid are added through a tap funnel, and in the case of minced flesh 5 c.c. of liquid paraffin are also added. The receiver (100 c.c. capacity and graduated) is provided with a mixture of 5 c.c. of 5% benzidine solution in 96% alcohol with 5 c.c. of 30% acetic acid to which 5 c.c. of 3% hydrogen peroxide are added. The adapter attached to the condenser terminates in a bulb with a small opening and reaches to the bottom of the receiver. The contents of the flask are boiled so that the sulphurous acid passes into the receiver and is precipitated as benzidine sulphate. If no precipitation occurs sulphurous acid is absent and distillation may be discontinued. Generally it is sufficient to distil over about 60 c.c. Five min. after conclusion of the distillation the benzidine sulphate is transferred to a weighed Gooch crucible by suction, washed twice or thrice with 5 c.c. of water, dried at 105° for 30 min., cooled in a desiccator, and weighed. The weight of precipitate $\times 0.234$ gives the weight of sulphur dioxide present.

Interference by other substances (*e.g.*, formaldehyde) is practically excluded for reasons given. W. J. BOYD.

Determination of boron compounds in foods and drugs. I. A. S. DODD (Analyst, 1929, 54, 645—650).—An historical survey of the determination of boron accentuates the importance of separating the boron compounds from organic matter, eliminating phosphates and carbonic acid, and of the choice of suitable indicators and media for the titration.

D. G. HEWER.

PATENTS.

Production of dough for making bread and other baked products. F. PASSEK and H. BOLLMANN (B.P. 305,217, 29.1.29. Ger., 3.2.28).—Bread containing soya flour can be prepared satisfactorily by incorporating also wet gluten in the dough mix, and adding a proportion of leaven and a small quantity of calcium carbonate.

E. B. HUGHES.

Treatment of dairy products. S. B. CHAMBERS (U.S.P. 1,735,313, 12.11.29. Appl., 19.8.25).—Raw cream (100 pts.) mixed with malt diastase (1½ pts.) of diastatic strength 140° F. (Lintner test) is held first at 51.5—57.5° for 1½ hrs., then at 65° for about 5 min., and afterwards cooled and churned, giving butter of superior keeping qualities.

E. B. HUGHES.

Preserving vegetables. FROZEN FOOD PRODUCTS, LTD., and A. H. PENFOLD (B.P. 321,448, 9.8.28).—The vegetables (which may be in layers or in containers which need not be airtight) are dusted with dextrose, rapidly frozen at —27°, and maintained at not above —1°.

E. B. HUGHES.

Process and apparatus for production of puffed [food] product. ANDERSON PUFFED RICE Co., Assees. of A. P. ANDERSON (B.P. 300,194, 23.8.28. U.S., 8.11.27).

Separation of granular material (B.P. 321,465).—See I. **Ozonisation of oils** (B.P. 321,633).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Evaluation of ipecacuanha root and its preparations. H. BAGGESGAARD-RASMUSSEN and H. WAEL [with E. V. CHRISTENSEN] (Dansk Tidsskr. Farm., 1929, 3, 250—272).—The methods for the extraction and determination of the alkaloids in ipecacuanha preparations have been studied, and the following procedure is recommended. The powdered root (2.5 g.) is moistened with 2 g. of 10% ammonia in a stoppered flask, and 50 g. of ether are added. After keeping for 30 min. with frequent vigorous shaking, the solution is filtered and 40 g. of the filtrate (= 2 g. of the root) are evaporated to dryness; a few more c.c. of ether are then added and evaporation is repeated. The residue is dissolved in 2 c.c. of alcohol and 5 c.c. of 0.1N-hydrochloric acid, 20 c.c. of water and 5 drops of methyl-red are added, and the excess of acid is titrated with 0.1N-borax. The number of c.c. of hydrochloric acid used multiplied by 1.20 gives the percentage of alkaloids present, calculated as emetine. Unless two successive extractions

with ether are made the results are about 3% too low, but may suffice for technical purposes. Fluid extracts and infusions must be concentrated to 5 c.c. and 0.5 g. of gum tragacanth added before the extraction with ether in order to prevent retention of alkaloids by the insoluble residue. No satisfactory method of analysis could be found for syrups containing 0.01–0.02% of alkaloid, the results invariably being much too low.

H. F. HARWOOD.

Determination of morphine in opium according to the method of the French Pharmacopœia. M. BONHOURE (J. Pharm. Chim., 1929, [viii], 10, 442–446).—The procedure of the French Codex (1908) method for the determination of morphine in opium and its preparations introduces a regular and constant error due to the morphine remaining in solution. A critical examination of the method showed that 50 mg. should be added to the result. With this correction consistent and accurate values are obtained.

E. H. SHARPLES.

Identification of apiole. J. KING (Analyst, 1929, 54, 567–573).—The application of the usual methods used for the examination of apiole to commercial apioles shows that the determination of the methoxyl content, by a modified Zeisel process, is the most distinguishing criterion taken in combination with physical data. The apparatus used has ground glass joints wherever iodine, hydriodic acid, or organic iodine compounds may come in contact with them, and carbon dioxide is used to sweep the decomposition products into the absorption tubes. The oil is weighed into an open glass capsule which is lowered into the first bulb, 20 c.c. of 57% (by wt.) hydriodic acid are added, and purified carbon dioxide is passed at the rate of 3–5 bubbles per sec. The reaction bulb is kept at 140° and the second bulb, containing red phosphorus suspended in water, at 70°; the absorption bottles each contain 10% alcoholic silver nitrate solution. The reaction takes about 60 min., after which the alcohol is evaporated, boiling water and a little dilute nitric acid are added, and the silver iodide is collected and washed. An examination of 7 commercial apioles showed a great divergence in composition: one sample was an almost pure glyceride; one was grossly adulterated with castor oil; two English apioles contained, respectively, about one third and one half of their weight of glyceride; two samples with low methoxyl content and saponification values were probably distilled rather than extracted oils. In order to obtain information by the sulphuric acid colour test, 0.033 g. of apiole was stirred with 10 c.c. of concentrated sulphuric acid, a part of the homogeneous mixture diluted with sulphuric acid until a concentration of 1:7500 was obtained, and the colour immediately read in the tintometer. Comparison of the red component with that given by known specimens showed great variations.

D. G. HEWER.

Luminescence analysis of homœopathic potency [of drugs]. H. NEUGEBAUER (Pharm. Zentr., 1929, 24, 1511–1515).—The colours obtained with various drugs in increasing concentrations and under specified conditions are described.

S. I. LEVY.

Iodised malt. L. MEYER [with H. BEUTELSPACHER] (J. Landw., 1929, 77, 275–286).—The iodine content of barley malt was increased by malting with potassium iodide solutions, maximum values being reached with a 5% solution when 36% of the iodine used was taken up by the grain. The percentage utilisation by the barley of the iodine of the malting liquor was greater when smaller concentrations were used. The iodine absorbed by the malt exists partly in the form of organic compounds insoluble in alcohol. The proportion of such compounds may be increased by the above treatment to 100 times its normal value. The total iodine absorbed by the malt is determined by the concentration of the potassium iodide solution used in the malting.

A. G. POLLARD.

Determination of arsenic in some phenylarsinic acids. A. LEULLER and Y. DREYFUSS (J. Pharm. Chim., 1929, [viii], 10, 446–448).—The following modification of the French Codex method for the determination of arsenic in sodium arsinitate is quicker and gives results of the same accuracy with atoxyl, stovarsol, and novarsenobenzol. The substance (0.1–0.2 g.) is gently boiled with 3 c.c. of a 1:2 mixture of 60% perchloric acid and concentrated sulphuric acid until the mixture is decolorised. The product is then transferred into an Erlenmeyer flask with 20 c.c. of water, 2 g. of potassium iodide are added, and the precipitate is redissolved by the addition of a little water. If no precipitate is formed sulphuric acid is added drop by drop until precipitation commences and this is then redissolved as above. After removal of most of the iodine by warming, the solution is cooled and the elimination is completed by addition of 0.1N-sodium thiosulphate and a slight excess of sodium bicarbonate. The arsenious acid is titrated with 0.1N-iodine (cf. Fleury, A., 1920, i, 578).

E. H. SHARPLES.

Spirit of camphor. N. SCHOORL (Pharm. Weekblad, 1929, 66, 977–986, 1001–1009).—The limits laid down by the Dutch Pharmacopœia are for camphor content 9.8–10.2% and for the dilute alcohol used as solvent 61.5–63.5%; acetone may be absent or 0.5% may be present. The effects of variations within these limits on the sp. gr., refractive index, rotatory power, and point of permanent separation of camphor on addition of water are considered. Lattice diagrams are given from which the composition may be read off when the sp. gr., refractive index, or the former and the point of separation have been determined, due allowance being made for the effect of acetone, if present. Since the sp. vol., molecular refraction, and sp. refraction are additive, the respective constants for the camphor used may be calculated.

S. I. LEVY.

Ethereal oil of green violet leaves. Japanese peppermint oil. Testing of blossom extracts. I. Sulphur compounds in mignonette extract. H. WALBAUM and A. ROSENTHAL (J. pr. Chem., 1929, [ii], 124, 55–62, 63–66, 67–79, 80).—See B., 1929, 1049, 1032, 1032, 1049.

Determination of hexamethylenetetramine. REBAGLIATI.—See III. **Boron determination in drugs.** DODD.—See XIX.

PATENTS.

Production of a hormone from female internal secretory organs. SOC. CHEM. IND. IN BASLE, Assocs. of S. FRÄNKEL (B.P. 292,962, 27.6.28. Ger., 27.6.27).—Extracts of the organs, after preliminary removal of phosphatides, cholesterol and its esters, fats, and free fatty acids, the last preferably by the addition on alkaline earth or lead acetate in the presence of methyl alcohol as solvent, are treated in light petroleum solution with an electrolyte, *e.g.*, potassium sulphate, silver nitrate, the anions and cations of which form salts insoluble in light petroleum with the cations and anions, respectively, of residual soaps of unsaturated fatty acids. After removal of the precipitated salts the product is crystallised from, *e.g.*, absolute ether at a temperature preferably below -50° , or is purified by conversion into its dibromide followed by debromination, *e.g.*, with a zinc-copper or zinc-mercury couple. The product, a hormone, $C_{16}H_{28}O_2$, has $[\alpha]_D^{25} +6^{\circ} 41'$, m.p. below 0° ; it is the lactone of an open-chain *hydroxy-acid* (m.p. about 40°) having a double linking in the Δ^{12} -position, and yields with hydrazine hydrate and thionyl chloride a compound, m.p. 104.5° , probably a bimolecular *hydrazide*, $C_{32}H_{60}O_3N_4$. L. A. COLES.

Manufacture of dry yeast for medical and pharmaceutical purposes. C. MASSATSCH, Assr. to MATRO GES.M.B.H. (U.S.P. 1,736,657, 19.11.29. Appl., 21.12.27. Ger., 4.4.25).—See B.P. 300,039; B., 1929, 73.

Acridine derivative for therapeutic purposes. E. ROTHLIN and F. MÜLLER, Assrs. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,738,449, 3.12.29. Appl., 22.12.27. Switz., 6.1.27).—See B.P. 283,184; B., 1929, 699.

Barbituric acid derivative. F. BOEDECKER (U.S.P. 1,739,662, 17.12.29. Appl., 21.11.25. Ger., 6.12.24).—See B.P. 244,122; B., 1927, 237.

Ozonisation of oils (B.P. 321,633).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photography. II. Rôle of sensitisers in photography and the latent image. F. E. E. GERMANN and D. K. SHEN (J. Physical Chem., 1929, 33, 1583—1592; cf. A., 1929, 893).—The 17% developability of the silver iodide-gelatin emulsions previously investigated (*loc. cit.*) is increased to 100% by treatment with metol, pyrogallol, quinol, amidol, "glycine," or sodium hydrogen sulphite. Sodium nitrite or sulphite, or acetonesemicarbazone is less effective. Since the sensitisers have no effect on the speed of the emulsion, the term sensitiser should be confined to substances which increase the developability of the grains. The rôle of sensitisers is discussed, and their action is considered to be merely one of preventing or retarding reversal. Solarised grains of silver iodide emulsions are practically identical with normal unexposed grains so far as behaviour towards sensitisers is concerned. Both the speed and sensitivity of normal iodide plates are reduced by several hours' treatment with chromic acid. Resensitisation restores

sensitivity but not speed, supporting the conclusion that sensitisers only increase developability. Trivelli's hypothesis of the latent image (A., 1927, 1154) is modified in order to correlate latent image formation with reversal. Sensitive specks are assumed to consist of silver germs and a molecular complex of the sensitiser which function during exposure as an iodine acceptor, and these sensitive complexes permit the development of exposed grains. The speed, however, depends on the size of the silver germs which are formed during the preparation of the emulsion, and hence cannot be altered by sensitisation. The sensitising complex undergoes a continuous decomposition by light, and thus brings about reversal when the sensitivity material is destroyed. The sensitisers are not optical ones, since the spectral emissivity of the sensitised plate is identical with that of a normal plate, and the insensitivity of silver iodide emulsions is not due to the Herschel effect.

L. S. THEOBALD.

Chloro-iodo-bromo-emulsion for colour sensitising. S. LANGGUTH (Brit. J. Phot., 1929, 76, Col. Suppl., 42—44).—An emulsion (the formula and making of which are given) containing silver chloride, iodide, and bromide in the ratio of 2:1:70 was found to be specially suitable for sensitising by bathing. Increased iodide content raised the contrast but resulted in incomplete fixation with sodium thiosulphate, whilst a larger proportion of chloride rendered the plates liable to fog. Details are also given for the pretreatment of the glass, and for the coating, drying, and sensitisation of the plates. The presence of lithium chloride is stated to improve the keeping qualities of both the sensitised and unsensitised plates. J. W. GLASSETT.

Optical sensitisation of silver halide emulsions. S. E. SHEPPARD and H. CROUCH (Z. wiss. Phot., 1929, 27, 180—192).—See B., 1928, 503.

Test for thiosulphates. JELLY and CLARK.—See VII.

PATENTS.

Photo-chemical production of printing plates. DR. BEKK & KAULEN CHEM. FABR. G.M.B.H., and M. THIMANN (B.P. 321,086, 3.12.28).—A sensitive chromate-colloid layer on a metal plate is covered after exposure but before development with a layer of resin or varnish, *e.g.*, by rubbing with a solution of sandarac in amyl acetate, the unexposed portions of the chromate-colloid layer are washed away together with the overlying varnish by treatment with water, and the exposed metal is etched, *e.g.*, with a solution containing ferric chloride, nitric acid, and potassium iodide. The surface is then covered with a fat colour, which is subsequently removed, together with the underlying layers, from the unetched portions by treatment with spirit or with sodium hydroxide solution which may also contain sodium carbonate. L. A. COLES.

Photographic or photo-mechanical reproduction of surfaces. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 320,892, 23.7.28).—Wood, leather, textiles, etc. of which the surface pores have been filled with material capable of absorbing X-rays, *e.g.*, by rubbing.

with a paste of barium sulphate and collodion, is pressed on to a photographic plate and X-rays are passed through the material. L. A. COLES.

Preparing pictures from light-sensitive layers containing organic colloids. G. KÖGEL (U.S.P. 1,724,666, 13.8.29. Appl., 11.8.28. Ger., 2.9.26).—See B.P. 315,236; B., 1930, 38.

[Washing apparatus for] photography. G. C. BEIDLER (B.P. 321,700, 18.6.28).

Photometer [for astronomical purposes]. C. ZEISS (B.P. 307,880, 9.3.28. Ger., 15.3.28).

Cellulose compositions (B.P. 319,073).—See V. Transferring veining etc. of wood (B.P. 321,591).—See IX.

XXII.—EXPLOSIVES; MATCHES.

Calibration of cordeau detonant for measuring time in the Dautriche method of determining detonation velocities. H. SELLE (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 420—422).—The accuracy of the detonation velocities of explosives as determined by the Dautriche method depends on the accuracy with which the detonation velocity of T.N.T. in cordeau detonant is known. The latter was determined (a) by the Reichsanstalt spark chronograph, (b) by the U.S.A. Ordnance Dept. spark chronograph, and (c) by the Reichsanstalt optical chronograph. Ten determinations by each method gave values (m./sec.) for (a) 4740—5230 (mean 4985), for (b) 4630—5130 (mean 4996), and for (c) 4970—5035 (mean 5000). The optical chronograph gives the best agreement between the determinations and the mean value has probably an error of ± 5 m./sec. The detonation velocities of cordeau detonant from various sources should not vary more than 2%. The detonation velocity of fulminate fuse was found to be 5251 m./sec. by the spark-chronograph method.

S. BINNING.

Vapour pressure of nitroglycerin and nitroglycol. (A) A. MARSHALL (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 422—423). (B) P. NAUM and K. F. MEYER (*Ibid.*, 423).—(A) The vapour pressure at 25° of nitroglycerin was found by Chiaraviglio and Corbino (cf. A., 1913, i, 1299) to be 0.00012 mm. Hg and not 10 mm., as recorded in Landolt-Börnstein's tables. Ignition and subsequent exposure *in vacuo* are insufficient to remove moisture from the guhr used by Naum. For accurate determinations of vapour pressure by the static method it is necessary to boil the substance in the measuring apparatus, which is impossible in the case of nitroglycerin. (B) It is maintained that the technique adopted was quite sufficient to remove all traces of moisture from the guhr mixture. S. BINNING.

Solvent recovery. BODEWIG.—See I.

PATENTS.

Liquid air or liquid oxygen explosives. LIQUID OXYGEN EXPLOSIVES, LTD. From PETITS-FILS DE FRANÇOIS DE WENDEL & CIE. (B.P. 321,886, 19.1.29).—A highly dried cellulosic substance, e.g., sawdust, is

used as an absorbent for liquid oxygen. Additional cellulosic substances, metallic powders, cooling salts, or absorbent substances saturated with water may be used, preferably in separate bags embedded in the centre of the cartridge. S. BINNING.

[Intermittent] detonating firework composition. W. F. GEHRIG (U.S.P. 1,736,743, 19.11.29. Appl., 5.3.28).—The composition, which is non-hygroscopic, comprises an alum, at least one sulphur-phosphorus compound, and at least one chlorate, together with a mixture of an alkaline-earth oxide and chloride as a binder. F. G. CLARKE.

XXIII.—SANITATION; WATER PURIFICATION.

Active carbon. EVANS and others.—See II.

PATENTS.

Elimination of mercaptans or related compounds from air. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,406, 31.5.28).—Air containing mercaptans etc. (0.1%) is rendered unobjectionable by passing over a contact mass which acts as an oxygen carrier, e.g., copper oxide in the form of wire heated at 650—750°.

C. JEPSON.

Purification of polluted water, sewage, etc. by a chemo-biological-mechanical process. E. F. HARRISON, ASSR. to CHEMO-MECHANICAL WATER IMPROVEMENT CO., INC. (U.S.P. 1,734,011, 29.10.29. Appl., 25.2.26).—The polluted water mixed with previously precipitated matter (enzyme and bacteriophage) is withdrawn from the lower portion of a tank and projected with considerable force, through air-bearing jets, into the upper surface of the remaining liquor.

C. JEPSON.

Boiler compound. W. L. KNIESEL (U.S.P. 1,735,402, 12.11.29. Appl., 14.11.27).—The compound comprises sodium silicate 1200, caustic soda 120, hemlock extract containing tannic acid 10, catechu gum 10, and sorghum molasses 12 pts.

H. ROYAL-DAWSON.

Production of soft water from sea water. M. M. G. MALCOR (F.P. 630,533, 8.3.27).—Sea water etc. is partly frozen and the ice is separated and melted for use as soft water, whilst the mother-liquor is evaporated for the recovery of the dissolved salts.

L. A. COLES.

Process for sterilising and oligo-dynamically activating substances [e.g., water]. G. A. KRAUSE (B.P. 293,385, 20.6.28. Ger., 5.7.27).—The sterilising power of an oligo-dynamically active metal, e.g., silver, copper, or their alloys, depends *inter alia* on the area of the surface exposed, and is therefore increased when the metal is in the form of thin plates (e.g., from silver nitrate or tartrate decomposed by heat) or of a deposit on an inert base, e.g., asbestos, to which it may be applied by heating a previously deposited metal gel (cf. B.P. 279,085; B., 1929, 540).

C. JEPSON.

Emulsions for boiler-scale removal (B.P. 321,337).—See I. Removal of silicic acid from liquids (B.P. 295,317).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JAN. 31 and FEB. 7, 1930.*

L—GENERAL; PLANT; MACHINERY.

Line-co-ordinate charts for representing chemical engineering data. E. A. RAVENSCROFT (Ind. Eng. Chem., 1929, 21, 1203—1210).—In the usual or Cartesian system of co-ordinates, a line represents an equation and a point a solution thereof. In the line-co-ordinate system, in which the x and y axes are parallel, a point represents a linear equation between x and y , and any straight line drawn through this point represents a solution. A series of equations between x and y each corresponding to a different value of a third variable z can be most simply represented by line co-ordinates in which they become points on a curve. Examples given are: a vapour-pressure chart in which the vapour-pressure curves of 50 substances are indicated by points, a latent heat-temperature chart, and a humidity chart for air-water. The last-named is much simpler than the Cartesian chart from which it is derived, and enables humidity to be determined directly from wet- and dry-bulb temperatures. In this and in all other cases the Cartesian curves must be modified if necessary so that they become straight lines before conversion into line co-ordinates.

C. IRWIN.

New method of heating chemical plant. A. ERNST (Chem. Fabr., 1929, 495—497).—Coils are wound on the external walls of the vessel to be heated, copper strips hammered in between the coils and the walls of the vessel, and the coils spot-welded to the walls.

S. I. LEVY.

Locating the obstruction in a clogged line of the [ammonia] compressor. J. RATHBUN (Ind. Eng. Chem., 1929, 21, 1257—1258).—A trap on the suction line to an ammonia compressor became partly blocked with congealed oil, probably owing to the evaporation of liquid ammonia remaining in the trap during pumping-down to effect a repair. The line on the other side of the obstruction remained under pressure, thus causing difficulty in locating the trouble. Such an obstruction can be identified by flooding the coils with liquid ammonia, when frost should disappear up to the point of blockage. Good oil and proper drainage are necessary.

C. IRWIN.

Correcting engine tests for humidity. D. B. BROOKS (Bur. Stand. J. Res., 1929, 3, 795—806).—Data obtained on a 6-cylinder automobile engine indicate a loss of engine power with increasing humidity proportional to the volumetric loss of oxygen content of the atmosphere. It is shown that power and fuel consumption may be corrected by subtracting observed water-vapour pressure from atmospheric pressure and using the result in place of barometric pressure in the usual correction formula. Simple nomograms are given for obtaining the humidity correction at different altitudes.

C. J. SMITHELLS.

Technique of practical lubrication. A. MOSSEY (Petroleum, 1929, 25, Motorenbetrieb, 2, No. 12, 3—10).—The disadvantages of different types of greases, vaseline, and mineral and fatty acids as lubricants are discussed. An ideal lubricant is defined as one which has a low friction coefficient and a horizontal viscosity curve. Mineral oils usually vary greatly with temperature, whilst fatty oils tend to decompose and deposit metallic salts, and compounding of these oils does not eradicate such disadvantages. Experiments show that for hot-running machines, better results are obtained with clear, low flash-point cylinder oils than with black, asphaltic, high flash-point cylinder oils. In automobile engines, water emulsions give rise to more trouble than is caused by a dilution of the lubricant with fuel, but so long as the consistency of the emulsion is no thicker than cream, it seldom causes danger. Non-emulsifiable oils are unsatisfactory since the settled water is drawn in by the pump and delivered in place of oil. The addition of a topped mineral oil instead of rape-seed oil for compounding is recommended.

W. S. E. CLARKE.

Filtration. I. G. BOZZA and I. SECCHI (Giorn. Chim. Ind. Appl., 1929, 11, 443—448, 487—492).—Experiments on the filtration of water, calcium chloride solutions, alcohol, benzene, petroleum, and petroleum-oil mixtures through beds of quartz and galena particles of different sizes show that: (1) Darcy's law is mostly rigorously obeyed, sensible deviations therefrom occurring only with very fine and non-homogeneous galena under rather high pressure-gradients. (2) With washed and very homogeneous quartz, the filtration constants found correspond satisfactorily with those given by King and Slichter (Principles and conditions of the movement of ground waters, 19th Ann. Rep. U.S. Geol. Survey, 1914), but do not agree with those calculated according either to Emersleben's theory (Physikal. Z., 1925, 26, 601) or to a simplified hypothesis in which the filter is likened to so many capillary tubes. (3) Through one and the same filter bed, a liquid of high surface tension passes more readily than one with a low surface tension. Thus water and aqueous solutions have filtration constants about 1.3 (1.5—1.8) times as great as those of alcohol, benzene, and oils with quartz (galena) beds. Considerable influence is hence exerted by the mutual characters of the solid and liquid, this being probably related to the inter-surface tensions between the two.

T. H. POPE.

Apparatus for measuring the permeability of surfaces of membrane hide, leather, wood, etc. by liquids and gases. M. BERGMANN (Chem. Fabr., 1929, 527—529).—In the apparatus for liquids the material to be tested is stretched transversely to a downward

* The remainder of this set of Abstracts will appear in next week's issue.

current of the liquid. A manometer and a regulator to compensate for the back-pressure are provided, and the volume of liquid passing is measured. The gas apparatus is kept under constant pressure by a given head of water in a tube with overflow, and the gas passing the membrane is measured by displacement.

C. IRWIN.

Simple volumenometer, and its employment for determining the porosity and the true and apparent sp. gravities of porous masses. R. WASMUHT (Chem. Fabr., 1929, 520—522).—A simple glass apparatus is described by means of which the volume of a solid may be read off directly. With porous substances, the fluid employed is water if the true volume is to be determined, and mercury if the total volume (solid + pores) is required.

S. I. LEVY.

Distillation in heated pipes. MAYER.—See II.

PATENTS.

Furnaces. CARBORUNDUM CO., LTD. From CARBORUNDUM CO. (B.P. 321,281, 25.9.28. Addn. to B.P. 257,261; B., 1927, 753).—The invention described in the prior patent is extended to combustion spaces (of boilers or metallurgical furnaces) operating with a flame temperature in excess of 1400°, the fuel ash being cooled at least 100° below the flame temperature, but not lower than 400°, and caused to congeal on the walls. If air-cooling alone is insufficient, water passages (carrying, e.g., the boiler-feed water) may be inserted in the air-jacket).

B. M. VENABLES.

Muffle furnace. F. C. GREENE and O. H. HERTEL (U.S.P. 1,730,570, 8.10.29. Appl., 13.6.25).—A furnace suitable for heating a vertical retort as described in U.S.P. 1,730,569 (B., 1930, 91) is divided vertically into a number of zones. On one side is a long, vertical, heat-supplying chamber, and on the other a similar exhaust chamber. The transference of heat can be individually regulated through each zone by means of dampers.

B. M. VENABLES.

Rotary kiln. A. J. BRIGGS, Assr. to INDUSTRIES OF AMERICA, INC. (U.S.P. 1,734,417, 5.11.29. Appl., 27.1.27).—A rotary kiln is provided with telescopic joints at one or more intermediate points, the outer ends being fixed axially.

B. M. VENABLES.

Heating and controlling chemical reactions at high temperatures. C. FIELD, Assr. to CHEM. MACHINERY CORP. (U.S.P. 1,734,329, 5.11.29. Appl., 15.7.22).—Mercury vapour is used both in a jacket and in direct contact with the liquid, which must not react with the mercury.

B. M. VENABLES.

Heat exchangers. R. C. CROSS and H. M. WARE (B.P. 321,833, 21.11.28).—A form of heat exchanger having three concentric tubes with thin walls and narrow annular spaces is described.

B. M. VENABLES.

Drying, waste-heat recovery, and cooling system. F. S. BOLTZ (U.S.P. 1,731,290, 15.10.29. Appl., 19.12.27).—In a laundry or similar plant the humid air from the drying cylinders is treated with water sprays, and the water caused to give up its heat to the cold water entering the plant.

B. M. VENABLES.

Desiccation apparatus. W. H. DICKERSON, Assr. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,734,289,

5.11.29. Appl., 9.3.23).—A spraying device for the liquid is situated at the top of a tower, and is supplied from a feed tank by a pump. Between the pump and sprayer a by-pass, with pressure regulator, is taken back to the feed tank; besides serving to keep the pressure constant, the by-pass permits rapid circulation in the feed tank and prevents sedimentation. The portion of the conduit beyond the by-pass (i.e., the net feed) is heated.

B. M. VENABLES.

Washing of materials such as ores, coals, etc. L. HOVOIS (B.P. 294,210, 12.7.28. Belg., 20.7.27).—The apparatus comprises a stratifying trough with upward-current separating columns below. When cleaning coal, the large lumps of coal and shale will stratify in a sloping feed-trough, with mixed smaller-size particles between and among the large lumps. The large shale passes through adjustable openings in the bottom of a horizontal continuation of the trough and meets upward currents of water; part of the latter flows through the ports into the trough, thereby largely preventing any coal from passing, but any coal that does get through is carried off through a horizontal passage below the trough by the other portion of the upward-current water. There are about as many columns as there are openings in the trough, and the horizontal product from one column may flow into the next.

B. M. VENABLES.

Impact pulveriser. W. R. WOOD, Assr. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,734,661, 5.11.29. Appl., 1.8.23).—A pair of oppositely revolving wheels have intercalating U-shaped rims, the flanges being provided with apertures through which the material passes by centrifugal force. On leaving any aperture the material is projected tangentially against the flange rotating in the opposite direction.

B. M. VENABLES.

Shear surface ring [hammer] for pulverising machines. R. J. WILLIAMS and C. A. WETTENGEL, Assrs. to SOUTHERN MANGANESE STEEL CO. (U.S.P. 1,733,637, 29.10.29. Appl., 11.12.26).—Hammers for use in disintegrators are formed as discs with wavy edges and having central holes larger than their supporting pins.

B. M. VENABLES.

Centrifugal mixing machines for sand etc. G. SAMM (B.P. 321,899, 19.3.29).—A plain disc and an annular disc are spaced apart by radial arms and provided with a shaft which may be either horizontal or vertical. The whole is rotated at centrifugal speed while the material to be mixed is fed through the centre of the annular disc.

B. M. VENABLES.

Separation of dry materials. BIRTLEY IRON CO., LTD., and C. W. H. HOLMES (B.P. 321,670, 7.5.28).—A shaking table with riffles and upward air currents is described. Among other points, all the material is delivered over an edge (or both edges in a twin table) which is (or are) parallel with the axis of reciprocation.

B. M. VENABLES.

Separator. [Stream-line slime settler.] M. SROCKHOFF (U.S.P. 1,732,386, 22.10.29. Appl., 6.8.27. Ger., 9.8.26).—A liquid containing solids in suspension is caused to flow continuously between a number of inclined plates, which are closely spaced so that the flow is stream-line. The slime collected on the plates slides off at the bottom into a worm conveyer or other means

of removing it as thick pulp. Baffles are provided so that there is no flow of liquid through the lower ends of the plates, and these portions, at least, of the plates are imperforate.

B. M. VENABLES.

Centrifugal machine. L. D. JONES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,733,266, 29.10.29. Appl., 27.10.27).—A centrifugal basket of the strainer type is driven by a sleeve shaft, within which is a solid shaft driving (at a different speed) a helical scraper to discharge the solids; the strain on the scraper is reduced by making the basket conical. The walls of the basket are formed from a number of rings which nearly touch each other and are supported by an external spider. The discharge of liquid (and fine solids) is through the continuous annular slits between the rings, and these are kept clear by thin plates extending into them and attached to the scraper.

B. M. VENABLES.

Tubular filter press. H. H. CANNON, Assr. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,734,325, 5.11.29. Appl., 28.5.28).—A number of tubular filter elements are non-leakably secured in tube plates and surrounded by a shell from which the filtrate is withdrawn. The domed ends of the shell form headers for the ends of the tubes, one header being divided into two compartments. The prefilter is supplied to one of the compartments and passes longitudinally through one group of tubes and back through the other, being exhausted as thick pulp through the other compartment of the divided header.

B. M. VENABLES.

Filter. E. J. SWEETLAND, Assr. to UNITED FILTERS CORP. (U.S.P. 1,734,652, 5.11.29. Appl., 17.5.24).—The filter has a fixed casing with short, hollow, rotating shafts through each end, and inside the casing the shafts expand into a rectangular framework which supports the filter leaves.

B. M. VENABLES.

Methods and means for evaporation or distillation. A. E. WHITE. From SWENSON EVAPORATOR Co. (B.P. 321,698, 18.5.28).—The evaporator is of the vertical tubular type with forced circulation. The upper outlet ends of the tubes extend some distance into the vapour chamber and are never allowed to become drowned, and the weak liquor must enter the lower ends of the tubes with a velocity of at least 5 ft./sec. Though, to avoid the vapour chamber, the inlet pipe for heating steam or vapour is below the ends of the tubes, a baffle is provided in the steam space to cause the actual first contact of the steam to be at the top of the bundle of tubes.

B. M. VENABLES.

Evaporation of liquids. A.-G. KUMMLER & MATTER (G.P. 455,101, 9.1.23).—Steam-heated tube evaporators are provided with a series of closed supply tanks the capacity of which is several times as great as that of the evaporator. Means are provided for preventing the mixing of the more and the less concentrated solutions in the tanks and for filtering the solution while passing through the tanks. The evaporator is so arranged that a separation of liquid and vapour takes place in such a manner that the vapour can be compressed and returned to the heating chamber of the apparatus while the solution is returned to the supply tanks.

A. R. POWELL.

Multi-stage evaporation process. ATLAS-WERKE A.-G., Assees. of S. VON LE JUGE (G.P. 455,070, 12.6.25).

—The liquid to be evaporated passes through a series of closed preheating vessels and is then sprayed successively into vertical cylindrical vessels through perforations in the bottom. The upper diameter of the cylinders is greater than the lower, and every cylinder is provided with apparatus for drying the vapours and passing the heated gases back through the earlier preheaters.

A. R. POWELL.

Continuously-operating saturation vessel. R. EHRLHARDT (Austr. P. 108,260, 11.3.26. Ger., 31.12.25).—An apparatus for saturating liquids with gases comprises a cylindrical vessel, the top diameter of which is greater than the bottom. The gas passes into the bottom of the vessel and is thoroughly mixed with the downwardly flowing liquid by means of a series of revolving paddles. The liquid is fed into the top of the vessel and passes out through an overflow tube in the bottom connected with a trough surrounding the upper rim and serving as a regulator for the height of the liquid in the vessel. Means are provided for observing and controlling the height of the froth which forms on the surface of the liquid and for taking continuous samples.

A. R. POWELL.

Air washer. H. KREISINGER, Assr. to INTERNATIONAL COMBUSTION ENG. CORP. (U.S.P. 1,734,677, 5.11.29. Appl., 4.12.25).—The gas is projected tangentially into the upper part of a cylindrical chamber, the flow being assisted and the gas humidified by jets of steam. After entering the chamber the whirling mixture of gas and steam is washed and condensed by means of water sprays.

B. M. VENABLES.

Centrifugal liquid purifier. J. W. ADAMS (U.S.P. 1,738,553, 10.12.29. Appl., 22.6.28. U.K., 27.7.27).—See B.P. 296,178; B., 1928, 773.

Method and apparatus for removing vapours. H. W. COWAN (U.S.P. 1,738,641, 10.12.29. Appl., 4.4.27. Can., 29.10.26).—See B.P. 279,816; B., 1929, 267.

Furnace walls [with fluid-cooled panels]. AMER. ENG. Co. (B.P. 308,342, 26.6.28. U.S., 22.3.28).

[Absorption] refrigerating systems. C. A. MASTERMAN, and GAS LIGHT & COKE Co. (B.P. 322,226, 1.9.28 and 25.6.29).

Continuously working absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 298,603, 20.9.28. Ger., 12.10.27).

Refrigeration apparatus. A. E. WHITE. From WALTHAM SYSTEM, INC. (B.P. 322,324, 21.11.28).

Treating waste liquors (B.P. 321,413).—See V.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coal-testing problems. N. PARTINGTON (Gas J., 1929, 188, 720–722).—The necessity for correct sampling is emphasised and a method suitable for a gas works which receives most of its coal by rail is described. A high moisture content in the coal increases the amount of coke consumed in the producers, thereby decreasing the amount of coke available for sale, increases the carbon dioxide content of the gas, lowers the liquor strength, and tends to decrease the working life of the retort and coal-handling plants. The moisture content of many washed coals varies more than the ash content,

and a sudden fall in retort-house efficiency can often be traced to increased moisture in the coal; daily moisture tests are recommended, also the sheeting of trucks containing small coal and which are likely to be several days *en route*. The ash content of washed coals is more consistent than that of dry cleaned coals; as coke is likely to be increasingly used for domestic purposes it is suggested that 5% of ash in the coal should be regarded as the limit. The Lessing test is used to determine the volatile matter and the swelling properties of coals and coal blends. A modified shatter test is described for estimating the liability of a coke to form breeze. Laboratory carbonisation tests on coal, as carried out by the Lessing test, show that the yields of tar and gas thereby obtained compare favourably with those of a works' experimental plant, carbonising 24 cwt. of coal per diem.

C. B. MARSON.

Examination of coal and coke by X-rays. C. N. KEMP (J. Roy. Soc. Arts, 1929, 78, 114—136).—After a brief account of previous investigations of the application of X-rays to the examination of coal, the author's own work in this field is summarised (cf. B., 1924, 931; 1925, 656; 1926, 937; 1927, 641; 1928, 916).

A. B. MANNING.

Low-temperature carbonisation: continuation of tests on Canadian bituminous coals. R. A. STRONG (Canada Dept. Mines Fuel Invest., 1927, No. 696, 32—45).—Experiments are described in which four coals have been examined in a laboratory low-temperature carbonisation assay apparatus at 600° (cf. B., 1929, 41). The cokes obtained varied considerably in appearance, two being very much swollen and fairly porous and the other two dense and hard. All the cokes were high in ash (16.2—19.8%), and it would be necessary to reduce the ash content of the coals by washing before they could be considered suitable for the production of domestic coke; the volatile matter varied from 6.2 to 7.4%; the calorific values of all the cokes were less than those of the corresponding coals and, on account of the high ash, they are considerably lower than those of average gas and by-product cokes usually sold for domestic purposes. Tar yields of 8.1, 14.1, 18.5, and 22.1 gals. per ton of coal carbonised, respectively, were obtained, and these, with one exception, are low; the light oils, which were stripped from the gas with activated carbon, gave yields of 1.3—2.5 gals./ton. The gas yield varied from 3450 to 4250 cub. ft./ton, with calorific values of 570—650 B.Th.U. per cub. ft.; the ammonium sulphate equivalent varied from 4.3 to 19.1 lb./ton of coal. The results of a detailed examination of the tars are also given. It is concluded that owing to the low tar yields of these and the 10 other coals previously examined (*loc. cit.*), the field for low-temperature carbonisation, from the viewpoint of oil yield, is decidedly limited in Canada.

C. B. MARSON.

Gasification of lignite, peat, and wood. E. MARCORRE (Chim. et Ind., 1929, 22, 877—889).—An Austrian lignite (moisture 50%) could not compete with coal as a fuel in its raw state, in spite of low first cost, nor was briquetting profitable. It might be possible to burn it at a power station placed close to the working, but gasification and long-distance transmission of gas is con-

sidered preferable. If electricity is also to be produced it should be generated by gas engines, not by turbines. The residue from high-temperature carbonisation is to be used for making bricks. The treatment of lignite in gas producers, as practised in Germany, is discussed, and attention is called to the device for distilling intractable producer-gas tar by mixing it to a paste with lignite dust. The gasification of peat or wood necessarily produces a low-grade gas on account of the high oxygen content of these materials. No advantage is obtained by passing the gas through a heated fuel bed for the reduction of carbon dioxide, as other reactions occur as well, and removal of carbon dioxide with milk of lime is expensive. The coke produced from peat or wood requires an excessive amount of water for quenching, and dry-cooling is desirable. The author concludes that the choice between carbonisation in various forms of retort and gasification as a means of utilising lignite depends on the character and location of the deposit.

C. IRWIN.

Benzene-pressure extraction of coal. J. D. DAVIS and D. A. REYNOLDS (Ind. Eng. Chem., 1929, 21, 1295—1298).—A series of six coals each representative of a distinct type as regards industrial carbonisation were subjected to the Fischer benzene-pressure-extraction process. With continued extraction both the solidity of the extract and the ratio of solid bitumen to oily bitumen increase. A bitumen was observed that is soluble in benzol under the conditions of the extraction, but which precipitates as a brown powder from the solution on cooling. Removal of soluble substance from a coal with either pyridine or benzene under pressure may cause its swelling properties either to increase or decrease, depending on the character of the coal. The authors' previous conclusions (B., 1926, 859) that solid bitumen causes swelling and is more strongly coking than oily bitumen are confirmed.

H. S. GARLICK.

Carbon black. II. Rôle of oxygen. C. R. JOHNSON (Ind. Eng. Chem., 1929, 21, 1288—1290).—The oxygen adsorbed on the surface of carbon black (as found by ultimate analysis or from the volatile matter content, to which it bears a fixed ratio) was not reduced by storage under nitrogen, but was somewhat increased by exposure to pure oxygen. It was increased by heating in air to 500° and decreased by heating with zinc dust, zinc oxide being formed. There is no definite connexion between iodine adsorption and oxygen content, but the adsorption of organic accelerators varies directly with the latter. This indicates the reason why carbon black of high volatile content gives poor physical properties when vulcanised with rubber.

C. IRWIN.

Modern coke ovens as gas producers in large gas works; their sphere of application and economic principles. R. MEZGER (Gas- u. Wasserfach, 1929, 72, 1189—1197, 1222—1227).—Various types of coke-oven plant are briefly described with the aid of diagrams. The problem of uniform heating of the oven walls receives attention, coke ovens gaining in this respect by comparison with other forms of gas retorts. A discussion of the economic aspects of coke-oven operation, with the aid of tables and curves showing the effects of various factors, including capital and working costs, leads to the conclusion that gas supply from

regional grouped gas undertakings is more economical than would be the supply of gas over the whole of Germany from centralised plant situated in the Ruhr area.

D. G. MURDOCH.

Review of the Koppers "C.A.S." process as applied to British conditions. E. W. SMITH (Gas J., 1929, 188, 767—768).—The "C.A.S." process, which has been devised for the removal of ammonia, hydrogen sulphide, and cyanogen compounds from coke-oven gas with the recovery of ammonium sulphate and sulphur, without introducing sulphuric acid from outside, is reviewed. It is concluded that (i) the "C.A.S." process is capable of being worked technically satisfactorily; (ii) the process will not enhance the value of ammonia in the carbonising industries; (iii) the process is not satisfactory for the removal of hydrogen sulphide to comply with British standards, which are markedly severe; (iv) even if the process as a whole can show a financial advantage on paper, the chemical complication and specialised technical supervision render the process unsuitable for British practice; (v) discrepancies in the claims made for this process may be attributed to differences between British and German conditions and practice.

C. B. MARSON.

Recovery of ammonia [in gas manufacture] by the semi-direct process. G. E. FOXWELL (Gas J., 1929, 188, 768—770).—Parker's conclusions (cf. B., 1930, 43) as to the flexibility, the difficulty of producing high-quality sulphate, and the economic costs of the direct and semi-direct processes, together with the necessity for modification of the last-named at a gas works, are examined and criticised. It is contended that (i) whilst the direct process is not so flexible as the indirect process, in that it can only produce ammonium sulphate, this is only partly true of the semi-direct process; (ii) there is no difficulty in making sulphate of the highest quality by the semi-direct process; (iii) although it is not possible to make sulphate at a profit by the indirect process as operated at a gas works, it is possible to make it at a profit by the semi-direct process; (iv) the semi-direct process would not require modification for installation at a gas works, provided that the gas works were of a reasonable size.

C. B. MARSON.

Lubrication of gas mains by means of oil fog. O. H. BLACKWOOD and P. G. EXLINE (Ind. Eng. Chem., 1929, 21, 1258—1260).—A persistent oil fog suitable for the internal lubrication of gas mains is composed of particles 1 micron or less in diam. and falling at speeds of about 12.2 cm./min. Such a fog, though difficult to control, may be produced cheaply and in large quantities by condensation methods, e.g., by injecting a stream of hot, saturated gas into a current of colder gas. Atomisation is simpler and more easily controlled, but is subject to limitations, since about 99% of the oil blown out of the atomiser is too coarse for use, and only a relatively small quantity of fog is produced by a single spraying unit. Oil fog may be detected and its density measured by aspirating samples through weighed filter papers, or the oil may be dyed and the tint produced on a filter paper in a tar camera compared with laboratory standards. The persistence of a fog is

determined by measuring its density at various periods subsequent to its formation.

H. S. GARLICK.

Primary tars obtained with the "Italian system" furnace. D. MENECHINI (Giorn. Chim. Ind. Appl., 1929, 11, 433—438; cf. B., 1929, 1037).—When distilled in a current of superheated steam in a special apparatus, the tar obtained in this furnace from Thornley gas coal (and Valdarno lignite) gives the following results (in kg.): Tar obtained per metric ton of the dry coal, 125 (43); oils obtained in a first distillation to remove the pitch, 52.7 (22.8); phenolic (acid) compounds, 7.9 (7.4); redistilled neutral oils, 26.3 (12.8); total residue, comprising hard pitch and distillation residue, 85 (19.6); light neutral fractions, b.p. below 170°, 2.2 (0.2); medium fractions, b.p. 170—230°, 5.8 (2.4); heavy fractions, b.p. 230—270°, 6.3 (3.7); oils, b.p. above 270°, 12 (12.8). Normal tar from the same Thornley gas coal (50 kg. per metric ton) gives: oils from first distillation to separate the pitch, 27.5; these give: redistilled neutral oils 21, containing naphthalene and anthracene 6.3 and phenolic compounds 1.2. The pitchy residues of the primary tars exhibit excellent agglomerating properties, that from the lignite especially having marked plasticity. The fractions with high contents of acid compounds should be of value for impregnating timber, but there seems no convenient means of utilising directly the neutral oils. The heavy oils are unfit for use as lubricants, owing to their ready alterability, this being due to the presence of a large proportion of unsaturated compounds.

T. H. POPE.

Chemistry and physics of road tar. H. MALLISON (Gas- u. Wasserfach, 1929, 72, 1215—1222).—Methods for determining the physical properties and chief chemical constituents of the tar are summarised, and their significance is discussed. It is anticipated that the British Road Tar Association's tar viscosimeter will be adopted in Germany as the official apparatus for the determination of viscosity. The addition of more than 20% of petroleum bitumen to tar is held to produce considerable flocculation of the free carbon and to yield a mixture lacking in homogeneity; addition of Trinidad asphalt produces no coagulation of free carbon. Tar emulsions are briefly considered, and simple tests of their suitability for road purposes are described. One such test consists in pouring 10 g. of emulsion evenly over the surface of a glass plate (10 × 10 cm.) edged with pitch, and then dusting with 25 g. of specially selected fine sand. The plate is allowed to dry in the air until a definite proportion (about 80%) of the known amount of water originally present has evaporated and is then immersed in an inclined position in water for 12 hrs. Inspection of the plate after this period yields a valuable indication of the breaking properties of the emulsion and of the tendency of the separated tar to become re-emulsified.

D. G. MURDOCH.

Treatment of pyroligneous acid and extraction of acetic acid and alcohol. C. MARILLER (Chim. et Ind., 1929, 22, 868—876).—It is now usual in the wood-distillation industry to remove tar separately by partial cooling. The vapours are then either condensed and further worked (indirect process), or treated directly. The Brégeat cresol process does not appear

to have been anywhere adopted. The indirect process necessarily involves a greater steam consumption, but this has been reduced in America by multiple-effect evaporation. One possible means of avoiding the production of calcium acetate may be the catalytic oxidation of acetic acid vapour to acetone by barium or magnesium acetate. The Suida process effects the direct production of acetic acid by the use of a wood-tar distillate of b.p. above 210° . The pyroigneous acid is first freed from alcohol in the usual way, and is then distilled in a tubular heater connected to an absorption column. The enriched solvent from the latter is freed from water in an auxiliary column, and passes to the stripping column, which is operated under a vacuum of 720 mm. at $155\text{--}160^{\circ}$. The product of this contains 85—95% of acid, and can be rectified if desired. The apparatus has not yet been adapted to the direct process. The steam consumption is estimated at 13—15 kg./kg. of 100% acid and the acid recovery at 95%. The process is being widely adopted both at wood-distillation plants and acetate silk works.

C. IRWIN.

Pyrolysis of the paraffins. E. N. HAGUE and R. V. WHEELER (Fuel, 1929, 8, 512—524, 560—587).—Previous work is reviewed. Experiments have been carried out to study (i) the effect of heating methane, ethane, propane, and butane in a silica or hard-grass bulb; (ii) the effect of allowing these gases to flow through a heated tube in a circulation apparatus; and (iii) the effect of passing the same gases, and also pentane and hexane, in the form of vapour, as a stream through a heated tube. From the first series of experiments the decomposition points of the gases were found to lie within the ranges: methane $650\text{--}685^{\circ}$, ethane $450\text{--}485^{\circ}$, propane $425\text{--}460^{\circ}$, and butane $400\text{--}435^{\circ}$. At temperatures below 500° the decomposition of the gases, as measured by the rate of increase of pressure, appeared to be unimolecular. Above 500° there was a rapid initial change of pressure, followed by a period of constant rate of increase. With propane and butane carbon deposition and tar fog were observed at $575\text{--}600^{\circ}$. In the second series of experiments similar pressure-time curves were obtained to those in the first. With propane and butane liquid decomposition products were observed at 700° and appeared in quantity at 800° . From an examination of the products obtained in the third series of experiments it is concluded that the primary decompositions, occurring below $700\text{--}750^{\circ}$, involve a rupture of the chain at any position with the production of an olefine and the complementary paraffin or, at the limit, hydrogen. Following these primary decompositions many complicated reactions occur during "pyrosynthesis" at the same or higher temperatures. With ethane at temperatures above 700° , the hydrogenation of fugitive "residues," CH_2 , appears to take place (cf. Bone and Coward, B., 1908, 886). At comparatively low temperatures, subsequent to the formation of ethylene, butadiene is produced from each of the paraffins (cf. Norton and Andrews, A., 1886, 604). Above $700\text{--}750^{\circ}$ benzene and other aromatic hydrocarbons are produced from each gas, methane included, probably through the intermediate formation of ethylene and butadiene. The first step in the forma-

tion of benzene from methane may be represented by $\text{CH}_3\cdot\text{H} \rightarrow \text{CH}_2 + \text{H}\cdot\text{H}$, two of the CH_2 residues then combining to form ethylene. The optimum yield of benzene from methane is obtained at $1000\text{--}1100^{\circ}$.

A. B. MANNING.

Principles of distillation in heated pipes in the petroleum industry. A. W. J. MAYER (Petroleum, 1929, 25, 1673—1679).—Mathematical. Previously published work of a large number of observers on the factors controlling stream-line and turbulent flow of liquids in pipes and on the effect on the flow of bends in the pipes is discussed, with special reference to its application to pipe stills and the pumping of hot oil.

W. S. E. CLARKE.

Lubrication. MOSSER.—See I. **Fertilisers.** MATIGNON.—See XVI. **Fermentation of cellulose.** BORUFF.—See XVIII.

PATENTS.

Production of a fuel from pulverised coal. R. HADDAN. From TRENT PROCESS CORP. (B.P. 321,695, 14.5.28).—A pulverised fuel suitable for use in internal-combustion engines is formed by blending two or more pulverised coals, which may differ in degree of fineness (from 300-mesh upwards), in density, and in volatile matter content. The proportions and characters of the respective fractions are so chosen that the composite fuel gives the optimum engine performance.

A. B. MANNING.

Production of improved solid fuel from the distillation of carbonaceous materials. L. C. KARRICK (B.P. 321,969, 10.8.28. Addn. to B.P. 261,362; B., 1928, 218).—In modification of the process of the prior patent, the activated coke residue after dry-quenching with steam is immersed in a vapour or solution so that it absorbs ingredients, e.g., nitrates or chlorates, copper chloride, oil of pine or cedar, etc., which impart to it desirable kindling, light-giving, and/or odour-producing properties.

A. B. MANNING.

Fuels [for internal-combustion engines etc.]. D. GARDNER (B.P. 321,701, 17.7.28).—Carbon produced as described in B.P. 292,798 (B., 1928, 631) is dispersed in 2—6 pts. of another combustible material. The latter may be a gas, e.g., methane or water-gas; a liquid, e.g., an aromatic, hydroaromatic, or terpenic hydrocarbon; or a substance, e.g., paraffin wax or naphthalene, solid at the ordinary temperature.

A. B. MANNING.

Manufacture of carbon black. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,402, 3.8.28).—Carbon monoxide, preferably under a pressure of 100—200 atm., is passed over a catalyst which is solid under the working conditions and which comprises a metal or compound of the iron group to which is added a suitable activating material. The latter may consist of an oxide of manganese, vanadium, chromium, or uranium, etc., an alkali hydroxide or carbonate etc., or a molybdate or tungstate. The temperature of the reaction is preferably maintained between 300° and 400° .

A. B. MANNING.

Coke ovens. DR. C. OTTO & Co. G.M.B.H. (B.P. 294,992, 3.8.28. Ger., 3.8.27).—Each of the vertical heating flues of a twin-flue coke oven is provided with

two or more inlets for rich fuel gas at different levels. All the vertical pipes leading to the inlets at the same level in alternate flues communicate with a separate supply conduit which can be connected by means of a change-over valve to the gas supply as required. The gas supply can thus be immediately changed over from the high- to the low-level inlets, and, conversely, by a single adjustment of the valve, independently of the reversal of the gas supply from one set of flues to the other. If desired, the flues of one set only may be supplied with two or more gas inlets, each flue of the other set having a single inlet at a level intermediate between those of the first set. A. B. MANNING.

Obtaining more economical running of coke-oven plants etc. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES. From INTERNAT. BERGIN COMP. VOOR OLIE-EN KOLEN-CHEMIE (F.P. 630,329, 5.3.27. Ger., 19.11.26).—The carbonisation plant is run in co-operation with a hydrogenation plant. The distillation gases poor in hydrogen are mixed with the rich gases from the hydrogenation process and utilised in any suitable manner. The gases of high hydrogen content evolved during the later stages of the same distillation process are used for the hydrogenation of the tar in the berginising plant.

A. B. MANNING.

Retort for the low-temperature carbonisation of coal. G. BEGAS (G.P. 454,691, 27.1.25).—The walls of the retort are formed by two vertical, concentric cylinders; in the annular space between them is a helical grate, some of the bars of which are attached to the inner cylinder and the others to the outer, so that by giving a to-and-fro motion to the cylinders the coal in the retort is moved along the grate. The throughput of the retort is large, but regular and controllable; no dust is formed during the process. A. B. MANNING.

Apparatus for extracting values from coal and like materials. F. C. GREENE and I. F. LAUCKS, ASSRS. to OLD BEN COAL CORP. (U.S.P. 1,730,569, 8.10.29. Appl., 5.7.19).—An apparatus for the low-temperature distillation of coal comprises a number of units each consisting of a vertical, metallic, tubular retort within a vertical tubular furnace, and a spiral is provided on the outside of the retort to cause the heating gases to rotate while passing upwards through the annular space. The coal space within the retort is also annular owing to the presence of a hollow, rotating shaft which carries a worm to impel or regulate the travel of the coal (or coke) downwards. Vapour mains are provided both at the top and bottom of the retort beyond the heating zone and are maintained under suction. The tubular, hollow shaft is perforated within the retort and extended upwards beyond it; the top is closed by a valve which is held closed by the normal suction in the retort, but will open in the event of pressure developing in the retort. B. M. VENABLES.

Vertical retorts. C. W. TOZER (B.P. 321,708, 16.8.28).—Separate charging and discharging means are provided for each chamber of a multiple-chambered retort. The former comprises a vertically movable ram, and the latter a curved or sloping portion of the chamber which, while preventing discharge by gravity, permits free discharge of the carbonised material in quantities deter-

mined by the displacement of the charge caused by the movement of the ram. A. B. MANNING.

Apparatus for carbonising coal and other fuel. A. CHABOT (B.P. 321,827, 14.11.28).—The fuel is carbonised in moulds forming part of an endless conveyer which carries the fuel through a horizontal, externally heated, tunnel retort. The fuel is fed from a hopper into the moulds and is compressed therein by means of a reciprocating piston. The retort is divided into two longitudinal compartments by a horizontal, perforated plate, or by a number of juxtaposed, transverse rollers. The conveyer fits in the lower compartment; the upper is provided with outlet pipes and forms a collecting chamber for the distillates. The upper compartment may be subdivided by baffles into chambers from which tars and gases of various qualities may be separately collected. The retort may be heated by the combustion gases from a furnace, the gases travelling in the direction opposed to the motion of the fuel, so as to heat consecutively the portion of the tube forming the carbonisation chamber, the portion forming the distillation chamber, and the portion forming the drying chamber.

A. B. MANNING.

Treatment of peat. E. VON SPRINGBORN (B.P. 321,411, 4.8.28).—The peat is cut into thin layers transversely to the general run of the fibre, and then pressed through an apertured plate on to a movable carrier. It is dried thereon by natural or artificial means and is then carbonised in the presence of a minimum quantity of steam and evolved gases, which are withdrawn from the retort as they are formed.

A. B. MANNING.

Apparatus for production of combustible gases from granular and dust fuels. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 321,422, 2.6. and 17.8.28).—Granular fuel containing fuel dust is introduced near the bottom of a generator and part of the gasifying agent is admitted in an upward direction in order to support the fuel and keep it in suspension in the gasification chamber, into which the other part of the gasifying agent is admitted at different levels. The hot gaseous mixture which still contains fuel dust is then conducted through a large secondary gasification chamber. Producer-gas and water-gas may be produced alternately if a suitable heat accumulator, consisting, e.g., of a chequer work of fireclay bricks, is provided in the generator. A. B. MANNING.

Gas-generating apparatus. E. L. FISCHER (U.S.P. 1,736,586, 19.11.29. Appl., 2.2.26).—A setting of retorts is placed above a water-gas generator, the passage between the two forming a combustion zone. Water-gas is withdrawn from, and air and steam are admitted to, both the top and bottom of the generator.

B. M. VENABLES.

Water-gas generators. J. PINTSCH A.-G. (B.P. 295,717, 13.8.28. Ger., 19.8.27).—An apparatus for the continuous generation of water-gas from powdered fuel has a reaction chamber in the form of a vertical cylindrical pipe, which is surrounded by the heating apparatus wherein a current of steam and water-gas is raised to the temperature necessary to react with the fuel. The powdered fuel is introduced continuously, preferably

at the top of the reaction chamber, the bottom of which is widened in order to reduce the velocity of the gases and allow the ash to settle out. The annular heating apparatus is divided into two chambers which are alternately heated and used for heating the current of gas and steam.

A. B. MANNING.

Continuous generation of water-gas [from powdered fuel]. GAS- U. TEER GES.M.B.H. (B.P. 296,064, 23.8.28. Ger., 24.8.27).—The apparatus consists of a central chamber situated between two regenerator chambers. A part of the water-gas produced together with the powdered coal or coke and steam is passed to one regenerator wherein the mixture is raised to the temperature necessary for the production of gas, while the other regenerator is being heated by the combustion therein of another part of the gas produced. The central chamber is designed to act as an ash-separating means. If desired, each regenerator may be divided by means of a partition, whereby the currents of gas and steam are raised separately to the gas-producing temperature.

A. B. MANNING.

Continuous production of water-gas and hydrogen from gaseous hydrocarbons. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES. FROM DEUTS. BERGIN-A.-G. F. KOHLE- U. ERDÖLCHEMIE (F.P. 630,327, 5.3.27. Ger., 22.11.26).—The gaseous hydrocarbons are incompletely burnt with air or oxygen, and the resulting gases are freed from water vapour and carbon dioxide.

A. B. MANNING.

Apparatus for the purification of gas. W. WILSON (B.P. 321,536, 26.10.28).—The gas is passed transversely through a travelling column of oxide moving down a louvred structure. Two such structures are enclosed within corresponding vertical chambers connected by upper and lower gas conduits; one of the latter is provided with a change-over valve having a gas inlet and a gas outlet. Baffles are provided within each chamber, whereby the gas is constrained to pass and repass through the oxide. The oxide-feeding and -discharging connexions are preferably in the form of gas locks.

A. B. MANNING.

Washing of gas. R. NORGATE (B.P. 321,252, 20.8.28).—The gas is passed through a series of chambers each containing an ejector which produces a fine spray of the washing liquid and is operated by part of the gas to be treated, under a pressure of about 5 atm. The washing liquid is maintained at a constant level in each chamber. Each ejector has a submerged liquid inlet, an inlet for the pressure gas, and a second gas inlet opening in the chamber above the level of the liquid therein.

A. B. MANNING.

Purification of industrial gases with the recovery of by-products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,982, 23.8.28).—Industrial gases are freed from ammonia, hydrogen sulphide, and hydrocyanic acid by washing with a solution containing ammonium polythionate, ammonium thiosulphate, and sufficient ammonia to absorb completely the hydrogen sulphide and hydrocyanic acid, the excess of ammonia being subsequently converted into sulphate by the addition of the requisite amount of sulphur dioxide. If desired, the process may be carried out in two stages,

as described in B.P. 292,669 (B., 1928, 632), by removing an additional quantity of hydrogen sulphide in the first stage so that the ammonia available in the second stage completely suffices for combining with the remaining hydrogen sulphide and hydrocyanic acid. After boiling the spent liquor, the ammonium sulphate and thiocyanate are separated by fractional evaporation.

A. B. MANNING.

Removal of ammonia from coal-distillation gases. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 322,049, 13.10.28).—The condensed gas liquor, heated to 90–100° if necessary, is brought into intimate contact with a portion of the hot gases, and is thereby deprived of its free ammonia. The de-ammoniated liquor is then cooled and used to wash the cooled gas in the usual scrubbers. The final liquor, which is of substantially higher concentration than usual, is then distilled or otherwise treated in known manner.

A. B. MANNING.

Extracting benzene hydrocarbons from distillation gases. C. STILL (B.P. 300,964, 15.11.28. Ger., 21.11.27. Addn. to B.P. 293,702; B., 1929, 916).—To recover the benzene from the wash oil used for the extraction, the latter is preheated to 130° by heat exchange with hot residual oil and by means of a steam coil. It is then discharged into a column still where, by means of open steam, the benzene and some naphthalene are removed. The residual oil, after cooling, is returned to the top of the scrubbing tower. The vapours from the still, after condensation and separation of water, are fed into a fractionating column heated by closed steam coils and the benzene is distilled from the naphthalene residue. The residue, not sufficiently cooled to cause separation of naphthalene, is returned to the scrubbing tower at a point below the exit of the scrubbed gas. The quantity being small, its elevated temperature is of little importance, and difficulty owing to its possible benzene content is overcome by not feeding it into the top of the scrubbing tower.

T. A. SMITH.

Porous masses for the storage of explosive gases or fuels. J. HAUSEN (F.P. 627,753, 18.1.27. Ger., 20.1.26).—Before introducing the porous material, powdered pumice, or carbon, etc. into the container it is impregnated with inorganic or organic substances, e.g., ammonium sulphate, oxalate, or carbonate, which are vaporised or gasified by partial decomposition of the explosive gas, e.g., acetylene.

A. B. MANNING.

Production of aqueous dispersions of pitch, bitumens, resins, etc. F. B. DEHN. From FLINTKOTE Co. (B.P. 321,721, 21.5.28).—An insoluble mineral powder, e.g., clay or bentonite, is used as a dispersing agent, and the p_H of the dispersion is adjusted to give the optimum emulsification. The p_H may be controlled by suitable choice of the mineral powder used as dispersing agent, by the addition of acidic or basic materials to the pitch, bitumen, etc., or by the addition of an acid, an alkali, or a buffer salt to the emulsion.

A. B. MANNING.

Refining of crude mineral oils. W. McI. CRANSTON (B.P. 322,167, 24.8.28).—Crude oil, mixed with an equal weight of fine bituminous material containing not

less than 20% of volatile matter, is distilled at ordinary pressure up to 180° in a still fitted with agitators. The pressure is then raised to 200 lb./in.², and the distillation carried up to 320° with the admission of steam for hydrogenation purposes. Cracking takes place, and the residual pitch and coke are obtained as a fine powder or in coherent form. The residue is a useful fuel which can be stored without danger of spontaneous combustion. Access of moisture should be prevented.

T. A. SMITH.

Manufacture of lighter-coloured products from earth-oil residues, asphalt, and/or similar substances. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 313,433, 30.8.28. Holl., 11.6.28).—Light-coloured asphalts are prepared from ordinary asphalts or cracking-plant residues by the removal of asphaltenes. A solution of the asphalt is treated with sulphuric acid, or with decolorising earths, or the asphaltenes are precipitated by the addition of light hydrocarbons poor in aromatic constituents, or a combination of these treatments may be used. Molten Java asphalt, to which 200% of benzine has been added, is stirred with 2% of sulphuric acid. After settling and tar removal the solution is treated with 3% of bleaching earth at 60° and filter-pressed. The asphalt is recovered from the solution by steam-distillation until the required penetration is obtained. The light-coloured asphalt is suitable for the preparation of emulsions, lacquers, varnishes, asphalt paints, etc.

T. A. SMITH.

Gasifying of heavy oils. C. CHILOWSKY (B.P. 296,785, 4.9.28. Fr., 8.9.27).—The gasification of heavy oils, particularly for use in internal-combustion engines, is brought about by partial combustion of the oil in the presence of steam. The air and steam are heated to 500–900° before coming in contact with the oil spray. The apparatus consists of a perforated mass of catalytic material in which the final gasification is brought about at temperatures up to 1100°. The hot gases produced pass through an annular space surrounding the catalyst, the outer wall of this space being a thin sheet of non-corrosive metal through which heat is transmitted for preheating the air and steam used in the partial combustion. A variety of designs of apparatus to give good heat transfer between the hot gas and the air are given. The apparatus is surrounded by heat-insulating material.

T. A. SMITH.

Production of light oils by cracking of low-temperature tar. ZECHÉ M. STINNES (G.P. 454,764, 12.8.21).—The gases and tar vapours leaving the retort at temperatures above the dew point of the tar are mixed with superheated steam and passed over heated porous material, e.g., low-temperature coke. In addition to light oils, ethylene, propylene, butylene, and butadiene are produced.

A. B. MANNING.

Treatment [cracking] of heavier hydrocarbons. C. A. JENSEN. From JENKINS PETROLEUM PROCESS Co. (B.P. 321,929, 20.8.28).—A horizontal cylinder, situated above an inclined bank of heated tubes, is connected in closed circuit with the tubes. Circulation along the cylinder and through the tubes is brought about by means of a suitable piston. Contact material is added to the oil to be cracked, and assists in keeping the tubes

free of deposited carbon. Cracked vapours are taken from the top of one end of the cylinder to a dephlegmating column and are scrubbed by the addition of the cracking stock to which contact material has been added. The cracking stock is thus preheated and the insufficiently cracked fractions are returned to the circuit. The pipe carrying the cracking stock from the dephlegmating column to the cylinder is divided and carried along the bottom of the cylinder to the farther end, where it delivers in an upward direction and along the flow of the circulating oil. The apparatus works under any desired pressure. The addition of cracking stock and the withdrawal of distillate and of a proportion of the uncracked residual oil are continuous.

T. A. SMITH.

Treatment of hydrocarbon gas or the like. EMPIRE GAS & FUEL Co., ASSCES. of J. C. WALKER (B.P. 290,613, 5.5.28. U.S., 17.5.27).—Hydrocarbons, particularly gaseous paraffin hydrocarbons, are converted into oxygenated compounds, such as alcohols and aldehydes, by partial oxidation at an elevated temperature, e.g., 420–480°, and, if desired, under pressure, preferably above 200 lb./in.², and in the presence of a suitable catalyst, the oxygen-supplying material, e.g., air, being added in an amount equivalent to less than 0.1 vol. of free oxygen to 1 vol. of hydrocarbon. The hydrocarbon may be preheated to within 100° of the reaction temperature and the amount of oxygen added so regulated that the heat of reaction just brings the mixture to the required reaction temperature. The products may be cooled by heat interchange with the mixture entering the reaction zone; after condensation of the liquid oxidation products the residual gas may be subjected to a further partial oxidation.

A. B. MANNING.

Manufacture of hydroaromatic hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,445, 3.9.28).—The catalysts which may be used in the removal of sulphur compounds, such as thiophen, from crude benzene by hydrogenation under pressure, are capable, at slightly higher temperatures, of producing hydrogenation of the aromatic hydrocarbons. With these catalysts it is unnecessary to use pure hydrocarbons. The catalysts consist of compounds of elements of groups V and VII. Crude benzene and hydrogen passed over molybdenum trioxide at 460° and 200 atm. is converted quantitatively into cyclohexane. The catalyst is more efficacious when mixed with zinc oxide, manganic oxide, or metal powder. Toluene is hydrogenated at 450° in the presence of cobalt molybdate at 120 atm. A 1:1 mixture of benzene and naphthalene is converted at 200 atm. and 450° in the presence of a molybdenum-chromium catalyst into a mixture containing 50% of cyclohexane, 10% of decahydronaphthalene, and 40% of tetrahydronaphthalene. In some cases temperatures up to 800° may be necessary, and the operation may also be carried out in the liquid phase.

T. A. SMITH.

Manufacture of fuel containing alcohol. W. STEIGER (B.P. 309,155, 28.6.28. Ger., 7.4.28).—Alcohol containing up to 20% of water may be used as an engine fuel when added to turpentine which has been

saturated with acetylene and to which benzene, benzene, or toluene have been added. Before the addition of the alcohol, the gum-forming constituents of the turpentine are precipitated by the addition of commercial lysol or cresol containing neutral soap. After decantation, 4–5 vols. of alcohol are added, and no separation takes place.

T. A. SMITH.

Catalytic gas generator. F. UMPLEBY (U.S.P. 1,738,620, 10.12.29. Appl., 29.1.27. U.K., 29.1.26).—See B.P. 269,269; B., 1927, 435.

Incorporation of material to be briquetted with a liquid building medium [by spraying nozzles]. E. KLEINSCHMIDT (B.P. 322,220, 31.8.28).

Liquid fuel burners. P. FARROW, and CANNING TOWN GLASS WORKS, LTD. (B.P. 322,257, 10.9.28).

Liquid fuel burners. P. TARAGNO, and SOC. ANON. PER L'IMPIEGO RAZIONALE DEGLI OLII COMBUSTIBILI I.G.N.E.A. (B.P. 322,337, 28.11.28).

[Fuel shut-off device for] furnaces heated by oil burners. R. THÉRY (B.P. 303,510, 31.12.28. Fr., 6.1.28).

Muffle furnace (U.S.P. 1,730,570).—See I. **Unsaturated hydrocarbons** (B.P. 322,284).—See III. **Bituminous compositions** (B.P. 321,948).—See IX. **Treatment of metal** (U.S.P. 1,736,282).—See X. **Insulating materials** (B.P. 322,208).—See XI. **Salts of sulphonic acid** (B.P. 321,980).—See XIII. **Higher alcohols, acetone, etc.** (B.P. 322,029).—See XVIII.

III.—ORGANIC INTERMEDIATES.

Detection and determination of benzoic and salicylic acids, particularly in foodstuffs. H. C. S. SNETHLAGE (Chem. Weekblad, 1929, 26, 604–605).—Methods are suggested by which clear aqueous filtrates, suitable for extraction with a mixture of ether and light petroleum, may be obtained in different cases. The acids are extracted from the organic solvents by sodium hydroxide, and detected and determined colorimetrically by means of the reactions with ferric chloride.

S. I. LEVY.

2:4-Dichloro-1-aminoanthraquinone. I. GUBELMANN, II. J. WEILAND, and O. STALLMANN (Ind. Eng. Chem., 1929, 21, 1231–1232).—This compound cannot be prepared by the methods used for the corresponding dibromo-derivative, for which it may serve as a cheaper substitute in the preparation of dyes not containing the original halogen, but is obtainable as follows: *m*-Dichlorobenzene is treated with phthalic anhydride and aluminium chloride, the resulting 4':6'-dichloro-*o*-benzoylbenzoic acid, m.p. 100–101°, being converted into its 3'-nitro-derivative, m.p. 174°, which is reduced to the 3'-amino-compound, m.p. 164°. This is then condensed to the final product, m.p. 205–206°, in presence of sulphuric acid. The yields in each step are high, the total yield being about 70%.

T. H. POPE.

Extraction of acetic acid and alcohol. MARILLER. **Pyrolysis of the paraffins.** HAGUE and WHEELER.—See II. **Glycerin analysis.** PETERSON.—See XII. **Enzymes as reagents.** BARGELLINI.—See XVIII. **Sodium citrate.** CATULLO.—See XX.

PATENTS.

Manufacture of unsaturated hydrocarbons of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,284, 11.10.28).—The capacity of metals of group VIII to act as catalysts in the hydrogenation of oxides of carbon to liquid products is increased by the addition of small amounts of sulphur to the alkaline catalyst. This lengthens the life of the catalyst, causes the production of liquids of lower b.p., and promotes the formation of unsaturated compounds. The proportion of sulphur (free or combined) should be 0.15–0.4% of the metal, and of alkali metal (free or combined) 0.3–0.6%. Addition of selenium or tellurium produces a similar result. The catalysts are best prepared by the addition of the alkali sulphide or hydrosulphide to the hydroxide of the metal obtained by precipitating a solution of a salt. The solution should be acidified with nitric acid and precipitated by ammonia or alkalis; the precipitated hydroxide should be well washed. 200 c.c. of an iron catalyst, the preparation of which is described, when dried at 170–180° and reduced at 350° produces, from 1 m.³ of a gas containing 25% CO and 70% H₂, at 260°, and by passing once over the catalyst 25 c.c. of liquid hydrocarbons of which only 20% boils above 180°. With no sulphur present a similar catalyst yields a product of which 55% boils above 180°. The bromine values of the respective products are 140 and 95. Gaseous unsaturated hydrocarbons containing 3–5 carbon atoms are also obtained, and their formation may be favoured or hindered by appropriate reaction conditions. A catalyst containing cobalt yields 36 c.c. of liquid products (bromine value 130–135, 10% boils above 180°) from 1 m.³ of gas. The unsaturated hydrocarbons are of value as anti-knocks or, after condensation by means of aluminium chloride etc., for the production of lubricating or insulating oils. They may also be used for the preparation of benzene hydrocarbons, alcohols, etc.

T. A. SMITH.

Catalytic reduction of oxides of carbon and organic oxygen compounds. SELDEN CO., ASSEES. OF A. O. JAEGER (B.P. 306,471, 14.4.28. U.S., 21.2.28).—Oxides of carbon, ketones, aldehydes, or carboxylic acids are reduced with hydrogen etc. in the gaseous phase at high temperatures in presence of contact masses comprising base-exchange substances (other than multi-component zeolites) in which a catalyst is present in chemical combination or physical association; stabilisers and/or stabiliser-promoters may be added.

C. HOLLINS.

Manufacture of acetaldehyde from acetylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,542, 3.12.28).—Ammonium salts, or ammonia and acid, are added to the mercury solutions to prevent precipitation of sludge and prolong the activity of the catalyst.

C. HOLLINS.

Production of acetic acid [from acetaldehyde]. S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 319,433, 7.7.28).—A 10–25% solution of acetaldehyde in acetic acid is oxidised by air or oxygen in very finely-divided form, produced, e.g., by forcing the gas through a porous plate at the bottom of a tower into which the solution is fed. Cooling or heating coils

may be used, and the exit gases may be scrubbed with acetic acid.

C. HOLLINS.

Concentration of acetic acid. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 298,137, 3.10.28. Ger., 3.10.27).—Dilute acetic acid is distilled with butyl acetate to remove water, and the ester is then removed by adding the calculated quantity of water (liquid or as steam) and distilling off the azeotropic mixture. Other esters of b.p. between 103° and 133° may be used.

C. HOLLINS.

Manufacture of aliphatic [acetic] esters and acids. H. DREYFUS (B.P. 319,030, 16.3.28).—Methyl ether and carbon monoxide are passed at 100–150 atm. over a catalyst (sodium formate or methoxide) at 300–400° to give methyl acetate. Other ethers in analogous manner yield esters of higher acids.

C. HOLLINS.

Manufacture of lactic acid esters. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 319,043, 14.6.28).—Magnesium lactate, dried at 110–120°, is finely powdered, mixed with alcohol, and sulphuric acid is added below 20°. After keeping for 24 hrs. the mixture is warmed for 6 hrs. at 30° and nearly neutralised (using light Congo paper) with magnesia. The whole is then heated at 70° for 6 hrs., carefully neutralised at 30°, and worked up as usual.

C. HOLLINS.

Manufacture of vic-trihalogenobenzenes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 319,149, 4.10.28).—The Sandmeyer reaction is applied to 2:6-dihalogenosulphanilic acids and 2-halogeno-*m*-phenylenediamine-5-sulphonic acids, the amino-group or groups being replaced by halogen, and the sulphonic group is then removed by hydrolysis with sulphuric acid. The preparation of 1:2:3-tribromobenzene, m.p. 87°, and 1:2:3-trichlorobenzene, m.p. 54°, is described.

C. HOLLINS.

Manufacture and purification of triaryl phosphates. (A, B) IMPERIAL CHEM. INDUSTRIES, LTD., W. GIBSON, C. R. HENSHAW, and (A) J. B. PAYMAN (B.P. 322,036 and 322,057, [A] 27.9.28, [B] 24.10.28).—(A) Phenols are heated with phosphorus oxychloride at about 200° in the presence of small quantities of organic bases; the products are plasticisers for cellulose ester solutions. (B) The crude products are stirred at about 100° with small quantities of oleum (20% free SO₃), then washed with water until free from acid, and dried. The products may be used direct or distilled *in vacuo*.

L. A. COLES.

Separation, isolation, and purification of aromatic hydroxyaldehydes. GRAESSER-MONSANTO CHEM. WORKS, LTD., and D. P. HUDSON (B.P. 318,939, 11.5.28).—Vanillin and isovanillin are separated by fractional acidification of their aqueous sodium salts, isovanillin being precipitated first, or by treatment with just sufficient sodium hydroxide solution to dissolve the vanillin. Similarly, guaiacol may be separated from vanillin, salicylaldehyde from *p*-hydroxybenzaldehyde, pyrocatechol monoethyl ether ("ethacol") from bourbonal, etc. For sharp separation dilute solutions of the alkali salts and dilute mineral acid or very weak acids should be used, and the acid must be added slowly with vigorous agitation.

C. HOLLINS.

Manufacture of alkoxy-3-[hydr]oxythionaphthens [alkoxythioindoxyls]. I. G. FARBENIND. A.-G. (B.P. 298,493, 8.10.28. Ger., 8.10.27).—Aluminium chloride may be used for the cyclisation of alkoxyarythioglycolic acids, preferably in presence of a diluent (chlorobenzene) and at low temperatures (30–40°). The preparation of 5-chloro-7-methoxy-4-methylthioindoxyl, 6-ethoxy- β -naphthathioindoxyl, m.p. 142–144°, and 5-ethoxy- α -naphthathioindoxyl, m.p. 157–158°, is described.

C. HOLLINS.

Manufacture of methyl alcohol. H. DREYFUS (U.S.P. 1,738,989, 10.12.29. Appl., 27.5.26. U.K., 13.6.25).—See B.P. 262,494; B., 1927, 124.

Treatment of hydrocarbon gas (B.P. 290,613). **Hydroaromatic hydrocarbons** (B.P. 322,445).—See II. **Salts of sulphonic acids** (B.P. 321,980).—See XIII. **Higher alcohols, acetone, etc.** (B.P. 322,029). **Denaturing alcohol** (B.P. 298,611).—See XVIII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of a green vat dye from 1:12-perylenequinone. F. BENSA (B.P. 300,922, 19.11.28. Austr., 19.11.27).—1:12-Perylenequinone is heated at 165° with aluminium chloride and benzoyl chloride (1.5 pts.), and the product is purified by vatting.

C. HOLLINS.

Manufacture of yellow azo dyes [for wool]. I. G. FARBENIND. A.-G. (B.P. 290,253, 11.5.28. Ger., 11.5.27).—Level wool dyes fast to light are obtained by coupling halogenated 1-*o*-sulphophenyl-3-methyl-5-pyrazolones with diazo- or tetrazo-compounds other than diazotised aminoacylated *o*-aminophenolsulphonic acids. Examples are: sulphanilic acid \rightarrow 1-(4'- or 5'-chloro-2'-sulphophenyl)methylpyrazolone; *o*-chloroaniline \rightarrow 1-(*p*-chloro-*o*-sulphophenyl)methylpyrazolone.

C. HOLLINS.

Manufacture of disazo dyes [for wool]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 318,882, 11.6.28).—2:3-Hydroxynaphthoic arylamides are coupled with a diazotised aminoazo-compound containing two sulphonic groups or one sulphonic and one carboxylic group to give blue to blue-black wool dyes fast to washing, hot-pressing, and light. Examples are: naphthionic acid \rightarrow Cleve acid \rightarrow 2:3-hydroxynaphthoic *p*-aniside; α -naphthylamine-3:6-disulphonic acid \rightarrow α -naphthylamine \rightarrow anilide; 5-aminosalicylic acid \rightarrow Cleve acid \rightarrow anilide.

C. HOLLINS.

Manufacture of [black tetrakis-]azo dyes [for printing on silk]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 319,407, 23.6.28).—Benzidine-disulphonic or -dicarboxylic acid or a derivative is tetrazotised and coupled with 2 mols. of a middle component (the same or different), and the product is tetrazotised and coupled with 2 similar or dissimilar mols. of a 2:8-aminonaphthol (or its *N*-aryl derivative) carrying either two sulphonic groups in the naphthalene nucleus or one such sulphonic group and a sulphonic or carboxylic group in the *N*-aryl residue. Examples are: benzidine-2:2'-disulphonic acid \rightarrow 2 mols. of Cleve acid \rightarrow 2 mols. of H-acid; benzidine-3:3'-disulphonic acid \rightarrow 2 mols. of

oresidine \rightarrow 1 mol. of H-acid and 1 mol. of *m*-carboxyphenyl- γ -acid; benzidine-2:2'-disulphonic acid \rightarrow 1 mol. of oresidine and 1 mol. of Cleve acid \rightarrow 2 mols. of H-acid.

C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Microbiology of wool. Enhancement of "mildew" by soaps and vegetable oils. R. BURGESS (J. Text. Inst., 1929, 20, T 333—372).—Mildew on cellared or aged and stored wool goods is due to mould fungi rather than to bacteria. Soaps, wool creams, and the majority of conditioning fluids enhance its development on account of the alkali present in them, which, by combining with the wool, renders it more easily attacked. Another factor in the case of hygroscopic soaps is the increase in the range of relative humidity over which mould growth can occur. Vegetable oils such as olive oil enhance mildew growth, but to a smaller extent than soap, and in this connexion the nutritive value of the oil is important. The presence of foets in the oil does not increase liability to mildew. The commercial conditioning liquid "Hygrolit" and two other fluids resembling it retard the mould growth, as also do the lower fatty acids, such as hexoic, octoic, and lauric acid; on the other hand, more complex fatty acids, *e.g.*, oleic acid, favour the growth. Mineral oils have a protective effect and are not used as food by mould fungi. The greenish-yellow stains commonly found on mildewed wool are caused by mould action on the soap present. Washing scoured or unscoured wool in several changes of hot water reduces the liability to mildew. The reaction range over which mould growth can occur on wool is considerable, since both acid and alkaline conditions favour it. It is recommended that, if possible, the use of conditioning liquids should be discontinued, and water used instead. The practice of dispatching goods in air-tight cases is unsound, since the stagnant conditions so produced favour the growth of fungi by setting up localised regions of moisture condensation.

B. P. RIDGE.

Physical and chemical characteristics of hemp stalks and of seed flax straw. E. R. SCHAFER and F. A. SIMMONDS (Ind. Eng. Chem., 1929, 21, 1241—1244).—Analyses of hemp fibre (*Cannabis sativa*) grown on peat marshes in Wisconsin show it to be practically identical with flax straw (*Linum usitatissimum*). The growing of hemp for paper-making in these areas is discussed.

A. G. POLLARD.

Chemical characteristics of different celluloses. S. FOTJEW and W. JAKIMANSKI (Zellstoff u. Papier, 1929, 9, 834—835).—The percentage of ash, of ether- and alcohol-soluble resin, α -cellulose content, pentosan content, the copper number and permanganate number (as indicative of the lignin content) of different cellulose materials have been determined in order to afford a means of distinguishing between them. Sulphate differs from sulphite-cellulose in that it has a lower resin content, lower copper number, and a somewhat higher pentosan content. Straw cellulose has a very high ash and pentosan content and a correspondingly lower α -cellulose content. Bleached sulphite-cellulose has a smaller lignin, pentosan, and α -cellulose content than

unbleached. Purified cellulose has a smaller lignin, ash, and resin content, and a much larger α -cellulose content than ordinary bleached material. Methods used in the determination of these properties are given.

B. P. RIDGE.

Preparation of hydrocellulose in the cold and by disintegration. J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 79—80).—A process is described for obtaining from cotton a hydrocellulose suitable for the preparation of smokeless powders. The cotton (400 g.) is treated for $\frac{1}{2}$ hr. with hydrochloric acid (5 litres, d 1.14). The material is washed with water and finally with dilute sodium carbonate solution; 83.5% of the acid is recovered.

R. K. CALLOW.

Determination of hydrocellulose and oxycellulose in commercial celluloses. J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 76—78).—A preliminary note on the difficulty of obtaining concordant results by various published methods.

R. K. CALLOW.

Action of caustic soda on cellulose. I. FUKUSHIMA and Y. TAKAMATSU (J. Cellulose Inst., Tokyo, 1929, 5, 255—262).—The solubility of cellulose in sodium hydroxide solutions increases with decreasing temperature, and maxima are shown for 10, 8, and 7—8 vol.-% solutions at, respectively, 20°, 7°, and —10°. Maximum solubility coincides with maximum swelling at 1° in an 8 vol.-% solution. Maximum contraction of cellulose (as ramie sliver) occurs with 10 and 12% solutions at 20° and 4°, respectively, and contraction also increases with decreasing temperature. Contraction appears to be intimately connected with solubility and swelling, and it is thought that in mercerisation contraction and dissolution occur before swelling. The viscosity of solutions of regenerated cellulose in cuprammonium increases with increasing solubility of the cellulose during mercerisation, *i.e.*, with increasing removal of hemicellulose. The viscosity of viscose prepared from alkali-cellulose depends on the temperature of mercerisation. The hydrolysis value (Schwalbe) of regenerated cellulose increases with increasing concentration of the sodium hydroxide used, and also with decreasing temperature. Temperature has therefore a considerable effect on the properties of cellulose during and after mercerisation.

B. P. RIDGE.

Control of viscosity of solutions of cellulose. F. OLSEN and H. A. AARONSON (Ind. Eng. Chem., 1929, 21, 1178—1181).—By carefully controlled action of acids on cellulose it is possible to bring about considerable physical disintegration of cotton fibres with a minimum of chemical degradation. Such conditions result in a very considerable decrease in the viscosity of cuprammonium solutions of the product. Treatment with 5% sulphuric acid at 50° for 16 hrs. is convenient (cf. U.S.P. 1,615,343; B., 1927, 580). A. G. POLLARD.

Desulphurisation of viscose [rayon] silk. Y. KAMI and T. MATSUYAMA (J. Cellulose Inst., Tokyo, 1929, 5, 293—304).—Viscose rayons of 120 and 150 denier were treated with sodium sulphide solutions of various concentrations for 20 min. at 50°, and of the same concentration at different temperatures. The concentration of the bath before and after the immersion was determined iodometrically, whilst the sulphur

content of the desulphurised material was found by the oxidation method. Provided that sufficient sodium sulphide to react with the sulphur in the material is present, and that a sufficiently long time of reaction is given, the desulphurisation process is independent of the concentration of the bath. Temperature and the total quantity of sodium sulphide present are of little importance if the concentration of the solution exceeds 20 g./litre, and in desulphurisation by the steeping process the use of a solution of this concentration for 15 min. at 25–30° is recommended, whilst for the "passage" process a higher concentration and temperature are necessary. The relation between sulphur content (S%) of the desulphurised rayon and the temperature (θ°) of the bath is given by the empirical formula $S = 76.970 - 0.8973\theta$, from which it is evident that it is impossible completely to remove the sulphur in the rayon by means of sodium sulphide. Iodometric analysis of the desulphurising liquor does not distinguish between mono- and poly-sulphides, hence the active power of the solutions (*i.e.*, the quantity of monosulphide present) cannot be determined by this method. Treatment of the rayons with solvents for sulphur (*e.g.*, carbon disulphide) and with agents which react with sulphur to form water-soluble compounds (*e.g.*, ammonium sulphide or ammonia) shows that desulphurisation occurs not as the result of direct dissolution of sulphur, but as a result of the action of chemical reagents which convert it into compounds which are readily soluble in water.

B. P. RIDGE.

Production and treatment of cellulose in the paper industry. J. STRACHAN (Inst. Chem. Eng., Nov. 27, 1929. Advance copy. 4 pp.).—An outline is given of the sources of cellulose for papermaking, and the various pulping processes are described. Of the more recently suggested processes, that in which neutral sodium sulphite is used is the most promising, sodium sulphite having little or no degrading effect on cellulose at moderate cooking temperatures. Efficient recovery of the black liquor is, however, difficult. Electrical conditions have a marked effect on digestion processes in general, and by the application of very small electrical charges from an outside source satisfactory digestion may be accomplished at lower temperatures and with a reduced consumption of chemicals.

D. J. NORMAN.

Oak chips from tan-works as raw material for paper. W. NERECITSKIJ (Zellstoff u. Papier, 1929, 9, 835–836).—Waste chips may be used for the manufacture of cellulose, but the bark, which cannot be removed after the lixiviation process, is detrimental to the properties and quality of the pulp. Oak wood contains 5–6.5% of tannin material, of which the heartwood contains 82%, the sapwood 2%, and the bark 16%. By de-barking the wood and removing the sapwood before extraction an improved tannin extract may be obtained. The sapwood could then be used for the manufacture of cellulose, whilst a second tannin extract could be obtained from the bark and the residue of the latter would serve as fuel. Alternatively the bark could be burned without extraction, thus giving an increased yield of heat. De-barked, lixiviated waste chips give 51.3% of unbleached cellulose or 46.1%

of bleached material; this corresponds to 27.7% on the original wood. The unbleached pulp may be used for the manufacture of packing papers, the price of which is lower than that of similar papers from straw cellulose. The properties of such papers and of pasteboards made from waste chip cellulose are also superior to those of corresponding materials from straw cellulose. For the economical conversion of the waste chips into paper the lixiviation and paper works should have a common steam-power plant.

B. P. RIDGE.

Chintschin's sizing process [for paper]. A. MARTJEMIANOV (Zellstoff u. Papier, 1929, 9, 838–839).—Papers of different kinds were sized by the usual process (in which the necessary ingredients, pulp, filling, rosin, alumina, etc. were simultaneously mixed together before beating), and by Chintschin's method, in which softened water was used and the alumina was added 30–40 min. after admixture of the rosin milk with the other ingredients. In the latter case, 41.6–50% saving of rosin and 27–33% of alumina was effected. This behaviour is similar to that found for the Delthirma process. The most satisfactory results were obtained by use of a size containing 50–54% of combined rosin. The superiority of Chintschin's process is demonstrated by a comparison of the rosin and alumina consumptions on a price basis.

B. P. RIDGE.

Action of liquids on paper. J. STRACHAN (Proc. Tech. Sect. Papermakers' Assoc., 1929, 9, 220–228).—An outline is given of the effect of beating and different methods of sizing on the penetration of paper by water, oils, varnishes, etc. Since resin sizing confers only increased resistance to wetting by aqueous liquids and has no appreciable influence on the absorption of oils and varnishes, it is suggested that a considerable saving might be effected and a better paper produced if resin sizing were omitted entirely from most printing papers. The resistance of paper to penetration by all liquids, particularly oils and varnishes, increases with the degree of beating and the closeness of the sheet.

D. J. NORMAN.

Testing crude pasteboard by its absorption of anthracene oil. KORN (Papier-Fabr., 1929, 27, 765–766).—In this test samples of material each weighing 1 g. are slowly immersed in the oil, allowed to drain completely, weighed, and the percentage of oil absorbed is found. Conditions affecting the results obtained have been investigated, using an oil of d 1.095, and samples weighing 1 g. cut square in shape, and also larger ones of 1 sq. m. surface area. The time of draining is shorter for the small than for the large test pieces, and, in general, it is shorter for those of higher weight per sq. m. than for those of lower weight. The longer the time of hanging after complete drainage, the smaller is the oil absorption; the values are 0.5–2% and 4–11.6% lower, respectively, if weighing is done 1 hr. and 18 hrs. after drainage is complete. Better average results are obtained by using large rather than small samples, but the former give lower values than the latter. The length of the test pieces used is important, since if long narrow strips are taken instead of squares lower values are given, hence it is necessary to use samples of definite surface dimensions in carrying out the test.

B. P. RIDGE.

Extraction of acetic acid etc. MARILLER.—See II. Leather and sulphite-cellulose extract. MERRILL and BOWLUS.—See XV. Fermentation of cellulose. BORUFF.—See XVIII.

PATENTS.

Manufacture of textile fibres. TEXTILES (NEW PROCESS), LTD. (B.P. 321,903, 18.4.29. Fr., 8.10.28).—Waste fibrous vegetable materials such as remnants of jute sacks are wetted with a dilute solution of olein, magnesium sulphate, potassium nitrate and phosphate, heated to about 30°, heaped, and allowed to ferment for 24–48 hrs. After being unravelled, they are boiled with a dilute solution of caustic soda or sodium carbonate and magnesium chloride, which solution may have been used for a previous treatment, washed, bleached, and passed into a bath containing soap, vaseline oil, and a suitable alkali. Finally the material is dried to a water content of 30%, carded, and again dried. F. R. ENNOS.

Improvement of vegetable fibrous material. L. LILIENTFELD (B.P. 320,062, 2.7.28).—Textile materials are treated with a solution of a cellulose derivative containing the CS·S group from which cellulose may be regenerated, *e.g.*, viscose, and are then treated before, during, or after regeneration of the cellulose with shrinking agents, *e.g.*, strong caustic alkalis or mineral acids. Gas-forming compounds, *e.g.*, carbonates, may be incorporated with the viscose solution, also fillers, pigments, softening agents, etc. Materials dressed in this way have a soft feel, good extensibility without cracking of the viscose layer, and are substantially unaffected by washing. Thirty-four examples are given. D. J. NORMAN.

Treatment of wood, fabrics, and other cellulosic materials. (SIR) G. C. MARKS. From CURTIN-HOWE CORP. (B.P. 321,786, 9.10.28).—The material is rendered non-inflammable and resistant to insect and fungus attack by impregnation with a solution containing ammonia and upwards of 5% of arsenious acid, capable of depositing free arsenious oxide on exposure to air. F. R. ENNOS.

Washing of wool and other textile materials and compositions for use therefor. N. E. WILLIS (B.P. 321,729, 21.7.28).—The scouring liquid consists of a dilute aqueous solution containing a buffer salt (trisodium phosphate, borax, or ammonium carbonate), whereby the alkalinity is controlled between p_H 7·5 and 11, a water-soluble carbohydrate (glucose), an emulsifying protein, and an antiseptic. F. R. ENNOS.

Production of cellulose from wood. G. GURTNER (Swiss. P. 123,468, 23.3.25. Addn. to Swiss P. 120,266).—Wood is treated with dilute hydrochloric, sulphuric, nitric, or acetic acid, and is subjected to mechanical treatment in the presence of part of the acid until loosening of the fibre structure occurs. B. P. RIDGE.

Cellulose compounds for use in the manufacture of transparent paper and artificial silk. C. RUZICKA (B.P. 321,521, 5.10.28).—Cotton cellulose, wood cellulose, or the like is intimately mixed with phenol, or a soap-forming fatty acid or resin, and is then treated with strong alkali solution, *e.g.*, caustic soda solution (d 1·36),

and left to ripen at the ordinary temperature for a period varying from a few hours to a month or more. The mass is then dissolved by churning in caustic soda solution of d 1·18, optionally with addition of carbon disulphide to facilitate dissolution. The resulting cellulose solution when precipitated with, *e.g.*, 15% sulphuric acid gives products (apparently esters of cellulose with fatty or resin acids) which are particularly suitable for artificial filaments or films which require to show resistance to water. Suitable proportions are 162 pts. of cellulose, 57 pts. of castor oil fatty acids, and 288 pts. by wt. of caustic soda solution (d 1·36), with a ripening time of one month at 15·5°. Hardening agents, *e.g.*, formaldehyde, may be used at any stage of the process. D. J. NORMAN.

[Production of] artificial silk and like filamentary material. C. H. FIELD and D. HASLETT (B.P. 319,294, 13.6.28).—Artificial filaments consisting of a mixture of cellulose xanthate and cellulose acetate and/or cellulose nitrate are obtained by spinning a solution of cellulose acetate or nitrate and cellulose xanthate in epichlorohydrin, mono- or di-chlorohydrin, an acetin or like ester of glycerol, or glycerin itself into an acid coagulating bath, *e.g.*, a hydrochloric acid-ammonium chloride bath. The xanthate may be prepared and ripened in the usual way, but is preferably allowed to coagulate spontaneously, and should contain a minimum quantity of water. A suitable spinning solution is made by mixing equal parts of a 5–7% solution of cellulose acetate in epichlorohydrin and an 8–12% solution of cellulose xanthate in glycerin and dichlorohydrin (1 : 1). D. J. NORMAN.

Production of artificial filaments, yarns, or threads. BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and W. I. TAYLOR (B.P. 321,762, 13.9. and 18.12.28).—In the production of threads of cellulose acetate silk by the dry-spinning method, the filaments are drawn at varying linear speeds by suitable means, such as passing them round a roller the peripheral speed of which is varied, whereby a more or less frequent, regular, and systematic variation in denier is imparted thereto. F. R. ENNOS.

Manufacture of artificial threads or filaments. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 320,106, 5.7.28).—In the manufacture of artificial filaments by the dry-spinning process, starting-up is facilitated if a vacuum is applied to the outer face of the jet until an uninterrupted flow is obtained through all the orifices. Suitable devices are described. D. J. NORMAN.

Manufacture of artificial silk and the like. H. P. BASSETT and T. F. BANIGAN, ASSRS. to H. P. BASSETT (U.S.P. 1,736,280, 19.11.29. Appl., 19.5.24).—Freshly-spun cuprammonium silk filaments are washed to remove excess alkali, twisted while wet, again washed, partly dried without being under tension, and the moisture content is suitably reduced while under tension. P. E. L. FARINA.

Manufacture of artificial [silk] threads. L. LILIENTFELD (B.P. 319,293, 11.6.28. Addn. to B.P. 281,352; B., 1928, 228).—The concentration of caustic soda solution specified in the earlier patent is extended to

include solutions of 2–5% concentration (calc. as NaOH). D. J. NORMAN.

Manufacture of artificial thread from viscose. L. LILIENFELD (B.P. 321,679, 11.8.28. Addn. to B.P. 274,521 and 274,690; B., 1927, 745).—A stream of viscose is coagulated by passing it into an atmosphere containing a gaseous mineral acid or acid anhydride (hydrochloric acid, sulphur trioxide) with or without an inert diluent such as air or carbon dioxide, whereby threads of high dry tenacity exceeding 2 g. per denier are obtained. F. R. ENNOS.

Viscose artificial silk. C. R. LINKMEYER (F.P. 631,674, 18.3.27).—The precipitation liquid coming from the spinning machine is heated to boiling, whereby the fine particles of viscose which separate out during the precipitation are converted into other substances which dissolve in the precipitation bath. B. P. RIDGE.

Production of regenerated cellulose structures [from viscose]. S. DUNLOP. From E. H. MORSE (B.P. 320,161, 24.7.28).—Viscose films of improved strength and flexibility are made by passing an unsupported film of viscose solution of the usual degree of ripeness first through a coagulating, but not cellulose-regenerating, bath and then through a series of regenerating baths of gradually increasing acidity. An 18% solution of ammonium sulphate containing 1% or less of sulphuric acid at 24° may be used as the coagulating bath, and a solution containing 10% of ammonium sulphate, 5% of sodium sulphate, and about 2% of sulphuric acid as the first regenerating bath. The second (and third) regenerating baths may contain 13–17% (5%) of sodium bisulphate and 1.5–2.5% (3.5–4%) of free sulphuric acid, and the final bath sulphuric acid of about 6% concentration. The temperature of these baths is preferably about 24°. D. J. NORMAN.

Manufacture of cellulose ethers. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,651, 9.8.28).—Water-insoluble cellulose ethers are purified by extraction with a mixture of water and a water-soluble solvent, e.g., dilute alcohol, acetone, or acetic acid, to which are added a small quantity of alkali and a bleaching agent (hydrogen peroxide), whereby the constituents of low viscosity which swell in water are removed. F. R. ENNOS.

Treatment of cellulose acetates. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 321,648, 12.7.28).—To modify their solubility, cellulose acetates dissolved in anhydrous or aqueous phenols or polyphenols are saponified at 30–70° by means of alkaline phenoxides, the amount of the latter used being that which is theoretically necessary for fixing the quantity of acetic acid which it is desired to eliminate from the cellulose acetate. F. R. ENNOS.

Manufacture of plastic masses from cellulose ethers. I. G. FARBENIND. A.-G. (B.P. 295,940, 20.8.28. Ger., 19.18.27).—The cellulose ether, in particular ethyl cellulose, is mixed with an alcohol of the terpene series and, if desired, with a solvent (alcohol, benzene) and/or a softening agent (benzyl alcohol, isobutyl phthalate). F. R. ENNOS.

Manufacture of materials from cellulose ethers and esters. I. G. FARBENIND. A.-G. (B.P. 295,942, 20.8.28. Ger., 19.8.27).—Films and fibres of enhanced tensile strength and extensibility are made from solutions of cellulose ethers and esters (other than nitrocellulose) in a solvent in which carbon disulphide is the main constituent. F. R. ENNOS.

Cellulose foil. L. RADO (B.P. 305,571, 22.10.28. Ger., 7.2.28).—The foils are rendered waterproof and their content of softeners is retained therein by coating them by the use of adhesives on one or both sides with thin metal foils; in the former case the other side is covered with a celluloid or cellulose nitrate solution. F. R. ENNOS.

Manufacture of valuable products from carbohydrates. W. TRAUBE (B.P. 294,572, 5.7.28. Ger., 27.6.27).—Cellulosic materials, starch, or other carbohydrates are treated in the dry state with sulphur trioxide vapours either *in vacuo* or in admixture with an inert gaseous medium, e.g., dry air, until the parent material has absorbed 50% or more of its weight of sulphur trioxide. The brownish, semi-solid reaction product is partly or completely soluble in water, the solubility depending on the amount of sulphur trioxide absorbed, and gives, on neutralisation of its aqueous solution with caustic potash, compounds of the general formulae $C_6H_5O_5(SO_3K)_3 \cdot H_2O$ and $C_6H_5O_5(SO_3K)_4 \cdot H_2O$. These products have low copper numbers, give highly colloidal solutions, and may be used as intermediates in the manufacture of cellulose esters or as protective colloids. D. J. NORMAN.

Manufacture of valuable products from carbohydrates. W. TRAUBE (B.P. 322,003, 30.8.28. Addn. to B.P. 294,572; preceding abstract).—The carbohydrate is esterified as in the prior process except that the sulphur trioxide is dissolved in a liquid medium inert to the reagents under the reaction conditions (carbon disulphide). F. R. ENNOS.

Flexible sheet material. [Glass substitute.] BRIT. CELANESE, LTD. (B.P. 308,587, 13.3.29. U.S., 23.3.28. Addn. to B.P. 307,462).—Iron or steel wire netting etc. is coated with a solution of a wholly or partially polymerised vinyl compound resin with or without an organic derivative of cellulose (cellulose acetate), polymerisation of the former substance being completed, if necessary, after application of the coating material. The quantity of this coating may be either sufficient or insufficient to fill the reticulations, which, in the latter case, are completely closed by application of a further coating consisting of a solution of cellulose acetate with or without a content of the polymerised vinyl compound resin. F. R. ENNOS.

Treatment [varnishing] of fish-bone bristles. GIMMY & DIEPOLD GES.M.B.H. (B.P. 305,484, 4.2.29. Ger., 4.2.28).—To prevent softening by moisture absorption, the bristles are varnished with a methyl-alcoholic solution of pinic acid resin, sylvic acid, and colophony, to which vesuvium is subsequently added. F. R. ENNOS.

Operation of paper mills. Manufacture of paper. D. K. PATTILLO and J. H. MACMAHON, ASSRS, to

MATHIESON ALKALI WORKS, INC. (U.S.P. 1,733,070—1, 22.10.29, Appl., [A] 23.12.27, [B] 12.1.28).—(A) Prior to the papermaking operation, the stock is sterilised by treatment with sufficient chlorine to give it a residual chlorine content of 0.5—1 pt. per million, whilst the white water formed in the operation is treated with a coagulating agent (alum) to yield a stable, sterilised, short-fibred product. (B) The half-stock or fibre-water mixture, after formation in the beaters and when at its maximum dilution, is treated with chlorine to sterilise it and to remove slime therefrom, and is finally made into paper. **F. R. ENNOS.**

Manufacture of carbon papers and the like. **J. Y. JOHNSON.** From **I. G. FARBENIND. A.-G.** (B.P. 321,538, 29.10.28).—Improved results are obtained in the preparation of carbon papers if the oils which are normally used as softeners are partly or wholly replaced by suitable liquid or low-melting aromatic or hydro-aromatic substances such as aryl phosphates, methyl-diphenylamine, phthalic acid esters of ethylene glycol monoalkyl ethers, etc. **D. J. NORMAN.**

Manufacture of cigarette paper. **J. F. M. CRO-LARD,** and **BRIT.-AMERICAN TOBACCO CO., LTD.** (B.P. 322,149, 3.4.29).—In order to obviate the tendency of the paper to become discoloured, stained, or spotted after contact with tobacco, the pulp is mixed with a solution containing stearin and borax, the former being precipitated on the pulp by means of aluminium sulphate etc. before running off from the beater. **F. R. ENNOS.**

Preparation of transparent paper. **O. BRENNER** (F.P. 631,282, 2.3.27).—The paper is steeped for 12 hrs. in a 1% solution of ammonia, partially dried, covered with an oil varnish or polish by an immersion process, and dried. Transparent sheets of such paper, to be used for decorative purposes, should be covered with a rubber-oil varnish. **B. P. RIDGE.**

Coating of paper. **D. B. BRADNER,** Asst. to **CHAM-PION COATED PAPER CO.** (U.S.P. 1,733,524, 29.10.29, Appl., 8.11.26).—Paper in the form of a web or sheet is coated with an aqueous slurry containing raw starch and a pigment (china clay), the whole being subjected, by means of saturated steam, to sufficient heat without substantial drying in order to gelatinise the starch, and is afterwards dried and finished. **F. R. ENNOS.**

Manufacture of impregnated pasteboard articles. **M. P. OTTO** (B.P. 315,296, 11.12.28, Fr., 11.7.28).—Pasteboard articles which have been impregnated with drying oils are exposed to an atmosphere of ozonised air at the ordinary temperature to harden the oil. By thus avoiding drying at high temperatures the hygro-metric condition of the cellulose is substantially undisturbed and the article is not subject to subsequent curling and distortion. **D. J. NORMAN.**

Treatment of waste liquors from the making of paper pulp and similar liquors to generate heat and recover chemicals therefrom. **C. L. WAGNER** (B.P. 321,413, 7.8.28).—The waste liquor is sprayed downward through a refractory-lined vertical retort where it meets an upward current of heated air, which effects combustion of the volatile constituents of the liquor and allows the carbonaceous residue to fall through

the bottom of the retort into a second chamber, where the combustion process is completed by the introduction of further quantities of air. The alkali salts in the residue are recovered in the usual way. When once started, e.g., by first burning oil until the retort is sufficiently hot, the process is self-supporting and continuous, and provides sufficient heat to supply the whole of the requirements, other than mechanical power, of a well-equipped soda-pulp mill. The process can be applied to the waste liquor from the soda, sulphate, or mono-sulphite processes. **D. J. NORMAN.**

Mineral fibre paper and its manufacture. **G. C. MARKS.** From **PLASTIC, INC.** (B.P. 321,537, 26.10.28).—Asbestos fibre is formed into sheets with an insoluble metal salt of alginic acid. Thus 1000 pts. of wet kelp (100 pts. of dry kelp) are ground up with 15 pts. of soda ash and to this mixture sufficient 10% sulphuric acid is added to neutralise the alkali; the resulting coagulum is washed to eliminate natural mineral salts and is then pressed to remove water. Of this purified kelp, 15 pts. are beaten with 100 pts. of asbestos and 2.25 pts. of sodium carbonate and the mixture is sheeted on an ordinary papermaking machine. The formed sheet may be supercalendered without cracking and is stronger and more flexible than similar paper made with vegetable fibre. **D. J. NORMAN.**

Manufacture of artificial threads, filaments, ribbons, etc. **E. HAZELEY** and **E. A. MORTON,** Assrs. to **COURTAULDS, LTD.** (U.S.P. 1,739,458, 10.12.29, Appl., 7.9.28, U.K., 7.11.27).—See B.P. 305,279; B., 1929, 280.

Manufacture of textile materials [staple fibres]. **H. DREYFUS** (B.P. 322,417, 12.6.28).

Device for sucking air and other gases from centrifugal boxes used for spinning artificial silk threads. **H. WADE.** From **N. V. BOUWONDERNEMING KETABANG IV** (B.P. 322,675, 15.1.29).

Hardening of papier maché materials or the like [with hardened linseed oil varnish]. **G. OHLHAVER** (B.P. 295,259, 7.8.28, Ger., 5.8.27).

Triaryl phosphates (B.P. 322,036 and 322,057).—See III. **Plastic materials** (B.P. 322,158).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Defects [in dyed fabrics] caused by skin particles. **L. L. LLOYD** (J. Soc. Dyers and Col., 1929, 45, 343—344).—Serious defects in the appearance of dyed and finished woollen and particularly worsted fabrics may be caused by the presence of particles of sheep skin. In fabrics bleached by means of hydrogen peroxide the skin particles appear pale reddish-brown, which is deepened in tone by hot pressing or decatizing; the particles readily harden in such a way that they fracture adjacent wool fibres. In dyeing, skin particles absorb dyes more readily than the wool fibres, but in subsequent steaming and brushing the absorbed dye readily bleeds into the adjacent wool. Skin particles may be detected by first steeping in cold dilute caustic soda so that the substance assumes its normal structure sufficiently to be recognised under the microscope; the particles

may also be recognised by boiling with water and testing for glue, *e.g.*, by the tannin test. A. J. HALL.

Application of the locust bean in the textile industry, and especially in calico printing. G. TAGLIANI (J. Soc. Dyers and Col., 1929, 45, 344—349).—The locust bean contains no starch but about 50% of nitrogenous substances (hemicelluloses), and from it, after grinding, may be obtained a viscous gel suitable for use in textile printing. The adhesive properties of this gel are not high; it is readily susceptible to fermentation, and becomes more viscous in the presence of an organic acid but less so with mineral acids. All alkalis gelatinise the endosperm gel to a transparent mass, but this may be inhibited by pre-fermentation, by pre-acetylation, or by adding suitable protective colloids such as glycerin and glucose. The gel is precipitated by tannins in the cold, but the precipitate dissolves on warming or on the addition of excess of the precipitant. It forms a film on textile fibres which is not sticky but withstands friction and has a marked elasticity. A. J. HALL.

Improving the appearance of buntal fibre or of articles made therefrom. S. DEL MUNDO (Philippine J. Sci., 1929, 40, 515—517).—Hats made of buntal fibre from the leaves of buri palm (*Corypha elata*) are satisfactorily bleached by immersion in a dilute solution of potassium permanganate and sodium carbonate followed by treatment with water and acidified sodium thiosulphate solution. E. H. SHARPLES.

Dyeing of cherries. JEFFREY and CRUESS.—See XIX.

PATENTS.

Bleaching of textiles by means of solutions yielding nascent oxygen. H. WADE. From DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT, VORM. ROESSLER (B.P. 320,072, 3.7.28).—In bleaching textile materials by subjecting them to the action of rapidly circulating solutions of oxygen-liberating substances, undesirable decomposition of such substances as are produced by the catalytic effect of the materials of the apparatus is avoided by using cement or similar vessels or vessels of other materials coated with cement, and preferably painted with a solution of sodium silicate. Suitable apparatus is described. [Stat. ref.] A. J. HALL.

Dyeing [with oxidation dyes]. E. SCHUELLER (B.P. 307,732, 23.8.28. Fr., 12.3.28).—The elimination of toxic or unstable intermediate products formed during the dyeing of textiles and furs with aromatic phenolic or amino-compounds in the presence of oxidising agents is effected by washing the dyed material with a solution of an alkali or alkaline-earth salt (particularly an alkaline-earth chloride); this treatment fixes the fully oxidised products but not those incompletely oxidised. A. J. HALL.

Production of coloured materials made with or containing cellulose esters. BRIT. CELANESE, LTD. (B.P. 295,579, 10.8.28. U.S., 13.8.27).—Instead of applying vat and sulphur dyes to cellulose ester fabric from a dye bath necessarily containing a caustic alkali so that the silk is deleteriously affected, the fabric is printed all over with a paste containing the dye and

caustic alkali and then steamed; the fabric is not adversely affected under these conditions, although partial hydrolysis of the ester occurs. White or coloured discharge effects may be obtained by the usual methods.

A. J. HALL.

Laundering textile fabric. R. A. PHAIR and B. BUCARIA, ASSRS. to H. KOHNSTAMM & Co., INC. (U.S.P. 1,732,729, 22.10.29. Appl., 6.3.25).—During the bleaching operation a porous silica compound (kieselguhr) is added to absorb oily matters and assist in removing stains. F. R. ENNOS.

Manufacture of dyed artificial silk. H. J. J. JANSSEN, ASSR. to N. V. NEDERL. KUNSTZIJDEFABR. (U.S.P. 1,739,475, 10.12.29. Appl., 15.12.25. Ger., 15.12.24).—See B.P. 244,496; B., 1927, 406.

Dyeing of organic derivatives of cellulose. W. WHITEHEAD, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,738,978, 10.12.29. Appl., 11.12.26).—See B.P. 282,036; B., 1929, 169.

Apparatus for washing woven fabrics. M. FREIBERGER, and GES. F. TEXTILVEREDELUNG M.B.H. "GETEVAU" (B.P. 322,219, 30.8.28).

Drying system etc. (U.S.P. 1,731,290).—See I. Varnish-coated fabrics (B.P. 301,024).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Recovery of iodine in manufacture of nitric acid. J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 89—90).—The sodium hydrogen sulphate obtained in the manufacture of nitric acid from Chili nitrate contains iodate, and iodine is liberated when it is heated with water and scrap iron. By absorption of the iodine vapour in soda solution 15—50 g. of iodine may be recovered per ton of nitrate. R. K. CALLOW.

Properties of nickel in caustic [soda] evaporation. R. J. MCKAY (Ind. Eng. Chem., 1929, 21, 1283—1287).—Corrosion in caustic soda evaporators may be by the solution or by the steam. Tests over a long period with welded nickel tubes inserted in an evaporator showed a thinning of 0.06 mm. per year mainly from the steam side. Data of a similar nature from laboratory tests on nickel and other metals are given. Variations are due to variations of the conditions, but the corrosion rate is low enough for many years' service. Other experiments show that the corrosion of nickel by steam is negligible unless an unusual concentration of carbon dioxide is present. Galvanic corrosion of steel may occur at a point of contact with nickel. The production of caustic soda free from iron and copper is a desirable result of the use of nickel. The physical properties of nickel tubing and nickel tube sheets are discussed. Steel or cast iron evaporators may be lined with light-gauge nickel sheets electrically welded. C. IRWIN.

Reactions involved in the Hargreaves [saltcake] process. B. NEUMANN and H. KUNZ (Z. angew. Chem., 1929, 42, 1085—1087).—In this process sodium chloride is treated with sulphur dioxide (burner gases), air, and moisture at 500°. The principal reaction is probably $\text{SO}_2 + \text{H}_2\text{O} + 2\text{NaCl} = \text{Na}_2\text{SO}_3 + 2\text{HCl}$, the sodium sulphite being afterwards oxidised. The equilibrium

constant for this reaction was determined experimentally at temperatures from 350° to 600° and compared with values calculated from Nernst's formula. The experimental results were about 100 times the calculated values, the discrepancy increasing with the temperature. A turbidity was noticed in the oleum used to absorb moisture from the exit gases in the apparatus, and it was concluded that the variation was caused by decomposition of the sulphite formed: $3\text{Na}_2\text{SO}_3 + \text{SO}_2 + \text{H}_2\text{O} = 3\text{Na}_2\text{SO}_4 + \text{H}_2\text{S}$. This view was confirmed by determining the weight of sulphur formed. The residue in the reaction tube contained no sulphite, though air had been entirely excluded. The reaction $3\text{SO}_2 = 2\text{SO}_3 + \text{S}$, which might be supposed to occur and supply another explanation of the origin of the sulphur, is very unlikely under the conditions of working.

C. IRWIN.

Ratio of fluorine to phosphoric acid in phosphate rock. D. S. REYNOLDS, K. D. JACOB, and W. L. HILL (Ind. Eng. Chem., 1929, 21, 1253—1256).—Most American phosphate rocks contain 3—4% F. Florida pebble phosphate has an almost constant content of 3.9% irrespective of the phosphoric acid content. In other types (Florida hardrock, phosphates from Tennessee and Idaho) the fluorine content varies directly with the phosphoric acid, the ratio being constant for each type. The fluorine is present in the form of complex fluorophosphates, perhaps formed by reaction between phosphates and fluorides in solution. It tends to be higher in the older rocks.

C. IRWIN.

Manufacture of high-analysis phosphates. E. L. LARISON (Ind. Eng. Chem., 1929, 21, 1172—1175).—Phosphate rock as mined in Idaho contains about 6% of moisture. After crushing and drying it contains 32% P_2O_5 , together with 7% of organic matter, which is removed by calcining. It is then ground and agitated by compressed air with sulphuric acid in a continuous plant consisting of three large tanks. It then passes through Dorr thickeners and Oliver filters. Wood, hard lead, and alloys resistant to phosphoric acid are amongst the constructional materials used. The phosphoric acid is concentrated up to 44% P_2O_5 in vacuum evaporators using steam raised by the gases from reverberatory furnaces. The phosphoric acid is mixed with rock phosphate to form "treble superphosphate," the acid concentration used being the highest with which it is possible to obtain a uniform product. The product is aged for 30 days and then dried at below 200°. A certain amount of ammonium dihydrogen phosphate is also made by mixing phosphoric acid and ammoniacal liquor.

C. IRWIN.

Fertilisers. MATIGNON.—See XVI. **Sodium citrate.** CATULLO.—See XX.

PATENTS.

Process for the manufacture of alkali iodates and its application to the manufacture of oxygen. I. G. FARBENIND. A.-G. (B.P. 299,445, 16.8.28. Ger., 28.10.27. Addn. to B.P. 290,749; B., 1928, 523).—The reaction described in the prior patent is accelerated, even at ordinary pressure, by adding to the melt heavy-metal or earth oxides, e.g., 1% of bismuth oxide

or 4% of aluminium hydroxide. By raising the temperature of the melt to 575—600° oxygen is evolved, whilst the regenerated iodide can be used again. Gases containing oxygen may be used instead of oxygen.

P. E. L. FARINA.

Manufacture of [dehydrogenating] catalysts [hydrated copper oxide]. J. P. P. MAYOR, Assec. of Soc. ALSACIENNE DE PROD. CHIM. (B.P. 303,347, 13.8.28. Fr., 31.12.27).—Precipitated copper hydroxide or carbonate is heated at about 90° in the presence of the mother-liquor until the desired hydrate (e.g., $n\text{CuO} \cdot \text{H}_2\text{O}$; $n = 4$ or 6) is obtained, when the precipitate is stabilised, removed, washed, and dried *in vacuo*. The stabilising agents comprise alkaline-earth hydroxides which may be used in excess as the precipitants or may be added after precipitation, and soluble compounds, e.g., sugar or alkali salts, applied by washing the precipitate with solutions containing them and drying the impregnated hydrate.

L. A. COLES.

Production of anhydrous stannic chloride from metals [alloys] containing tin. G. A. FAVRE (B.P. 304,282, 7.1.29. Switz., 18.1.28).—The alloys are treated with chlorine at about 900° in a melting pot provided with inlets for the alloy and chlorine, with heating and cooling means, with a perforated annular device above the level of the molten metal for collecting the least volatile (lead and copper) chlorides as a liquid, and with a baffle plate below a vapour outlet above the pot. The escaping vapours pass through one or more coolers in which iron, aluminium, and antimony chloride etc. condense, and which may contain material to combine with excess chlorine, and thence into a condenser for the residual stannic chloride vapour.

L. A. COLES.

Manufacture of white titanic acid. I. G. FARBENIND. A.-G. (B.P. 296,730, 5.9.28. Ger., 6.9.27).—The ore, worked up by treatment with sulphuric acid, is hydrolysed in the presence of hydrofluoric acid or a soluble fluoride, water being added to prevent concentration of the free sulphuric acid formed; 89% of the titanic acid separates, after 6 hrs.' boiling, in a granular condition, which is easy to filter and remains pure white even after ignition at 900°.

P. E. L. FARINA.

Making base-exchange materials. A. S. BEHRMAN, Assr. to PERMUTIT Co. (U.S.P. 1,736,281, 19.11.29. Appl., 29.12.24).—Solutions containing about 31 mols. of sodium silicate and 8—10 mols. of basic aluminium sulphate are mixed, the concentrations being such that a gel is formed; the reaction mass is then dried and leached.

P. E. L. FARINA.

Manufacture of nitrous oxide. W. FRIEDERICH (B.P. 310,507, 15.2.29. Ger., 27.4.28).—A purer product with a yield 96—98% of the theoretical is obtained by adding to the ammonium nitrate to be decomposed 0.7—1.4% of ammonia, either in gaseous form or to a solution of the nitrate. In a continuous process an aqueous solution of ammonium nitrate is used, this being run into a reaction vessel made of suitable acid-resisting materials, which is heated to 200—240° and preferably contains a mixture of sodium and potassium nitrates.

P. E. L. FARINA.

Stabilising and improving the base-exchanging properties of silicates. A. ROSENHEIM (B.P. 302,690, 19.12.28. Ger., 20.12.27).—Glaucanite or material containing or resembling it is treated successively at the ordinary temperature or above with solutions of acid-reacting salts of metals which are at least bivalent (preferably trivalent, amphoteric metals, *e.g.*, ferric chloride), with solutions of alkali salts having an alkaline reaction (sodium silicate), and with a neutral alkali salt (sodium chloride) solution. The first two treatments may be repeated alternately until the water to be softened no longer becomes turbid when passed through the treated material, and the material may be washed with water between the different treatments.

L. A. COLES.

Recovery of sulphur from gaseous mixtures containing sulphur vapours. H. D. ELKINGTON. From COMSTOCK & WESCOTT INC. (B.P. 321,919, 17.5.28).—Mixtures of sulphur vapour with non-reacting gases, other than the mixtures claimed in B.P. 306,107 and 321,911 (B., 1930, 106), are brought in contact with solid condensing elements maintained at a temperature such that the condensed sulphur flows off in a freely liquid condition; *e.g.*, the gases are passed through the tubes of a tubular boiler at a temperature corresponding to a steam pressure of 2 to just under 6 atm., the tubes being provided with baffles to ensure good contact between the vapour and the condensing surface.

L. A. COLES.

Concentration and distillation of solutions of hydrogen peroxide. R. WIETZEL, L. SCHLECHT, and O. KÖHLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,738,625; 10.12.29. Appl., 18.1.27. Ger., 18.1.26).—See B.P. 264,535; B., 1928, 604.

By-products from gases (B.P. 321,982).—See II. **Sulphur and iron oxide from ores** (B.P. 321,911 and 321,920).—See X. **Electrolytic lead peroxide** (B.P. 299,306).—See XI. **Vermilion** (G.P. 453,523).—See XIII.

VIII.—GLASS; CERAMICS.

Continuous process for plate glass at the Ford River Rouge plant. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 1168—1171).—The continuous plant described was started by the Ford Motor Co. in 1923. Tank furnaces are used from which the glass flows to rolls. The furnaces are gas-fired and pyrometrically controlled, and have a capacity of 32 tons per 24 hrs. and a life of 11—12 months. The rolls, which are of cast iron and water-cooled, gradually become eroded. The annealing lehrs are also gas-operated. The processes of grinding and polishing are also described.

C. IRWIN.

Ultra-violet solar radiation and the solarisation of window materials. W. W. COBLENTZ and R. STAIR (Bur. Stand. J. Res., 1929, 3, 629—689).—Data are given on the variation of ultra-violet solar radiation with the time of day, season of the year, altitude, and geographical position, and pollution of the atmosphere. The amount of therapeutically useful ultra-violet radiation obstructed by common window glass, and by a number of the new glasses for transmitting short-wave radiation, is measured. These glasses are classified according to their transmissive properties at 302 mμ,

and vary from 25% to 90% as compared with 1% for common window glass.

C. J. SMITHELLS.

PATENTS.

Strengthened glass. P. H. HEAD (B.P. 321,977, 23.8.28).—Ethyl lactate is employed, alone or in combination with other solvents, to seal glass to celluloid or cellulose acetate. Edges are sealed by bevelling in V-shape and either cauterising or filling with oleaginous material. Alternatively, addition of small proportions of methylcyclohexanone or triacetin to the ethyl lactate renders it unnecessary to seal the edges of the sheets.

A. COUSEN.

Sterilisation of glass tubes and glass vessels. J. DICHTER (B.P. 305,185, 1.2.29. Ger., 1.2.28).—Glass tubing is maintained clean and sterile by closing the ends by fusion during the process of manufacture.

A. COUSEN.

Baking a highly refractory lining in metallurgical furnaces, especially induction furnaces, crucibles, and the like. S. WESTBERG (B.P. 310,458, 20.8.28. Ger., 26.4.28).—The refractory lining or crucible is placed in an induction furnace and a plug of conducting material is suspended within it. The plug is heated by induction and serves to heat by radiation the crucible to be baked. The refractory can thus be fired out of contact with corrosive slags etc. to a temperature above that at which it will be used. If the plug be made of a material having a sufficiently high m.p., the inner surface of the lining or crucible may be fused and will give, on solidification, a surface layer very resistant to gas, metal, or slag.

J. A. SUGDEN.

Manufacture of abrasive materials having an alumina base. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 292,611, 22.6.28. Fr., 23.6.27).—The admixture of 2% of fluorides or fluosilicates (preferably of the alkalis or alkaline earths) to ground bauxite enables the sintering and recrystallisation of the alumina to take place at 1300—1350°.

J. A. SUGDEN.

Method and apparatus for producing sheet glass. A. E. WHITE. From MISSISSIPPI GLASS CO. (B.P. 322,620, 13.11.28).

Manufacture of [multi]-coloured patterned glass and other transparencies or translucencies. B. R. BAYNE (B.P. 322,075, 9.11.28).

Glass substitute (B.P. 308,587).—See V.

IX.—BUILDING MATERIALS.

Setting and hardening of Portland cement in the presence of water-soluble compounds. H. W. GONELL (Z. angew. Chem., 1929, 42, 1087—1091).—The effect of the addition of various water-soluble compounds on the setting of Portland cement was studied by the microscopical examination of thin sections and of preparations of the finest grains of the cement (diam. = 10—40 μ) mounted in the solution on a slide. The former were much more difficult to study than natural rock slices, and clearer results were obtained with the powder. With sugar solution crystal formation was retarded and gel formation predominated.

Crystallisation may be evident only after months. Calcium saccharate has a similar effect, so that the result is not due to the solubility of calcium oxide in sugar solution alone. It is known that sugar acts as a stabiliser to many emulsions. Copper chloride and lead nitrate were found to lengthen to 7 days the time required for crystals to develop. Sodium carbonate was found to promote crystallisation, but the inferior final strength with its use is probably due to the suppression of the gel phase. Both gel and (later) crystal formation are essential to good setting. C. IRWIN.

Action of sulphates on Portland cement. I. Use of the expansion method in studying their action on Portland cement mortar and concrete. T. THORVALDSON, D. WOLOCHOW, and V. A. VIGFUSSON (Canad. J. Res., 1929, 1, 273—284).—To investigate the reproducibility of expansion tests, mortar bars 0.625 in. thick and 7.5 in. or 10 cm. long were made in different batches from Portland cement and standard sand (1:7.5 mix) with smooth ends of neat cement and cured for 7 days in moulds, followed by curing periods of 14 days, 1 month, or 3 months in distilled water or a damp closet. The times taken by the bars to give a certain percentage expansion when immersed in 0.15*M*-sodium sulphate showed definite fluctuations amongst batches of the same age, but the variations were slight compared with those shown by cements of different origin. Tensile tests on briquettes (1:5 mix) during immersion in 0.5*M*-sodium sulphate and -magnesium sulphate showed that a rapid rise in strength occurs during the first 2 days, followed by a period of no change and then by a rapid decrease. The expansion-strength curve is not linear, the tensile strength diminishing at a decreasing rate with respect to expansion. By determining the time required to attain a given expansion with 1:10, 1:5, 1:3, and 1:2 mixes of cement of different grades and standard sand it was shown that resistance to sulphate action increases rapidly with richness of mix and the superiority of good-quality cement is the more evident the richer is the mix. This increase in resistance with richness of mortar is more pronounced when graded sand is used.

H. J. DOWDEN.

Substitute for the compression test of concrete. G. J. GRIESENHAUER (Eng. News-Rec., 1929, 103, 846—847).—The cement, sand, and gravel separated from a weighed quantity of freshly made concrete by washing and sieving are dried and weighed, so that a check is obtained on the proportions used without waiting for the aggregate to set.

S. I. LEVY.

Utilisation of low-grade timber and waste wood. C. G. SCHWALBE (Z. angew. Chem., 1929, 42, 1118—1121).—A résumé of recent developments in the technology of wood and timber.

S. I. LEVY.

Primary tars. MENEGHINI. Road tar. MALLISON. —See II.

PATENTS.

Coloured bituminous compositions. C. H. GRIMSHAW (B.P. 321,948, 22.6.28).—A bituminous emulsion is mixed with clay and a colouring matter which may be a synthetic dye. The admixture is made while the constituents are cold. C. A. KING.

Treatment of wood and fibres. G. B. RICE, Assr. to CONSERVATION CORP. OF AMERICA (U.S.P. 1,732,379, 22.10.29. Appl., 3.3.26).—Wood is cleansed and stabilised by immersion in a solution containing a compound which exerts osmotic pressure in solution, *e.g.*, sugar, and a boron compound, *e.g.*, boric acid, which acts as a catalyst. The bath is slowly heated and boiled until no further scum is produced. F. G. CLARKE.

Manufacture of [fused] cements. A. BAUCHÈRE and G. ARNOU (U.S.P. 1,739,383, 10.12.29. Appl., 10.11.24. Fr., 4.12.23).—See B.P. 225,858; B., 1926, 159.

[Bitumen-coated] concrete. M. L. GLOVER (B.P. 321,976, 23.8.28).

Mixing machine (321,899).—See I. Dispersions of pitch, bitumen, etc. (B.P. 321,721)—See II. Treatment of wood etc. (B.P. 321,786).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Testing of sheet metal for deep drawing. M. SCHMIDT (Arch. Eisenhüttenw., 1929—1930, 3, 213—222; Stahl u. Eisen, 1929, 49, 1695—1696).—If a sheet of metal is placed on the stage of a 10-ton Amsler machine and a small plunger forced into it, then the curve showing the load against the depth of impression is found to rise to a maximum, then fall again. This maximum is a logarithmic function of the diameter of the plunger and also varies with the thickness of the sheet, but it never exceeds the breaking strength of the metal. The limiting case in which the drawing power is equal to the breaking strength is called the "drawing limit." By determining the maximum drawing power with two different plungers and the breaking strength on a sheet of known thickness, the drawing limit for any size of plunger may be calculated. The results obtained approximate closely to those obtained in actual commercial work, and give a far better indication of the behaviour of a metal in deep drawing than does the Erichsen test. Tests on cold-rolled iron sheets before and after annealing at various temperatures showed that a minimum drawing limit is obtained with annealed sheet having a coarse-grained structure. The effect of repeated drawing with plungers of decreasing diameter is not identical with that produced by a single drawing up to the limit, and hence it is concluded that cold plasticity and capacity for deep drawing are not interchangeable terms for the same property of a metal, but are closely related properties. A. R. POWELL.

Recent developments in corrosion-resistant and heat-resistant steels. J. A. MATHEWS (Ind. Eng. Chem., 1929, 21, 1158—1164).—It is recommended that all corrosion data should be expressed in depth of penetration in unit time. The properties of stainless steel (12% Cr), the stainless irons (low-carbon iron-chromium alloys), and the austenitic nickel-chromium steels also containing up to 5% Si are described. In considering the application of these it must be remembered that their resistances to corrosives may be either increased or decreased by impurities in the latter. Steels containing 10—12% Cr, 25% or 34% Ni, and 5% Si show good resistance to sulphuric acid, and are far

superior in this respect to those of older types. The scale of iron oxide formed by long exposure to high temperatures is in the case of these alloys formed in layers of which only the innermost contains more than traces of nickel or chromium. Tests of the time required to develop visible scale on various alloy steels are described. The high-chromium steels are very suitable in this respect, but when good physical properties at a high temperature are necessary the nickel-chromium-silicon steels are preferable. These can be satisfactorily welded, and have many applications in furnace work in the steel industry, in oil-cracking, etc. C. IRWIN.

Eutectoid transformation in relation to the mechanism of quenching and tempering aluminium bronze. I. OBINATA (Mem. Ryojun Coll. Eng., 1929, 2, 205—225).—The eutectoid transformation in copper-aluminium alloys containing 9.5—16% Al is investigated, and found to be a stepped transformation of the type $\alpha + \delta \rightleftharpoons \beta' \rightleftharpoons \beta$. With rapid cooling the change $\beta' \rightarrow \alpha + \delta$ is suppressed, and the mechanism of tempering is explained by the tendency for this change to occur. A homogeneous structure, β' , corresponding to austenite, can be obtained by quenching the alloy in toluene at -90° . C. J. SMITHELLS.

Alteration in mechanical properties of duralumin plates after corrosion by sea-water. E. HERZOG and G. CHAUDRON (Compt. rend., 1929, 189, 1087—1089).—Corrosion of duralumin plates 2 mm. thick by aerated sea-water for one month or by sea-water containing 0.3% of hydrogen peroxide or oxygen under 25 kg. pressure for 48 hrs. produces equal losses in weight (1—2%). The extensibility is decreased, but the breaking load is unaltered unless thinner plates are used, when this also is decreased. These results may be reproduced if small, shallow holes are bored in the original plates. The mechanical properties may be restored by polishing. J. GRANT.

Thermal and elastic properties of elinvar. F. J. SCRASE (J. Sci. Instr., 1929, 6, 385—392).—The temperature coefficient of the elastic constant of an elinvar spring has been found to be about one tenth that of a steel spring. C. W. GIBBY.

Wet method of treating mixed minerals [lead-zinc ores]. A. GLAZUNOV (Chim. et Ind., 1929, 22, 890—892).—The ore in question contained 4.0% Zn, 3.5% Pb, 0.3% Cu, with traces of silver and gold. It is proposed to heat it with 8% of sodium chloride at 500° and extract with dilute acid, bringing copper, zinc, and iron into solution. Precipitation of the copper is by means of scrap iron and of the iron and zinc by lime. The latter precipitate is to be treated in a Wetherill furnace to give zinc white. Gold and silver are extracted with cyanide from the residue from the acid treatment, after neutralisation, and the lead sulphate in the remainder is concentrated by flotation methods and smelted. A laboratory trial has given good yields (70% and upwards) by this procedure. C. IRWIN.

Effect of atmospheres on the heat treatment of metals. E. G. DE CORIOLIS and R. J. COWAN (Ind. Eng. Chem., 1929, 21, 1164—1168).—The definition of atmospheres as oxidising, neutral, or reducing is

unsatisfactory inasmuch as carbon dioxide or water vapour is capable of oxidising steel at high temperatures. The loss from this cause in the forging of steel and the subsequent pickling is a very serious one. It is shown that scale formation in forging is reduced with increase of carbon monoxide content in the atmosphere, and a gas-fired furnace which would maintain this condition together with the requisite temperature would offer a solution. Scaling during carbonising and reheating may be avoided by working in a stream of unburnt town's gas within a muffle. The difficulty of bright annealing, especially of brass, in an atmosphere of nitrogen is possibly due to the occlusion of gases such as oxygen at the metal surface. Hydrogen can be used for the annealing of nickel but not of copper, the latter being embrittled by the reduction of copper oxide. A successful bright-annealing process should also provide for the preliminary oxidation of any lubricant present. C. IRWIN.

Metallographic polishing. I. Automatic metallographic polishing machine. S. EPSTEIN and J. P. BUCKLEY (Bur. Stand. J. Res., 1929, 3, 783—794).—A description is given of such a machine designed to give reproducible results in a projected study of polishing methods. C. J. SMITHELLS.

Nickel in caustic soda evaporation. MCKAY.—See VII. **Resistance furnace for iron etc.** PASCHIKIS.—See XI. **Enzymes as reagents.** BARGELLINI.—See XVIII.

PATENTS.

Production of steel in hearth furnaces. J. GRYZ (G.P. 455,063, 12.7.23).—The charge is melted in a hearth furnace and the molten iron tapped into a second hearth furnace for conversion into finished steel. To keep the first furnace continually charged the materials are fed into it through a chamber heated by the waste gases, the scrap iron being packed in a wooden frame, which acts as a source of carbon. A. R. POWELL.

Case-hardening agent for iron and steel. J. REICH (Swiss P. 122,402, 2.7.26).—The damp articles are sprinkled with a mixture of powdered sodium chloride, potassium ferrocyanide, wheat flour, and crushed hooves of cattle, heated slowly to cherry redness, and quenched. A. R. POWELL.

Ferro-aluminium alloys. S. G. S. DICKER. From AUBERT & DUVAL FRÈRES (B.P. 321,936, 14.8.28).—Aluminium steels are produced by adding to molten steel an alloy of iron and aluminium containing about 60% Al. The alloy is made by first deoxidising iron with aluminium and then adding the required quantity to produce the alloy. C. A. KING.

Manufacture of alloys of iron and aluminium. C. P., O. F. A., and N. P. P. SANDBERG, and J. W. BAMPFYLD (B.P. 322,446, 3.9.28).—The alloys contain 11—16% Al and 89—84% Fe with or without the addition of 0.5% Si. They are cast at 1600° , and are said to be workable and highly resistant to corrosion. A. R. POWELL.

Manufacture of iron-chromium alloys. H. G. FLODIN (B.P. 322,189, 28.8.28).—A shaft furnace is charged with iron ore briquettes of such a composition

as to produce an iron of low carbon content; as the charge melts down it is covered with alternate layers of lime and with briquettes composed of chromite and ferrosilicon to give an alloy of the desired composition. The lime and chromite remain suspended in the slag first formed until the last-named is reduced to ferro-chromium, which then flows through the slag into the bath of molten iron. The iron ore briquettes are preferably reduced in a separate shaft by means of the gases evolved from the main shaft. A. R. POWELL.

[Iron-copper] alloys [containing chromium and/or nickel]. W. P. DIGBY (B.P. 322,216—7, 30.8.28).—(A) The alloy contains 20.5—34.5% Fe, 21—50% Ni, and the remainder copper; up to 20% of the iron may be replaced by an equal quantity of chromium and small quantities of manganese may be added as a deoxidiser. (B) The alloy is melted in a gas-fired crucible, which is then transferred to an induction furnace to ensure thorough mixing of the constituents. A. R. POWELL.

Mixing apparatus [for alloys]. G. A. SEELKY, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,734,747, 5.11.29. Appl., 24.7.28).—A bath of molten metal is caused to circulate through a vertical, submerged casing and back through the main bath. A hood, partly submerged, surrounds the casing and encloses a minor part of the surface of the bath. The alloying metal is admitted within the hood, which serves to prevent access of dross from the large part of the bath. A. R. POWELL.

Separation and refining of metal mixtures. R. LORENZ and W. FRAENKEL (G.P. 453,592, 9.8.24).—The alloy is melted under a salt layer containing the oxide or sulphide of one of the constituents, and the equilibrium is disturbed by passing chlorine into the salt layer to volatilise the chloride of one of the metals; a corresponding quantity of this metal is then dissolved out of the alloy by the slag. The chlorine may be generated by adding a chloride to the salt layer and electrolysis, using the molten alloy as cathode. The process is suitable for the separation of tin from lead-tin alloys. A. R. POWELL.

Concentration of copper ores. S. TUCKER (B.P. 322,527, 7.9.28).—The ore is heat-treated to reduce its copper content to metal as described in B.P. 250,991 (B., 1926, 590), and the treated ore is subjected to froth flotation in a pulp containing an alkali cyanide, sodium silicate, and a derivative of xanthic acid, together with the usual oils. A. R. POWELL.

Binding of fine ores, burnt pyrites, and the like. M. PASCHKE and E. SCHIEGRIES (G.P. 454,866, 17.8.26. Addn. to G.P. 453,469; B., 1929, 249).—Preheated air or oxygen is blown through the chamber in which the ore is mixed with slag as described in the prior patent. A. R. POWELL.

Treatment of iron [sulphide] ores. COMSTOCK & WESCOTT, INC., Assees. of E. W. WESCOTT (B.P. 306,107, 17.5.28. U.S., 16.2.28).—Iron sulphide concentrates are heated with dilute chlorine to form ferrous chloride and sulphur, which is collected in a suitable receiver. The ferrous chloride is then further chlorinated to form ferric chloride, which is vaporised and burned to

obtain ferric oxide and a mixture of chlorine and nitrogen. Two thirds of this gas mixture is used in the first stage of the process and one third in the second, whereby the process is made cyclic without the accumulation of nitrogen in the circulating gases. A. R. POWELL.

Recovering sulphur and iron oxide from iron sulphide ores or the like. H. D. ELKINGTON. From COMSTOCK & WESCOTT, INC. (B.P. 321,911 and 321,920, 17.5.28).—(A) Iron sulphide ores or mattes are heated at 325—400° in an atmosphere containing chlorine, whereby ferrous chloride is formed and sulphur volatilised. The issuing gases are passed into a condensing vessel containing steam pipes through which steam is passed at 2—6 atm. pressure so as to condense the sulphur to a mobile liquid. The ferrous chloride is then chlorinated further to produce ferric chloride, which is volatilised and burnt with the aid of producer gas to regenerate chlorine and form a coarse-grained ferric oxide. (B) In the process described above, the regenerated chlorine is used to chlorinate fresh quantities of ore, the air used to burn the ferric chloride is preheated by the waste gases, and the residue from the final chlorinating vessel is treated for the recovery of copper, zinc, and/or nickel chlorides. (Cf. B.P. 306,107, preceding.) A. R. POWELL.

Treatment of ores. S. G. S. DICKER. From BRADLEY-FITCH Co. (B.P. 321,943, 19.6.28. Cf. B.P. 319,392; B., 1929, 985).—Before leaching mixed manganese and iron ores with a solution of ammonium sulphate, the ore is heated in a reducing atmosphere and in the presence of water vapour within the range 450—850°, the lower temperature yielding an insoluble and magnetic iron oxide. The heating of the ore, reducing atmosphere, and water vapour required for the process are derived from the partial combustion of a combustible gas. C. A. KING.

Treatment of complex ores. GELSENKIRCHENER BERGWERKS A.-G., G. VERVUERT, and G. RIEBER (N.P. 42,997, 14.1.25).—The ore is mixed with a reducing agent and the mixture blown on a water- or air-cooled grate with a blast of hot air from tuyères situated above the grate. Volatile metals are thus expelled and can subsequently be collected as oxides, whilst copper, iron, and manganese collect as an alloy below the grate. A. R. POWELL.

Reduction of metal oxides. H. G. FLODIN (N.P. 42,996, 22.4.24).—Metal oxides which are reduced by carbon only above 1200° are mixed with the oxide of a volatile metal, e.g., zinc, and carbon and the mixture is heated to effect reduction of the added oxide, the resulting metal then acting as a reducing agent for the refractory oxide. A. R. POWELL.

Electrothermal production of zinc. FASTIGKETS-AKTIEBOLAGET ØRESUND (N.P. 43,049, 16.9.21).—The mixture of oxide and carbon is passed through a vertical cylindrical preheater provided with an axial screw or spiral conveyer and having its cross-section alternately constricted and opened out. A current of hot carbon monoxide from the later stages of the process is passed upwards through the ore to preheat and partially reduce it. A. R. POWELL.

Condensation of zinc vapours. A./S. MALMIN-DUSTRI (N.P. 43,232, 29.11.24).—The cooling medium

used is the vapour of a substance which condenses at temperature below the b.p. of zinc. A. R. POWELL.

Recovery of silver or silver and lead together from pure oxides. SOC. METAL. CHILENA "CUPRUM" (G.P. 447,686, 12.5.23).—The material, *e.g.*, litharge, red lead, or lead peroxide, is digested with a solution of sodium chloride to convert the lead and silver into soluble chlorine compounds which can be further treated in any suitable manner. A. R. POWELL.

Protection of metallic surfaces from corrosion. S. O. COWPER-COLES, L. TAYLOR, A. A. GOULD, and P. G. LUCAS (B.P. 322,296, 26.10.28. Addn. to B.P. 308,180; B., 1929, 523).—The sherardising operation claimed in the prior patent is carried out in a revolving, perforated drum surrounded by an external casing through which carbon dioxide is passed. A. R. POWELL.

Treatment of metal to remove scale. C. FISCHER, JUN., and L. A. STEGEMEYER, Assrs. to TWITCHELL PROCESS Co. (U.S.P. 1,736,282, 19.11.29. Appl., 25.5.28).—The sulphonated mineral oil product, recovered from the refining of such oils, is introduced into the mineral acid bath to inhibit the action of the acid on the metal. P. E. L. FARINA.

Electrolytic removal of metal from metallic bodies. S. O. COWPER-COLES (B.P. 322,118, 5.6.28).—The fin or splay may be removed from the eyes of needles after stamping by passing them through abrasive material, such as sand or pumice powder, at the bottom of an electrolytic bath in such a manner that the unwanted projections are dissolved away by anodic action and the scouring effect of the abrasive material. A. R. POWELL.

Rapidly producing uniform metal [*e.g.*, copper] deposits electrolytically. K. BREUSING and U. GOTTESMANN (B.P. 322,371, 28.12.28).—Electrolysis is effected in heated baths containing colloids and the bath is kept stirred by forcing air into the bottom through a porous clay diaphragm so that it ascends through the bath in fine streams. Good deposits of copper are thus obtained from acid sulphate baths with current densities up to 40 amp./dm.² A. R. POWELL.

Production of smooth and dense electrolytic metallic deposits. METALLGES. A.-G. (B.P. 299,725, 29.10.28. Ger., 27.10.27).—A colloid, *e.g.*, silicic acid, is either produced in the electrolytic bath by reaction between suitable substances introduced for that purpose, or is added directly to the bath. Thus an alkali silicate and sulphuric acid may be added to the bath employed for the electrolytic refining of copper, or zinc silicate for the electrolysis of zinc sulphate solutions. In all cases the added substances must be such that no appreciable quantities of foreign ions having a disturbing influence on the electrolysis are produced. J. S. G. THOMAS.

Enrichment of iron ore, blast-furnace flue dust, burnt pyrite, purple ore, and the like. H. A. MUELLER, Assr. to AKTIEB. FERRICONCENTRAT (U.S.P. 1,738,603, 10.12.29. Appl., 22.8.27. Swed., 28.10.26).—See B.P. 279,797; B., 1928, 715.

Manufacture of metal filaments. R. JACOBY and F. KOREF, Assrs. to GEN. ELECTRIC Co. (U.S.P.

1,739,234, 10.12.29. Appl., 1.6.22. Ger., 18.6.21).—See G.P. 371,623; B., 1923, 706 A.

Washing of ores etc. (B.P. 294,210).—See I. **Treatment of tin alloys** (B.P. 304,282).—See VII. **Furnace linings** (B.P. 310,458).—See VIII. **Magnetic alloys** (B.P. 321,957).—See XI. **Caoutchouc-coated metal** (B.P. 302,250).—See XIV.

XI.—ELECTROTECHNICS.

Electric resistance furnaces for the iron industry. V. PASCHKIS (Stahl u. Eisen, 1929, 49, 1685—1691).—An illustrated article describing modern types of electric furnace for the bright annealing of iron and steel. A. R. POWELL.

Construction of electric resistance furnaces. E. SCHWARZ-BERGMANN (Chem. Fabr., 1929, 519—520).—Data are given for furnace construction using chromium-nickel (chronin) wire. S. I. LEVY.

Electrolysis of water at high pressure. G. FAUSER (Giorn. Chim. Ind. Appl., 1929, 11, 479—487).—Theoretical considerations show that increase in the pressure is accompanied by increase in the amount by which the increment of the potential necessary to decompose water exceeds the ideal value of such increment. As regards the overvoltage, it is difficult to see how this can be diminished by effecting the electrolysis at a high pressure, since the gaseous layers surrounding the electrodes have then a higher density than at atmospheric pressure, and should thus offer increased resistance to the flow of the current. Further, any considerable influence of the pressure on the ohmic resistance of the electrolyte is excluded. Hence, of the various factors which intervene to modify the *P.D.* necessary for the electrolysis of water at high pressure, those increasing this *P.D.* prevail, so that there should be a global increment of the *P.D.* and of the energy required. It seems, therefore, that the saving in energy noted by various authors must be only apparent, and that the observed lowering of the *E.M.F.* must be counterbalanced by a smaller production of gas. Although the amounts of hydrogen and oxygen developed at the electrodes by the flow of a definite quantity of current are usually regarded as invariable, it is pointed out that the application of this law is limited in practice by secondary reactions between the elements separated. That nascent hydrogen and oxygen are able to react at the ordinary temperature is shown by the non-production of gas when water is electrolysed by a symmetrical alternating current. These conclusions are supported by the experimental results obtained on electrolysing water at pressures up to 1500 atm., these showing diminution in the current gas-yield with increase of pressure; hence the accompanying depression of the *P.D.* is to be ascribed to depolarisation phenomena. The author considers the practical and economic difficulties involved in the electrolysis of water at high pressures to be virtually insurmountable. T. H. POPE.

Treatment of cellulose. STRACHAN.—See V.

PATENTS.

Ironless induction furnace. HIRSCH, KUPFER- u. MESSING-WERKE A.-G. (B.P. 307,044, 6.9.28. Ger.,

2.3.28).—The inductor windings are divided into two separate coils energised from different phases of a low-frequency, polyphase current, and surrounding, respectively, the upper and lower parts of the furnace. Rings of insulated sheet iron separated by an air space or non-magnetic material are arranged between the coils.

J. S. G. THOMAS.

Electric [muffle] furnace. F. B. WOOLFORD (B.P. 322,367, 24.12.28).—Resistors are wound on or supported by silica pins or rods in a fireclay muffle furnace.

J. S. G. THOMAS.

[Electrodes for] electric furnaces. A. E. WHITE. From J. J. NAUGLE (B.P. 322,160, 25.7.28).—Fluid is discharged through apertures in a rotating disc-like electrode member, so that material being treated in the furnace is stirred.

J. S. G. THOMAS.

Soluble metallic electrode for galvanic cells. J. MOHR (G.P. 453,763, 6.10.26).—The soluble metal, e.g., zinc, is deposited upon a more electronegative metal, e.g., aluminium, so that on bringing this electrode in contact with electrolyte and passing a current an insoluble, transition resistance layer is formed on the electrode.

J. S. G. THOMAS.

Photo-electric cells. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of K. T. BAINBRIDGE (B.P. 303,476, 13.12.28. U.S., 4.1.28).—The inner wall of an evacuated vessel is coated with silver which is oxidised or provided with an adsorbed gas layer. Cæsium or other alkali metal is applied to the coating, and excess alkali removed by heating *in vacuo* at a high temperature.

J. S. G. THOMAS.

Treatment of gases and vapours with electric arc discharge. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,471, 29.8.28).—Gas or vapour to be treated is admitted through jets surrounding the electrodes or through the electrodes themselves, and is submitted to the action of parallel arc discharge in gaps in which the pressure of the gas or vapour is at least 100 mm. Hg, being higher in some parts, preferably at the electrode points, than at others.

J. S. G. THOMAS.

Apparatus for electro-osmotic separation from solutions of anodic or cathodic travelling particles. SIEMENS-ELEKTRO-OSMOSE GES.M.B.H. (B.P. 307,879, 2.3.29. Ger., 15.3.28).—Solution to be treated and one electrode are placed in a space which is separated, by a porous diaphragm impenetrable to the particles to be removed, from a second space containing the other electrode and a liquid which is a poor conductor of electricity.

J. S. G. THOMAS.

Separation of visible rays from ultra-violet rays by filtration. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,300, 20.7.28).—Radiation is passed through transparent sheets of cellulose or its derivatives or of condensation products of urea and formaldehyde stained with organic or inorganic colouring matters, e.g., fuchsin, crystal-violet.

J. S. G. THOMAS.

Apparatus for influencing substances by means of high-frequency electric energy. A. ESAU, and C. LORENZ A.-G. (B.P. 301,929 and 301,930, 7.12.28. Ger., 8.12.27).—(A) Substances are subjected to the

action of ultra-short electromagnetic waves of wavelength 10 m. and less. As a practical possibility the removal of dust from gases is cited. (B) Material is subjected to the process claimed in (A) between, but out of contact with, the plates of a condenser supplied with electrical energy of frequency of at least 3×10^7 periods/sec.

J. S. G. THOMAS.

Electrolytic production of protective coatings of lead peroxide on electrical conductors. SIEMENS & HALSKE A.-G. (B.P. 299,306, 10.10.28. Ger., 22.10.27).—Lead peroxide is deposited electrolytically upon anodes in an alkaline solution of a lead salt to which is added an organic compound of one of the following classes: (a) hydrocarbon compounds containing hydroxyl groups, (b) organic acids without hydroxyl groups, (c) carbon-nitrogen compounds, e.g., cyanides, thiocyanates, oximes.

J. S. G. THOMAS.

Electrical insulating materials. W. S. SMITH, H. J. GARNETT, J. N. DEAN, H. C. CHANNON, W. GARDNER, and H. F. WILSON (B.P. 322,208, 25.8., 11.10., and 14.11.28. Cf. B.P. 307,390; B., 1929, 400).—Thermoplastic cable insulation is composed of 30–60% of rubber, freed from resins and/or water-soluble or -insoluble compounds, mixed with bitumen (having neither free carbon nor ash content above 2%, and m.p. 100° or over). If desired, 10% of a flux, e.g., ceresin or other wax, may be substituted for bitumen to reduce the temperature at which the latter becomes sufficiently fluid for working. The rubber after being partially vulcanised at below the m.p. of the bituminous filler is then completely vulcanised at above 185° after addition of the powdered bitumen.

J. S. G. THOMAS.

Production of magnetic materials [brittle magnetic alloys]. STANDARD TELEPHONES & CABLES, LTD. From WESTERN ELECTRIC CO., INC. (B.P. 321,957, 22.8.28).—A mixture of nickel and iron, preferably containing more than 25% of nickel, melted in the presence of oxygen and an oxidising agent, e.g., iron oxide, is boiled until oxidised, poured into a mould, and allowed to solidify. The material is then rolled at a temperature above the recrystallisation temperature but not lower than the temperature at which it ceases to be malleable, and finally powdered.

J. S. G. THOMAS.

Rod for use in arc-welding or cutting. J. B. GREEN (U.S.P. 1,733,795, 29.10.29. Appl., 10.2.27).—By coating the rod with sufficient alumina, or compound thereof, alumina is introduced into the arc, and the voltage drop, with a normal welding length, is thereby raised.

F. G. CLARKE.

[Multi-layer] insulated [and fireproof electrical] conductors. ROCKBESTOS PRODUCTS CORP., Assees. of H. O. ANDERSON (B.P. 313,909, 18.10.28. U.S., 19.6.28).

[Block-type] galvanic batteries. L. and G. J. A. FULLER (B.P. 322,251, 6.9.28).

[“Sac”-type] electric batteries. E. P. GRIFFIN and W. A. BUCKLEY (B.P. 322,285, 11.10.28).

Incandescence electric tubes [for illumination in long lines]. H. HARTMAN (B.P. 296,315, 27.8.28. Ger., 26.8.27).

[Cathode-glow] electric discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 321,915, 13.7.28).

Röntgen tubes [with round focal spot]. RADIOLOGIE A.-G., and F. BENNEMANN (B.P. 322,680, 19.1.29).

[Projection] means for [electrolyte in] electroplating [baths]. W. J. PITT (B.P. 322,611, 6.11.28).

Furnace linings (B.P. 310,458).—See VIII. Removal of metal (B.P. 322,118). Metallic deposits (B.P. 299,725). Electrodeposition of copper (B.P. 322,371).—See X. Caoutchouc-coated metal (B.P. 302,250).—See XIV. Photochemical products (B.P. 321,992).—See XX.

XII.—FATS; OILS; WAXES.

Comparison of cacao butter and its substitutes. W. VAUBEL (Chem. Umschau, 1929, 36, 377—380).—Determinations made in an endeavour to discriminate between expressed and extracted cacao butter, fat from husks, etc., and substitutes are described. The author observes that the determination of the various bromine-iodine values (cf. B., 1927, 883) may be of assistance, but the corresponding values for the fats under comparison must be obtained under similar conditions; the bromine absorption of mixed fats is a new characteristic and not calculable directly from the known absorptions and proportions of the components.

E. LEWKOWITSCH.

Solidification curves of cacao butter. J. STRAUB and R. N. M. A. MALOTAUX (Chem. Weekblad, 1929, 26, 596—599).—The temperature-time diagrams of solidifying fats may be determined satisfactorily with the aid of a Dewar vessel cooled in water maintained at 10°. The two types of curves observed for cacao butter by Pichard (B., 1923, 1185 A) have been reproduced with this simple apparatus, and the differences between them traced to the presence of a non-fatty constituent which retards crystal formation; this substance may be removed by treatment with hydrochloric acid, after which the curve of the first type changes over to that of the second type.

S. I. LEVY.

Quality of fat from pigs. P. I. MEDVEDTSCHUK (Biochem. Z., 1929, 214, 282—309).—A method of taking samples of fat from living pigs and a filter-press in which the samples are pressed while hot are described. The most useful property of the fat from which to estimate the quality of the meat to be obtained from the animals is the iodine value, although the thiocyanogen value (cf. Kaufmann, B., 1926, 447) and the refractive index should also be determined. The Margosches method for the determination of iodine value is recommended.

W. MCCARTNEY.

Testing of pure lard. H. P. TREVITHICK and M. F. LAURO (Oil and Fat Ind., 1929, 6, [12], 21—23).—Hydrogenated vegetable fats have the same effect as tallow on the Bömer value of a lard, and this test offers a more convenient and certain method for the detection of such adulteration than the phytosteryl acetate test. The glyceride crystals obtained by two or three recrystallisations of 50 g. of fat from 50-c.c. portions of ether at 15° (5°, or anhydrous acetone, or a 3:1 ether-alcohol

mixture may be used for the first crystallisation) must not be melted before the determination of the m.p.; the fatty acids may be melted into the capillary tubes, but should be kept in a desiccator before making the m.p. determination to avoid absorption of ammonia. The Bömer value for a pure lard is not less than 71.

E. LEWKOWITSCH.

Fluorescence of lard. A. VAN RAALTE and A. VAN DRUTEN (Chem. Weekblad, 1929, 26, 602—603).—The discrepancies between the results obtained by the authors separately (B., 1928, 792; 1929, 607) are found to have been due to different conditions of working. Under the conditions employed by van Raalte unrefined fat does not fluoresce, whilst refined fats generally do.

S. I. LEVY.

Drying oils. XII. Changes in linseed oil, lipase, and other constituents of the flaxseed as it matures. E. R. THEIS, J. S. LONG, and C. E. BROWN (Ind. Eng. Chem., 1929, 21, 1244—1248).—A series of flaxseeds of varying degree of maturity were examined, and characteristics of the oils obtained are given. The seeds contained a lipin- and ester-hydrolysing enzyme, the lipolytic activity (greatest in the presence of water, preferably at 25°) decreasing as the seed matures. Certain inorganic salts (e.g., alkali chlorides) retard the action of the enzyme, which, however, is not entirely destroyed during commercial hot-pressing, and may be responsible for the increase of acid value of such oils on storage if moisture be present.

E. LEWKOWITSCH.

Analysis of glycerin and standardisation of reagent acid. W. A. PETERSON (Oil and Fat Ind., 1929, 6, [12], 15—18).—Details of provisional official methods for the standardisation of the acid (sulphuric or hydrochloric) to be used in the International acetin method for the analysis of crude glycerin are submitted for consideration by the American Oil Chemists' Society.

E. LEWKOWITSCH.

Seeds of *Hydnocarpus alcala*. DE SANTOS and WEST.—See XIII. Esters of *Hydnocarpus Wightiana* oil. COLE.—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Plasticity of paint. F. H. RHODES and J. H. WELLS (Ind. Eng. Chem., 1929, 21, 1273—1277).—The plastic characteristics of a paint depend on many factors, of which the following are the most important: concentration by vol. of pigment, fineness, size-distribution, and shape of pigment particles, wetting and flocculation of the pigment, nature and viscosity of vehicle, and the presence in the paint of substances causing "false body," e.g., water, soap. A series of plasticity determinations were carried out in a modified Bingham-Green plastometer in order to study these variables, and the results are tabulated and discussed. The effect of the acid value of the oil is considered at some length. Whilst increase in acid value of oil is found in most cases to increase the mobility of the paint, this is not true for zinc oxide paints, probably owing to soap formation. Pigments adsorb free fatty acids from linseed oil to markedly different extents, affecting both oil absorption and character of pastes formed. Nature of thinner is also of importance; e.g., turpentine lowers

yield point and increases mobility to a smaller extent than does a petroleum thinner or xylene. This is probably bound up with the tendency of turpentine to agglomerate the pigment. S. S. WOOLF.

Wetting of pigments and its relation to various paint characteristics. E. L. McMILLEN (Ind. Eng. Chem., 1929, 21, 1237—1239).—The wetting of lithopone by a number of liquids was studied according to Bartell's method (B., 1928, 1). Contact angles and adhesion tensions are compared with yield values for the mixtures concerned. It is shown that good wetting (high adhesion and small angle of contact) results in flocculation, high yield value, and low mobility, whilst very poor wetting causes practically no yield value and high mobility. Settling is greatest in those liquids which are the poorest wetters. These results confirm the ideas of such earlier workers as De Waele, Lewis, and Haller. S. S. WOOLF.

Pigment and oil. E. KLUMPP and H. MEIER (Farben-Ztg., 1929, 35, 599—601).—The authors' previous work (B., 1929, 989) is extended to mixtures of primary and secondary pigments, oil absorption-composition curves for which are given. From theoretical considerations the amount of oil absorbed by secondary particles is divided into "agglomerate" oil and "interstitial" oil. Curves based on the latter figures closely resemble those for mixtures of primary particles, and it is possible to calculate the size of the secondary aggregates from the position of the minimum in the curve. S. S. WOOLF.

Mechanism of baking process of oil varnishes. R. H. KIENLE and L. V. ADAMS (Ind. Eng. Chem., 1929, 21, 1279—1282).—Baking tests on a typical stoving varnish were carried out in a specially designed glass apparatus that permits the use of different atmospheres. The degree of baking was judged by observing the change in colour corresponding to the following states of the film: (a) dry to touch (V_s); (b) useful, i.e., non-tacky either warm or cold (V_u); (c) corresponding to one third the time taken to reach the "useless" state, when the film cracks or checks on the slightest abuse (V_r). The baking process involves oxidation and polymerisation. When oxygen is present the rate of baking at any temperature increases with the oxygen concentration. If oxygen be absent and vapours formed be removed as they are produced, baking proceeds independently of the gas atmosphere in which it is carried out. The baking process is expressed in the form of a general chemical equation. S. S. WOOLF.

Abietic anhydride. E. FONROBERT (Chem. Umschau, 1929, 36, 373—377).—The existence of abietic anhydride (cf. Fonrobert and Pallauf, B., 1926, 594; also Nagel, B., 1929, 219) is confirmed. It crystallises in shining scales (m.p. 151°, $[\alpha] -18^\circ$ in benzene) and is clearly distinct from abietic acid (prismatic crystals, $[\alpha] 0^\circ$ to -4° in benzene) and shows f.p. depressions (in benzene, nitrobenzene, camphor) equivalent to a bimolecular abietic acid derivative. It does not distil at 275°/3 mm. In contradistinction to Dupont's dehydrated abietic acid (Chim. et. Ind., 1927, 20, 1691), which gives normal unimolecular values, no ethane is evolved by the anhydride on treatment with magnesium ethyl bromide. On boiling Fonrobert's derivative with anhydrous ethyl

alcohol equivalent amounts of abietic acid and ethyl abietate are produced, affording conclusive proof of the acid-anhydride nature of the compound. A corresponding anhydride may be obtained from pyroabietic acid (cf. Fonrobert and Greth, B., 1929, 403). The m.p. 151—153° as given by Dupont for the dehydrated abietic acid is queried: by recrystallisation from anhydrous butyl ether the m.p. was raised to 165°.

E. LEWKOWITSCH.

Physical properties of shellac solutions. II. M. RANGASWAMI and M. VENUGOPALAN (Indian Lac Res. Assoc., 1929, Bull. No. 2, 17 pp.).—The experiments already described (cf. B., 1929, 64) are extended to the use of binary solvent mixtures of alcohols, alcohols and esters, and alcohols and ketones. As an efficient solvent mixture gives high "solvent power" with low viscosity of solution, the ratio of solvent-power number to relative time of flow of solution with reference to water has been taken, for purposes of comparison, as a measure of efficiency. From such considerations it is shown that a mixture of equal volumes of methyl and ethyl alcohols is the most efficient mixture of these two solvents. Corresponding advantages are not conspicuous in alcohol-ester mixtures (with the exception of ethyl alcohol-ethyl acetate), but alcohol-acetone mixtures show a marked peak in the efficiency-composition curve, and the desirability of employing non-solvents for admixture with alcohols on economic grounds is clearly indicated. S. S. WOOLF.

Resins in the seed coats of Philippine chaulmoogra seeds (*Hydnocarpus alcala*). I. DE SANTOS and A. P. WEST (Philippine J. Sci., 1929, 40, 485—489).—The oils obtained by extraction of the kernels, from which the seed coats (tegmen) had been removed, and from the seed coats themselves, of *Hydnocarpus alcala* had, respectively, d_4^{20} 0.9438, —; $[\alpha]_D^{20} + 46.1^\circ$, —; iodine value 84.1, 89.9; free fatty acid 0.25, 0.57; saponif. value 197.6, 197.9; n_D^{20} 1.4765, —; unsaponifiable matter 0.45, 0.76; resin acids nil, 3.73%; appearance white solid, dark brown oil.

E. H. SHARPLES.

Viscosity and plasticity. BLOM. **Adhesion tension and liquid absorption.** BARTELL and GREAGER.—See I. **Carbon black.** JOHNSON.—See II. **Rosin size.** DOLIWO-DOBROWOLSKIJ and KAHAN.—See V.

PATENTS.

Titanium pigment and paint forming durable outside coating. SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 302,659, 14.12.28. Czechoslov., 19.12.27).—Barium carbonate obtained by precipitation and preferably ground is added to titanium oxide obtained by hydrolysis from sulphate solutions and subsequently dehydrated, in sufficient quantity to provide 10—30% of barium carbonate in the resulting composite pigment. S. S. WOOLF.

Manufacture of heavy-metal salts of oil-soluble sulphonic acid and of drying oil compositions, paints, varnishes, etc. containing such salts. C. ARNOLD. FROM STANDARD OIL DEVELOPMENT CO. (B.P. 321,980, 23.8.28).—The residue of sodium sulphionate and petroleum oil which is derived from the crude sulphonic acid formed in the sulphonation of

petroleum oils is dissolved in aqueous alcohol, oil is removed by successive extractions with light petroleum, and the oil-free sodium sulphonate is recovered by evaporation of the solvent and dissolved in water. A water-soluble salt of a heavy metal, *e.g.*, lead, manganese, or cobalt, is added to the solution, and the precipitate of oil-soluble sulphonate is dried and purified by dissolving in organic solvents, thus separating insoluble inorganic matter. The sulphonate is used as a drier for paints, varnishes, etc. (cf. U.S.P. 1,735,493; B., 1930, 69).

S. S. WOOLF.

Wet process of production of vermilion, fast to light, from its elements. A. EIBNER (G.P. 453,523, 7.4.25).—The mass of black mercury sulphide or of cinnabar produced by shaking mercury and sulphur with a solution of liver of sulphur is dissolved in potassium sulphide and vermilion is recovered from the solution.

J. S. G. THOMAS.

Treatment of casein varnish-finished coated fabrics or articles and products thereof. E. I. DU PONT DE NEMOURS & Co. (B.P. 301,024, 10.11.28. U.S., 23.11.27).—A casein varnish is applied to the surface of rubberised fabrics etc. and is then halogenised, *e.g.*, with a 4% solution of bromine in carbon tetrachloride; free halogen and halogen acid are then neutralised, *e.g.*, with ammonia. Improvements in the quality of the surface and increase of waterproof characteristics of the film are claimed.

S. S. WOOLF.

Manufacture of plastic masses. R. BÜNZ (B.P. 322,133, 11.2.29).—Artificial stones, hard rubber-like materials, and grinding discs etc. are obtained from linseed oil by treatment with more than 35% of sulphur chloride and addition, with stirring, of mineral substances, *e.g.*, ground quartz sand, slate powder, emery.

S. S. WOOLF.

Manufacture of plastic and mouldable materials. H. P. BAYON. From MAGNASCO ROGGERO & Co. (B.P. 322,158, 21.7.28).—Cellulosic material, *e.g.*, wood pulp, cotton linters, is modified by treatment with a solution of an oxidising acid, *e.g.*, nitric acid alone or mixed with sulphuric acid, and preferably alkaline treatment. It is then incorporated under heat with an initial phenol-aldehyde condensation product which may be added as such or formed *in situ*. Halogenated aromatic hydrocarbons may be added at any stage of the reaction. The product may be stored indefinitely in an amorphous condition before being moulded under heat and pressure.

S. S. WOOLF.

Preparation of thermoplastic sheets. H. WADE. From BAKELITE CORP. (B.P. 314,937, 28.8.28).—A pulp composed of fibre and a reactive and plastic resin is prepared in the subdivided state and suspended in water or other liquid medium, a sheet is formed by laying this pulp and draining away excess suspension medium, and the residual suspension medium is dried out from the drained sheets under conditions which preclude the reaction of the resin to an infusible state and lessening of its plasticity. Drying may be effected by means of a current of heated and preferably pre-dehydrated air or other gas, the treatment being carried out under reduced pressure.

S. S. WOOLF.

Manufacture of artificial [polyvinyl] materials.

O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 322,157, 22.5.29).—Polyvinyl esters, alcohols, or ethers are heated at about 100° in the presence of catalysts, *e.g.*, hydrochloric acid, lactic acid, zinc chloride, time and temperature varying with the nature and amount of catalyst. If catalysts be absent, temperatures of about 200° must be employed. The resultant materials are elastic, more or less infusible, and insoluble or only very slightly soluble in the usual organic solvents.

S. S. WOOLF.

Nitrobenzene-sulphur resin. W. C. WILSON, ASST. to CUTLER-HAMMER, INC. (U.S.P. 1,732,453, 22.10.29. Appl., 22.6.25).—Nitrobenzene is refluxed with sulphur until resinified, a filler (asbestos) is added, and the mixture is cooled, ground, and screened. The product may be moulded cold and cured by heat treatment. Free nitrobenzene and other volatile substances are removed if the article be moulded hot, or if furfuraldehyde be added to the mix to increase its strength.

F. G. CLARKE.

Manufacture of acid- and water-proof black ink. J. INOUE (U.S.P. 1,738,998, 10.12.29. Appl., 9.6.25).—See B.P. 253,368; B., 1926, 638.

Production of plastics and elastic substances. KEMIKAL, INC., ASSEES. of I. S. MELLANOFF (B.P. 306,522, 28.8.28. U.S., 23.2.28).—See U.S.P. 1,681,155; B., 1928, 866.

Aqueous dispersions of resins (B.P. 321,721). **Products from asphalt etc.** (B.P. 313,433).—See II. **Glass substitute** (B.P. 308,587). **Varnish for fish-bone bristles** (B.P. 305,484).—See V. **Face paint** (B.P. 322,376).—See XXIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

"Dry rubber" content of latex from deep and shallow tapping. B. J. EATON and R. G. FULLERTON (J. Rubber Res. Inst. Malaya, 1929, 1, 132—134).—Tests on a series of samples over a year from two plantations in the coastal area of Malaya show that the "dry rubber" content of latex is generally greater from shallow than from deep tapping.

D. F. TWISS.

Effect of damp storage on raw rubber. B. J. EATON and R. G. FULLERTON (Rubber Res. Inst. Malaya, 1929, Bull. No. 2, 26 pp.).—Smoked sheet rubber and air-dried sheet rubber when kept in an atmosphere almost saturated with water at about 28° absorb moisture, reaching a maximum after several days. Almost invariably moulds develop from spores already present and a loss in weight occurs concurrently amounting after 20 months to as much as 28%; it is evident, therefore, that the rubber hydrocarbon itself is attacked to a considerable extent. "Spot disease" in crêpe rubber also causes a loss in weight, which is less, however, than that observed for sheet rubber. The growth of moulds does not reduce the nitrogen content of the rubber, although the character of the nitrogenous constituents may undergo alteration. Under the above conditions *p*-nitrophenol is not an absolutely effective preventive, but its introduction into the latex or application to the sheet considerably delays the development of mould; sodium trichlorophenoxide is less effective than *p*-nitrophenol, probably on account

of its conversion by acids into volatile trichlorophenol. The defect known as "white streak" in smoked sheet rubber is caused by absorption of water from a damp atmosphere and not to incomplete drying during the smoking operation. The pale amber colour frequently favoured in smoked sheet rubber is undesirable and indicative of too complete combustion of the fuel for the smoking chambers, so that little, if any, fungicidal creosote is absorbed by the rubber. D. F. TWISS.

Identification and determination of the yellow pigment of raw rubber. B. J. EATON and R. G. FULLERTON (J. Rubber Res. Inst. Malaya, 1929, 1, 135—148).—The unsaponifiable portion of the resins of raw *Hevea* rubber contains the yellow hydrocarbon pigment carotene. It is possible to determine the carotene in the acetone extract colorimetrically, using a standard solution of potassium dichromate for comparison.

D. F. TWISS.

Berginisation of raw rubber. H. I. WATERMAN, R. H. DEWALD, and A. J. TULLENERS (Kautschuk, 1929, 5, 200—202).—Samples of crêpe rubber were heated in an autoclave for 15 min. at 450° with and without the presence of hydrogen at 110 kg./cm.² pressure. The berginised sample yielded a volatile product in greater quantity and of lower refractive index, higher aniline point, and greater stability than the merely cracked sample.

D. F. TWISS.

Accelerators of vulcanisation [of rubber] and their use. F. EMDEN (Kautschuk, 1929, 5, 241—244, 269—276).—A list is given of 99 commercial accelerators of vulcanisation including their trade names and, in most cases, their chemical description. Such accelerators are grouped into "ultra-accelerators," *e.g.*, salts of dithioacids and the thiuram sulphides; "semi-ultra-accelerators," *e.g.*, mercaptobenzthiazole and some aldehyde-amine products; "moderately strong," *e.g.*, diarylguanidines, aldehyde-ammonia, and most aldehyde-amines; and "weak," *e.g.*, trisubstituted guanidines, formaldehyde-amines, and thiocarbamides. The members of these classes are considered individually as to such characteristics as method of formation, sp. gr., colour and general appearance, m.p., solubility, accelerative activity, and best conditions for use.

D. F. TWISS.

Ageing of vulcanised rubber under varying elongation. A. A. SOMERVILLE, J. M. BALL, and W. H. CORE (Ind. Eng. Chem., 1929, 21, 1183—1187).—Using a basic mixture of rubber 100, zinc oxide 5, whiting 60, stearic acid 1, sulphur 4, and diphenylguanidine 1½, the effect of various factors on the relative rate of ageing under different degrees of extension has been examined; vulcanised rings of "pure" rubber containing an antioxidant were also tested. In "bomb" ageing, the extent of deterioration was almost independent of the degree of extension; in a Geer oven, deterioration at 100% elongation was about 30% greater than for the unstretched material; whilst in ozone or sunlight, deterioration was greatest near an elongation of 5% or 10%. Cracking of stretched material did not occur in the bomb or in the Geer oven, but did so in ozone or sunlight, ozone possibly being responsible also in the last case. A modified

mixture in which the whiting was replaced by 40 pts. of carbon black gave similar cracking behaviour. A high proportion of sulphur favoured deterioration in sunlight or ozone, as also did under-vulcanisation. The presence of a filler such as whiting did not change the shape of the ozone- or sunlight-ageing curves, and whilst "pure" rubber bands with increase of stretch up to 700% showed progressively poorer ageing in the bomb, in ozone they exhibited the usual maximum deterioration at about 10% extension and a minimum at about 400%. The incorporation of tetramethylthiuram disulphide as a vulcanising agent and also mineral rubber led to some improvement in ageing in ozone or sunlight, but paraffin wax, although providing some protection in sunlight, permitted bad deterioration in ozone. The introduction of an antioxidant did not affect the degree of extension for maximum deterioration in sunlight or ozone, although it reduced the rate of ageing; also antioxidants giving comparable results in the oven and bomb tests gave unlike results in ozone or sunlight. Synthetic rubber behaved similarly to natural rubber.

D. F. TWISS.

Vulcanisation and structure of rubber. E. LIND-MAYER (Kautschuk, 1929, 5, 202—204).—Polemical in reply to Loewen (B., 1929, 444).

D. F. TWISS.

Permeability of rubber to air. I. Effect of temperature, pressure, and humidity. V. N. MORRIS and J. N. STREET (Ind. Eng. Chem., 1929, 21, 1215—1219).—Membranes 0.07 cm. thick, prepared from a mixture of rubber with sulphur 2.89%, zinc oxide 2.89%, and di-*o*-tolylguanidine 0.63% by vulcanisation at 149° for 30 min., were tested as to permeability by a manometric method. The permeability was approximately proportional to the pressure, but increased slightly more rapidly at higher pressures. Permeability increased rapidly with temperature, *e.g.*, by about 100% for a change from 26° to 37°. Brief immersion in water reduced the permeability, but prolonged immersion increased it considerably, probably by a loosening of the structure of the rubber; the effect of atmospheric moisture, however, was small.

D. F. TWISS.

Behaviour of progressively racked rubber with respect to ageing, hysteresis, m.p., and energy effects. H. FEUCHTER and E. A. HAUSER (Kautschuk, 1929, 5, 194—200, 218—227, 245—250, 276—283; cf. Feuchter, B., 1928, 377, 457).—An account of experimental results on the progressive racking of rubber and the nature of the spontaneous changes undergone by such rubber with time as indicated by its thermo-elastic behaviour, alteration in density, X-ray spectrum, resilient energy, etc. The experimental work is followed by a theoretical discussion.

D. F. TWISS.

Carbon black. JOHNSON.—See II.

PATENTS.

Manufacture of synthetic rubber. I. G. FARBERIND. A.-G. (B.P. 294,661, 27.7.28. Ger., 28.7.27. Addn. to B.P. 283,840; B., 1929, 485).—The polymerisation of butadiene, isoprene, etc. in aqueous emulsion in the presence of oxygen or of a substance yielding oxygen is accelerated by the additional presence of a water-

insoluble or colloid-soluble salt of a heavy metal, *e.g.*, cobalt, lead, manganese, chromium, or silver, especially of such metals as yield salts of value as siccatives.

D. F. TWISS.

Manufacture of synthetic rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,114, 17.12.28).—In the polymerisation of butadiene, isoprene, or similar hydrocarbons in an emulsified state, the process is expedited by the addition of polymerised or blown oils or of products derived from these, *e.g.*, by treatment with ammonia. Polymerised castor oil is mentioned.

D. F. TWISS.

Manufacture of articles of rubber or similar material. DUNLOP RUBBER CO., LTD., and G. W. TROBRIDGE (B.P. 321,913, 19.5.28).—An article of rubber of moderate thickness can be produced by a single immersion of a heated, non-porous former, *e.g.*, at 75°, into concentrated or into concentrated and compounded latex. After a very short time the former is withdrawn and the adherent substantially fluid deposit is allowed to set and dry.

D. F. TWISS.

Manufacture of treads for tyres. I. G. FARBERIND. A.-G. (B.P. 321,882, 30.8.28. Addn. to B.P. 299,037; B., 1930, 71).—The plastic products obtained by polymerisation of butadiene are employed as initial materials in the manufacture of treads for resilient tyres, whether solid or pneumatic. The processes of manufacture are similar to those used in the case of rubber. The treads so obtained maintain their elasticity to a great extent even at low temperatures.

D. F. TWISS.

Repair of pneumatic tyres. V. SONNINO and P. SAMA (F.P. 631,167, 19.3.27).—Sulphur and metallic oxides are added to rubber dissolved in benzene or carbon disulphide and the mixing is completed on warm rolls. Sulphur chloride is then added and the mixture is sheeted thinly. The sheet is then applied to the spot to be repaired, previously cleaned and painted with a rubber solution. A piece of fabric coated on one side with vulcanised rubber, and a translucent fabric, *e.g.*, tracing cloth, are finally applied in succession.

D. F. TWISS.

Treatment [increasing the age-resisting properties] of rubber and like material. GOODYEAR TIRE & RUBBER CO., ASSEES. OF (A) A. M. CLIFFORD, (B) J. TEPPEMA (B.P. 305,647 and 308,249, [A] 8.11.28, [B] 27.11.28. U.S., [A] 9.2.28, [B] 20.3.28).—(A) Compounds comprising two hydroxyaromatic molecules attached as in the formula $\text{OH}\cdot\text{R}\cdot\text{R}'\cdot\text{OH}$, where R and R' are aromatic groups, *e.g.*, β -dinaphthol, are effective antioxidants for rubber. (B) Aminoazo-substituted hydrocarbons, *e.g.*, *p*-aminoazobenzene or 4-aminoazo- α -naphthalene, are applied as antioxidants for rubber; they are substantially without accelerative influence on vulcanisation, and their inodorous and crystalline character is additionally advantageous.

D. F. TWISS.

Prevention of mould in rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 321,850, 3.12.28).—Mould fungi on rubber can be destroyed, and their growth prevented, by treating the rubber with a water-soluble inorganic acid containing one or more organic radicals, *e.g.*, alkyl, aralkyl, aryl, or cycloalkyl groups, derived from organic compounds which are soluble in

oils or fats, or with a water-soluble derivative of such an acid. Typical substances are sodium butylnaphthalenesulphonate, potassium methyl sulphate, and sodium ditolyl phosphate.

D. F. TWISS.

Accelerator of vulcanisation [of rubber]. GOOD-YEAR TIRE & RUBBER CO., ASSEES. OF J. TEPPEMA (B.P. 302,142, 27.8.28. U.S., 10.12.27).—Rubber is vulcanised in the presence of the reaction product of a 2-halogen-substituted benzthiazole and a dithiocarbamate, *e.g.*, 2-chlorobenzthiazole and sodium diethyldithiocarbamate.

D. F. TWISS.

Manufacture of caoutchouc-coated metal body. A. JENNY (B.P. 302,250, 12.12.28. Ger., 12.12.27).—The rubber layer formed on a metal such as aluminium by electrodeposition from an emulsion, *e.g.*, latex, is only feebly adherent and easily loosens on bending. This drawback is obviated by the addition of an alkaline solution of stick-lac or shellac to the rubber emulsion before electrodeposition.

D. F. TWISS.

Antioxidant or age-resister [for rubber]. A. M. CLIFFORD, ASSR. TO GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,739,480, 10.12.29. Appl., 10.12.27).—See B.P. 302,142; preceding.

Curing [vulcanisation] of [rubber] inner tubes. FIRESTONE TYRE & RUBBER CO. (1922), LTD. From FIRESTONE TYRE & RUBBER CO. (B.P. 321,937, 17.8.28).

Insulating materials (B.P. 322,208).—See XI. **Varnish-coated fabrics** (B.P. 301,024).—See XIII.

XV.—LEATHER; GLUE.

Control of vegetable tan liquors for tanning light leathers. Report of Committee, 1928—29 [of the American Leather Chemists' Association]. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 663—669).—Samples of hide powders tanned with quebracho, sulphite-cellulose, oak bark, quebracho-sulphite-cellulose extracts, and a blended liquor, respectively, were washed in Wilson-Kern extractors with distilled water for periods of from 1 hr. to 4 days, and each sample was dried and weighed to determine the increase in weight due to combined tannin. It was shown that the tannin-collagen compound was still being hydrolysed at the end of 4 days, contrary to Wilson and Kern's results (cf. B., 1920, 522A). With the exception of quebracho-tanned powder, the curves for removal of soluble matter from the tanned powders became practically linear after 24 hrs. and the slope of the linear portion was very small. The correct percentage of tannin in the extract was obtainable by extrapolation, but it was practically identical with that found by washing the powder for 16 hrs. only. D. WOODROFFE.

[Determination of acetic acid in calcium acetate in tan liquors.] Report of Committee [of the American Leather Chemists' Association] on control of vegetable tanning for heavy leather. D. H. CAMERON (J. Amer. Leather Chem. Assoc., 1929, 24, 669—677).—The acetic acid in the tail liquors is determined by distilling 10 c.c. of the liquor in steam and titrating the distillate with 0.1N-sodium hydroxide, using phenolphthalein as indicator. The calcium acetate is determined by acidifying a second 10 c.c. of the tan

liquor with phosphoric acid, distilling in steam, and deducting the acetic acid titration from the titration figure of this second distillate. Curves have been derived to show the amounts of acetic acid and calcium acetate in a series of "rocker vats," which show their distribution in the various tan liquors. Calcium acetate is detrimental to tan liquors, inhibiting acid-swelling, which is essential for favourable tannage.

D. WOODROFFE.

Calcium chloride in fellmongering practice.

A. M. WRIGHT and H. O. ASKEW (New Zealand J. Sci. Tech., 1929, 11, 157—165).—By mixing a solution of suitable concentration of calcium chloride with solutions of sodium hydroxide and/or sodium sulphide the alkalinity of the liquor was found to be diminished owing to the reaction of the calcium chloride with the sodium hydroxide, calcium hydroxide being precipitated. The alkalinity of 100 gals. of commercial sodium sulphide solution (d 1.12) was reduced 4% by adding 12.25 gals. of saturated calcium chloride solution. This mixture did not loosen the hair readily, and the best results were obtained with 1.75 gals. of saturated calcium chloride solution per 100 gals. of sodium sulphide solution. Less damage is done to sheepskins on which this modified "paint" is used in warm weather owing to the diminished alkalinity. Figures are given showing the alkalinity and sodium sulphide content of the treated pelts. Typical "paints" (d 1.236—1.260) of this type contain sodium hydroxide 4.15—4.35%, sodium sulphide 8.19—8.33%.

D. WOODROFFE.

Effect of temperature on chrome tanning.

H. B. MERRILL and H. SCHROEDER (Ind. Eng. Chem., 1929, 21, 1225—1227).—Pieces of pickled calf pelt were treated with a large excess of chrome-tanning liquor at temperatures varying from 10° to 50° and the rate of fixation was observed after periods of from 4 hrs. to 5 days. The fixation of chromic oxide increased with rise in temperature. The increase was greatest between 20° and 30°, and was 60% greater at 30° than at 20°. Leather tanned at 40° withstood the action of boiling water after 8 hrs.' tanning; that tanned at 20° after 48 hrs. The apparent acidity of the chromium salt fixed by the pelt was much higher at 20° than at 30°. Analysis of the liquors before and after use showed that the acidity, p_H value, and precipitation figure remained practically constant.

D. WOODROFFE.

Absorption by leather of sulphur compounds from spruce [and sulphite-cellulose] extract.

H. B. MERRILL and J. L. BOWLUS (Ind. Eng. Chem., 1929, 21, 1291—1292).—Analyses of seven calf leathers tanned with mixtures of vegetable tanning materials and sulphite-cellulose extracts showed 0.27—0.96% SO_3 (Procter-Searle), 4.98—9.34% total SO_3 (Wuensch), 0.50—0.85% SO_3 in ash, and 0.50—0.82% SO_3 extractable by sodium carbonate. Tests on hide powder tanned with mixed quebracho and sulphite-cellulose extracts and with sulphite-cellulose extract alone showed that the sulphur compounds absorbed by the powder from sulphite-cellulose extract were very resistant to hydrolysis, particularly with the sulphite-cellulose tanned powder. The ratio of the total material absorbed to the sulphur compounds absorbed from the mixed tannage

was independent of the time of tanning and of the p_H value, and was the same as the ratio of total tannin to sulphur compounds in the tan liquor. The sulphur-containing radical in the sulphite-cellulose is not removable by prolonged washing, and is an integral part of the tanning molecule. The Procter-Searle value based on leathers tanned with sulphite-cellulose extract is only a measure of the sulphur compounds which have been oxidised to sulphate during the ignition with sodium carbonate, which amount depends on the conditions of ignition.

D. WOODROFFE.

Cause of vein-like protuberances on finished leather. A. C. ORTHMANN and W. M. HIGBY (J. Amer. Leather Chem. Assoc., 1929, 24, 654—656).—It is concluded that such protuberances are due to coagulated blood left in the blood vessels owing to improper bleeding of the animal after death.

D. WOODROFFE.

Mould growth on leather and its prevention.

A. C. ORTHMANN and W. M. HIGBY (J. Amer. Leather Chem. Assoc., 1929, 24, 657—663).—Light-coloured spots, 0.1—10 mm. in diam., were observed on the grain side of coloured chrome-tanned calfskins, and could only be covered up by the application of heavy coats of pigment finishes, which detracted from the value of the leather. The spots are shown to be due to various mould growths on the leather in the "blue," arising from contamination with spores of the *Aspergillus* and *Penicillium* groups present in the materials and solutions used, particularly the chrome-tanning liquors and the cornflour used in the shaving operation. Growth of most of the moulds was prevented by adding an alcoholic solution of 5 oz. of β -naphthol to every 100 gals. of liquor in the tanning drums and "dip" vats, and of a 1:1 mixture of β -naphthol and phenol in the case of *Penicillium divaricatum*.

D. WOODROFFE.

Permeability of membranes. BERGMANN.—See I. **Oak chips from tan-works for paper.** NERECHTSKIY.—See V.

PATENT.

Waterproofing of leather for packing etc. J. J. DOUGHTEN (U.S.P. 1,735,564, 12.11.29. Appl., 11.12.28).—An untanned dressed hide is boiled in a lubricating oil for 60 min., immersed in glycerin, and cut into strips, braided, and used for packing shafts. D. WOODROFFE.

XVI.—AGRICULTURE.

Production of rational fertilisers based on phosphoric acid. C. MATIGNON (Chim. et Ind., 1929, 22, 860—867).—The preparation of phosphoric acid in the electric furnace is necessarily costly in energy. Its production by means of sulphuric acid introduces problems in purification. Urbain reduces phosphates in a shaft furnace to phosphorus, which is reoxidised in the upper and cooler part of the furnace. If a potash felspar such as leucite is used to supply the necessary silica, the process can be arranged so that the product KPO_3 is precipitated as dust from the gas. This process is being operated on the scale of 10 tons of materials per day. The furnace, which is not lined internally, is fed with briquettes of calcium phosphate, felspar, and coke breeze, mixed with lime, and a slag

cement is produced as by-product. An air blast is used preheated to 600°, and the potassium metaphosphate is recovered in a spray washer. It is considered that in a large furnace 5 pts. of coke would produce 1 pt. of metaphosphoric acid. This process could also be operated with an electric furnace, but with lower thermal efficiency. The acid metaphosphate solution may be used to bring a further quantity of tricalcium phosphate into solution, the calcium content being precipitated by addition of ammonium sulphate and neutralised with ammonia. A product is then obtained in the form of a neutral powder containing 30–32% P_2O_5 , 8–9% K_2O , and 9–10% N. The process might advantageously be attached to a coke-oven plant.

C. IRWIN.

Factors affecting the drillability of fertilisers.

A. L. MEHRING (Ind. Eng. Chem., 1929, 21, 1219–1223).—The effect of atmospheric conditions and fineness of grinding on the ease of distribution of fertilisers is examined. Temperature changes produced but little effect. All materials could be drilled at humidities below 50%, but above the hygroscopic point none could be handled satisfactorily. Fertilisers containing considerable amounts of material passing 200-mesh were too dusty when dry, but undrillable at greater humidities at which particles between 5- and 80-mesh worked easily. Ease of drilling varies inversely with the angle of repose of the fertiliser. Mixtures of particles of varying size, shape, and density tend to segregate in the distributor, producing notable differences in the proportions of plant nutrients applied to the soil. Best distribution was obtained with materials containing 20–30-mesh rounded particles having an angle of repose of 40°. Numerous records of typical fertilisers are included.

A. G. POLLARD.

Four-year field trials with six different nitrogen fertilisers. L. MAYER (Forts. Landw., 1929, 4, No. 6; Bied. Zentr., 1929, 58, 541–544).—Comparative field trials with a number of crops, using ammonium sulphate and chloride, sodium nitrate, calcium nitrate, cyanamide, and urea are recorded. Generally speaking, ammonium sulphate proved the most efficient, its value being emphasised during wet seasons. The crop increase per unit of fertiliser used was in most cases greatest with the smallest dressings applied, except in the case of cabbage treated with urea and with ammonium sulphate, where the second largest dressing was relatively the most efficient.

A. G. POLLARD.

Influence of increasing amounts of calcium sulphite and calcium sulphate on the germination and development of seedlings of cereals. K. SCHARER and W. SCHROPP (Landw. Versuchsstat., 1929, 108, 217–251; Bied. Zentr., 1929, 58, 558–560).—Germination and growth of wheat, rye, and barley in a loamy soil were not affected by small amounts of calcium sulphite, and only slightly depressed by larger quantities. Calcium sulphate, however, tended to decrease yields, the effect on wheat being less than on rye, barley, or oats. On low-moor and sandy soils both sulphite and sulphate tended to decrease yields without affecting germination. The effects were not traceable to changes in the p_H value of the soil.

A. G. POLLARD.

Fertiliser experiments with tobacco on the southern coast of Crimea in 1911–1919. W. E. EVKO (U.S.S.R. State Inst. Tobacco Invest., Bull. 55, 1929, 41 pp.).—Experiments carried out at Yalta, on a marl-slate soil, indicated that when 82 kg. N, 52 kg. P_2O_5 , and 75 kg. K_2O were applied (? per hectare) in the form of sodium nitrate, superphosphate, and potassium sulphate, respectively, the increase in yield of tobacco was 86%, 75% of which was due to the nitrogen and 10·5% to the phosphoric acid and potash. Farmyard manure in amounts of 18–100 tons gave increases of 16–35% in yield, and was chiefly useful in providing nitrogen and in improving soil texture. There are advantages in autumn application of artificial fertilisers.

E. HOLMES.

Comparison of the Mitscherlich and Neubauer methods for determining the nutrient content of soils. J. PÁZLER (Z. Zuckerind. Czechoslov., 1929, 54, 153–170).—The nutrient values of soils as determined by the two methods show good general agreement, but in the detailed figures some divergence is apparent. In soils of small phosphate content the Mitscherlich values are relatively lower than those of Neubauer, and the reverse is the case in rich soils. In soils containing much chalk Neubauer's method indicates lower phosphate contents than that of Mitscherlich. Potash contents determined by the two methods show poorer agreement than in the case of phosphates. By grouping the soils examined a certain parallelism between the two sets of results is obtained. In very rich soils both methods yield similar results, but in all others Mitscherlich's values are about 75% lower than Neubauer's. With clay soils Neubauer values are the higher, and approach double the Mitscherlich figures where, in addition, much chalk is present. On sandy soils containing much chalk Neubauer values are lower than those of Mitscherlich. The limiting values of the Neubauer determination on which manurial requirements are based must be selected with reference to the mechanical analysis and chalk contents of the soil examined.

A. G. POLLARD.

Process of nicotine formation during germination of tobacco seeds. G. S. ILIN (U.S.S.R. State Inst. Tobacco Invest., Bull. 57, 1929, 26 pp.).—Studies of the germination of tobacco seeds during 15 days, both in the light and in the dark, show that although nicotine is absent at first, after 5 days it is present in considerable and increasing quantities. The formation of nicotine varies with the decomposition of storage proteins and with the increase of soluble nitrogen compounds; production of the latter compounds reaches 25% of the initial proteins, both in the light and in the dark. The amount of amines produced is rather greater in seeds germinated in darkness. The relation formerly established between nicotine content and storage of proteins in ripening seeds is reversed during germination.

E. HOLMES.

Periodic harvesting of tuba root (*Derris elliptica*, Benth.). C. D. V. GEORGI and E. A. CURTLER (Malayan Agric. J., 1929, 17, 326–334).—The variation in the amount of ether extract from tuba roots of various ages was investigated; the optimum age for harvesting is at 25 months (about 1130 lb. of roots/acre as sole crop),

at which period both the ratio of "fine" to total roots and the ether extracts from "fine" and total roots are maximal. E. LEWKOWITSCH.

Variations in the amount of ether extract of tuba root (*Derris malaccensis*, Prain). C. D. V. GEORGI (Malayan Agric. J., 1929, 17, 361—363).—The amount of the ether extracts from "fine" and "coarse" roots of *D. malaccensis* (Erect Sarawak) are four and eight times, respectively, as great as those from *D. elliptica* (cf. preceding abstract); the optimum age of the plants for harvesting in order to obtain maximal yield of ether extract is about 23 months. Assuming that the toxicity of the root is related to the amount of extract, it seems advisable for insecticidal purposes to establish standards of valuation based on the latter.

E. LEWKOWITSCH.

New solvents for the active principles of pyrethrum (*Chrysanthemum cinerariæfolium*, Trev.). W. A. GERSDORFF and W. M. DAVIDSON (Ind. Eng. Chem., 1929, 21, 1251—1253).—A large number of solvents (alcohols, hydrocarbons, chlorinated hydrocarbons, etc.) completely extract the active principles (against the aphid *Myzus persicae*, Sulz.) from pyrethrum; most of these vehicles (except kerosene, xylene, and amylene dichloride), and especially ethyl alcohol, are suitable for application to plants as resistant as cabbage without injury to the foliage.

E. LEWKOWITSCH.

Toxicity of common arsenicals. L. FULMEK (Forts. Landw., 1929, 4, 209; Bied. Zentr., 1929, 58, 555—558).—The toxicity of a number of arsenites and arsenates was examined, using caterpillars of the cabbage white and vapourer moth and the leaf weevil. The order of toxicity of the arsenites was: $Mg > Pb = Ca = Cu > Fe > Zn$; and of the arsenates: $Pb > Cu > Ca > Mg > Zn > Fe$. The order of toxicity of these materials is not paralleled by their contents of water-soluble arsenic, but agrees closely with their solubilities in buffer solutions of pH 9.0, which is approximately that of the digestive juices of the insects.

A. G. POLLARD.

Decomposition and action of the nitrogen and carbon compounds in stall manure. K. SCHEIBE (Landw. Versuchsstat., 1929, 108, 61—114; Bied. Zentr., 1929, 58, 537—541).—In the fermentation of stall manure the loss of dry weight and of nitrogen was greater from a compressed than from a loosely-stacked heap. Losses of nitrogen from the upper portions of the stack were considerably reduced by banking with earth. In a comparison of hot-fermented manure (Krantz) with that from a closely compacted stack and from an ordinary yard stack, the rate and extent of nitrification, rate of carbon dioxide production in soil, and crop yields were in the (descending) order named. A. G. POLLARD.

Analytical-chemical determination of the noxious influence of sulphur dioxide on vegetation. M. KRAJČINOVIČ (Arh. Hemiju, 1929, 3, 187—192).—The volatile and fixed sulphur contents as well as the ash content of leaves in the vicinity of a galena roasting furnace are higher than the corresponding values of leaves in other localities. R. TRUSZKOWSKI.

Phosphate rock. REYNOLDS and others. **Phosphates.** LARISON.—See VII. **Artificial milk for cattle-rearing.** TERROINE.—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Losses in weight and sugar content of beet slices during brief steaming. F. KRYŽ (Z. Zuckerind. Czechoslov., 1929, 54, 170—171).—The steaming of fresh beet slices for 5 min. under conditions such that actual extraction with condensed water is avoided leads to a loss in weight of 12—14% and in sugar content of 4—5%. The bearing of this on certain commercial processes is indicated. A. G. POLLARD.

Influence of some non-sugars on the crystallisation of sugar. E. SOMMER (Z. Zuckerind. Czechoslov., 1929, 54, 133—140).—It is especially the nitrogenous non-sugars, betaine in particular, that exert an effect on the crystallisation of sugar, though amino-acids and colouring matters must also be included. The ratio of the molar concentration of sugar to the equivalent concentration of potassium and sodium ions is no measure of molasses formation, since even at a ratio of 1:1 sugar will crystallise out. J. P. OGILVIE.

Enzymes as reagents. BARGELLINI. **Gluconic acid.** MAY and others.—See XVIII.

PATENTS.

Carbohydrate products (B.P. 294,572 and 322,003).—See V. **Concentration of fruit juice** (F.P. 31,589, 32,286, and 32,298). **Higher alcohols, acetone, etc.** (B.P. 322,029).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Influence of the construction of the malt kiln on the colour of the malt. B. GRÖTZBACH (Woch. Brau., 1929, 46, 515—516).—Using a two-floor malt kiln with a relatively short outlet chimney, pale malt could only be obtained if the temperature were retained under 69°. When the kiln was converted into a three-floor type having a specially high outlet chimney with a revolving cowl, the kilning temperature could be raised to 87—93° without the malt becoming too deep in colour, even if made from highly nitrogenous barley. The new outlet chimneys have proved themselves constructionally sound and resistant to high wind velocities.

C. RANKEN.

Respiration of barley during malting. R. KÜHLES (Woch. Brau., 1929, 46, 489—491, 501—503).—The usual method of following the progress of respiration by the measurement of the carbon dioxide evolved by the grain during germination was replaced by one in which the amount of oxygen consumed was determined. The samples were taken at all stages of the malting operations, and after drying as rapidly as possible at 30—35° were ground and sieved. For the tests the finest flour was suspended in water which was saturated with oxygen. Respiration was activated by the addition of sodium zymophosphate and of phosphate buffers, a maximum being reached with the latter at about pH 7.6. The further addition of boiled yeast juice containing co-

enzyme did not influence the result. The small amount of oxygen taken up by the ungerminated barley rapidly increased during steeping and during the first days of germination. After the third day the values became almost constant, but increased later to a maximum on the upper floor of the kiln. A rapid decrease followed owing to the partial destruction of the respiration enzyme at the higher temperatures and to the proportionally great loss of water. The respiration was sensitive within wide limits of temperature, and barley germinated at high temperatures showed a greatly increased respiration.

C. RANKEN.

Nitrogenous constituents of wort and their assimilation by yeast. S. B. SCHRYVER and E. M. THOMAS (J. Inst. Brew., 1929, 35, 571—576).—A description is given of the modifications of known processes which were necessary for the determination of the total, ammonia-, amino-, amide-, and peptide-nitrogen in wort. The values of the peptide-nitrogen, which were obtained by determining the amino-nitrogen after hydrolysis of the wort, were inconsistent. The variations were least when the hydrolysis of the wort was effected by heating with 16% hydrochloric acid for 20 hrs., and no advantage was obtained by carrying out the hydrolysis in an autoclave with 5% sulphuric acid at 140° or 120°. Attempts to replace the Van Slyke method for the determination of amino-nitrogen by Sørensen's and colorimetric methods failed owing to the inability of obtaining indicators which would function with the dark-coloured hydrolysed wort. Apart from some increases in the amount of amino-nitrogen when the wort is treated with macerated yeast, the attempts to avoid the dark colour of hydrolysed wort by using enzymes in place of acids as agents of hydrolysis have so far given unfavourable results.

C. RANKEN.

Calculation of the original gravity of beer. L. HEINTZ (Woch. Brau., 1929, 46, 513—515).—The divergence of the true original gravity of a wort from the values calculated by the several known formulæ is due to calculating the extract and alcohol contents in terms of percentage by wt. (i.e., as g. in 100 g.). The errors grow greater as the gravity of the wort increases, and the slightly greater accuracy of the results given by the Schönfeld-Balling formula is due to fortuitous increases in the divisor. The greatest concordance between the calculated and observed results is obtained if the calculation is based on terms of weight-vol.-% (i.e., g. per 100 c.c.). Thus the simplest type of formula $O.G. = 2A + E_w$, where A and E_w are the alcohol and residual extract contents respectively, gives results which require no correction provided A and E_w are calculated in terms of weight-vol.-%.

C. RANKEN.

Determination of volatile acid in wine. K. WORNICH (Oesterr. Chem.-Ztg., 1929, 32, 190—191).—In the determination of acetic acid in wine by the official methods, high values due to the inclusion of sulphur dioxide in the total volatile acids may be obviated by oxidation with hydrogen peroxide. The apparatus recommended consists of a steam generator which encloses the bulb containing the sample of wine. The bulb is fitted with a trap and connects to a water-cooled

receiver. After conducting a blank experiment to free the apparatus from acid, 10 c.c. of wine and, if sulphur dioxide be present, 3 drops of 3% hydrogen peroxide are introduced into the apparatus and steam-distillation is allowed to proceed at such a rate that 100 c.c. of distillate are collected in $\frac{1}{4}$ hr. It is necessary to arrange that the volume of liquid in the bulb shall be the same at the beginning as at the end of the distillation. Titration of the distillate is performed just below the boiling temperature with 0.1N-caustic soda.

H. J. DOWDEN.

Distillery problems. I. Technical fermentation of raisins. I. B. BLEYER, W. DIEMAIR, and S. TAHSIN (Landw. Jahrb., 1929, 69, 361—389; Bied. Zentr., 1929, 58, 568—570).—The effect of substrate nutrients on the fermentation of raisin mash by various yeasts is examined. The addition of potato press-juice increases the soluble nitrogen content of the mash to a considerable extent, and to this fact, in conjunction with the simultaneous addition of vitamins and mineral matter, the accelerated fermentation and increased vitality of the yeast are attributed. The alternative addition of various ammonium salts as a yeast nutrient is examined and the alcohol yields are recorded.

A. G. POLLARD.

Distillery residue [with higher feed value]. E. LÜHDER (Z. Spiritusind., 1929, 52, 371—372).—The residues from distillation will yield a cattle feed with a higher nutrient content if the proportion of malt to potatoes in the mash be decreased. The dextrin content of the wort will be raised at the expense of that of the fermentable sugars, and fermentation will cease sooner, leaving a residue with a higher fodder value after distillation. The diminished yield of alcohol is partly compensated by the saving in malt and by the increased yield of fodder. In addition, to attain the standard production of alcohol, the distillery will require to remain longer active during the winter, and thus permit the cattle being supplied longer with fresh fodder at a period when no pasturage is available.

C. RANKEN.

Enzymes as reagents. G. BARGELLINI (Giorn. Chim. Ind. Appl., 1929, 11, 493—499).—The subjects dealt with include: specificity of enzyme action; identification of monoses by means of *Monilia balcanica*, *Krusei*, and *metalonlinensis*; differentiation of glucosides; hydrolysis of polyoses and its analytical applications; action of proteases; oxidases of *B. xylinum* and the use of *B. xylinum* to prepare ketoses difficult to obtain otherwise, to differentiate aldoses from ketoses, to differentiate and separate various stereoisomeric polyhydric alcohols, and to transform various ketoses into stereoisomeric polyhydric alcohols and *vice versa*; reductases of *B. mannicus*; action of *B. pyocyanus*; enzyme of *B. aliphaticum*; detection of arsenic, selenium, and tellurium by means of moulds etc.

T. H. POPE.

Fermentation products of cellulose. C. S. BORUFF [with A. M. BUSWELL] (Ind. Eng. Chem., 1929, 21, 1181—1182).—The production of carbon dioxide, methane, and hydrogen during the fermentation of cellulosic material by inoculation with sewage sludge is examined. Results indicate an approximation to the equation $C_6H_{10}O_5 + H_2O \rightarrow 3CO_2 + 3CH_4$. Maize stems

produced most gas when pretreated by soaking for 4 days in limewater. Suitable plant for the production of gas for domestic purposes on these lines is indicated. In plant residues generally, pithy matter is digested much more quickly than fibrous material. The value of this in connexion with paper-making is discussed.

A. G. POLLARD.

Hydrolysis of pectin by various micro-organisms. G. A. PITMAN and W. V. CRUESS (Ind. Eng. Chem., 1929, 21, 1292—1295).—The pectinase activity of various micro-organisms occurring on fruits has been investigated under aerobic conditions at room temperature in or on apple juice containing added apple pectin. *Penicillium glaucum* (*P. expansum*) and a *Pythium* species exerted the greatest hydrolytic action, the latter destroying practically all the pectin in 12 weeks at about 20°. *Bacterium aceti* and *B. amylovorus* showed little, and *Saccharomyces cerevisiae*, several strains of *Mycoderma*, and *Saccharomyces ellipsoideus* from grapes no noticeable effect on pectin. The appearance and texture of the pectin precipitate varied greatly, and one organism (*Colletotrichum*), although not greatly reducing the quantity of pectin as determined by Wichmann's method, changed its character so much that it refused to set; in this case the enzyme involved was apparently pectase. The viscosities of the liquids from most of the cultures followed in only a general way the apparent pectin content. The latter was not greatly reduced by the natural enzymes of apple juice during the period required for complete fermentation of the sugars by *S. cerevisiae* at 35°. Hydrolysis of pectin by *Penicillium glaucum* is more rapid in media of p_H 6 and 5 than in one of p_H 3. When present in a pectin stock solution, dextrose is not altogether objectionable as it protects the pectin against hydrolysis by *P. glaucum*.

T. H. POPE.

Semi-plant-scale production of gluconic acid by mould fermentation. O. E. MAY, H. T. HERRICK, A. J. MOYER, and R. HELLBACH (Ind. Eng. Chem., 1929, 21, 1198—1203).—The yield of gluconic acid obtained by fermentation of commercial dextrose solutions by *Penicillium luteum purpurogenum* depends on the ratio of the surface area (sq. cm.) of the mycelium to the volume (c.c.) of the solution and varies from 82% of the theoretical yield when this ratio approaches 1 to 30% when the ratio is 0.16. The most convenient practical value of the ratio is 0.25—0.3, 20—25% dextrose solutions then giving yields of 55—60% and actual maximum weights of the acid. Each m.² of the mycelium is capable of producing 4—4.5 kg. of gluconic acid in 14 days from the time of inoculation of the spores. Sub-surface agitation of the solution enhances the production of acid with all but highly concentrated (25%) glucose solutions. Variation in the hydrogen-ion concentration of the liquid exerts no marked effect on the oxidation. Shallow pans, 43 × 43 × 2 in., of aluminium (at least 99.45% Al with less than 0.1% Cu + Mn) give good results as fermentation vessels, yields of 52—61% being obtained after the first few fermentations. A number of such pans may be arranged in a rack, enclosed by removable wooden frames covered with four thicknesses of cheese cloth as a protection against foreign fungal spores and provided with peep-holes;

sterile air may be supplied by means of a fan-blower. Bacteria and yeasts do not thrive under the conditions of the fermentation, for which the optimum temperature is 25°. Stock cultures of the organism keep active far longer at 4° than at room temperature. To prepare agar cultures for immediate use, transfers are made from a tested stock culture on wort agar to slopes of a medium containing, per litre, 15 g. of bacto-peptone "Difco," 30 g. of commercial dextrose, and 30 g. of agar-agar, which favours a good vegetative growth and a uniform and heavy sporulation over the entire slope in 8—10 days at 25—30°. A suitable sugar solution contains 63 kg. of commercial (about 91.5%) dextrose, 0.315 kg. of sodium nitrate, 0.079 kg. of crystallised magnesium sulphate, 0.016 kg. of potassium chloride, and 0.009 kg. of phosphoric acid (100%) per 315 litres, this being sterilised in steam at 100°. The fermented solution is neutralised with calcium carbonate or thoroughly slaked lime (the p_H being kept below 4.5) and the calcium gluconate, after crystallisation, decomposed in hot 15—20% solution by the calculated amount of dilute sulphuric acid. The raw material costs \$4—5 per 100 lb., giving, for a 50% yield, \$8—10 as the cost of the acid, but at least one half of the dextrose lost should be recoverable by a secondary fermentation to either gluconic acid or alcohol.

T. H. POPE.

Unusual alcoholic fermentations. J. R. BOFF, JUN., H. BUTTLER, and W. MELCHIOR (Ind. Eng. Chem., 1929, 21, 1277—1279).—Fermentation of solutions of sucrose in pineapple juice yields pineapple wines showing no sweetness (1.8% of sugar), no marked flavour, and a high alcohol content (up to 19.2% by vol.). Among the various yeasts tested, the two wine yeasts, Tokay and Steinberg, stand out as alcohol producers, the maximum alcohol yield being obtained at about 15°. The must should consist, partly at least, of fresh juice, sterilised or old juices giving lower proportions of alcohol. Unlike what occurs with grape must, sugared pineapple must without much added water undergoes less attenuation than a mixture of 1 pt. of pineapple juice with 2 pts. of 35% sugar solution. Yeast revived from agar cultures is more active than yeast kept in liquid media for long periods.

T. H. POPE.

PATENTS.

Concentration of fruit juices and extraction of by-products therefrom. SOC. DES ÉTABL. BARBET (Addn. F.P. 31,589, 31.10.25; 32,286, 3.3.26; and 32,298, 17.3.26, to F.P. 615,942; B., 1927, 856).—A boiler of high capacity, which works without pressure, is used to heat simultaneously the preheater, concentrator, and pasteurising apparatus by circulating steam. Fruit refuse, such as grape skins, is dried in the evolved hot gases, and used as fuel for the boiler. The juice is concentrated to d 1.415 and the crystallisation of sugar and tartar initiated by stirring and by seeded dextrose. In the case of grape juice, the filtered syrup, which contains 4—6 times as much lævulose as dextrose, is slightly further concentrated, and by repeated crystallisation eventually yields lævulose free from dextrose.

C. RANKEN.

Manufacture of higher alcohols, acetone, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A-G.

(B.P. 322,029, 19.9.28).—A solution of sugars obtained by the action of acid on peat is fermented with bacteria of the *Amylobacter* group. Prior to inoculation, humic substances, tannin, etc. are preferably adsorbed and removed from the sugar solution by calcium phosphate precipitated in the solution by added water-soluble phosphates (cf. B.P. 318,649; B., 1929, 994).

C. RANKEN.

Denaturing of alcohol. I. G. FARBENIND A.-G. (B.P. 298,611, 11.10.28. Ger., 12.10.27).—The alcohol is mixed with the sparingly water-soluble higher fractions (boiling above 120°) of the natural pyridines, or with homologues of synthetic pyridines which are sparingly soluble in water. These denaturing agents may be mixed with each other or with the natural water-soluble pyridines of commerce.

C. RANKEN.

XIX.—FOODS.

Preparation of artificial milks for the rearing of cattle. E. F. TERROINE (Compt. rend., 1929, 189, 866—868).—The most satisfactory artificial milk is found to consist of water 1 litre, tapioca flour 112 g., leguminous flour 75 g. (pea flour 4.04% N, soya bean 7.38% N), malt extract 13 g., calcium citrate 2 g., sodium chloride 1.10 g., sodium bicarbonate 0.25 g. All fat in the diet is avoided. In the feeding of young pigs with this milk a greater increase in weight is observed over the same period than with cow's milk alone. With calves diarrhoea is apt to supervene and not more than 50% of the cow's milk can be replaced by artificial milk. Digestibility is ensured by previous slow cooking by the action of the amylase of the malt extract. The necessary vitamins are obtained by periodic feeding of portions of cow's milk. P. G. MARSHALL.

Bacteriological control of bottled milk pasteurised by the holding method. K. J. DEMETER (Milch. Zentr., 1929, 58, 367—372).—A bibliographical review is given of investigations on the thermophilic, heat-resistant, spore-forming, and non-spore-forming bacteria which are to be found in milk after pasteurisation. The best method of determining the efficiency of pasteurisation is the *B. coli* test, in which 0.1-c.c. samples are taken from five different parts of the milk and examined by the gentian-violet-lactose-peptone-bile method or the indole reaction. Not more than one sample should give a positive reaction. Pasteurised milk of good quality should contain not more than 30,000 living germs per c.c., and no milk should be offered to consumers if it contains more than 100,000 germs per c.c.

H. J. DOWDEN.

Potassium nitrate in Canadian cheese. F. C. HARRISON (Canad. J. Res., 1929, 1, 256—260).—Coloured cheeses containing potassium nitrate are frequently discoloured by flesh-pink patches. Sterile milk containing annatto and 0.05—0.1% KNO_3 when inoculated with bacteria from defective cheese produced identical discoloration. The organisms causing the pink coloration are nitrate-reducing, so that young cheeses give positive reactions for nitrite, but at a later stage the nitrate is converted into ammonia or free nitrogen. A schedule of nitrate-reducing organisms is given, not all of which produce the pink colour in nitrated annatto

milk. Similar bacteria have been isolated from potassium nitrate itself. Since annatto remains unchanged in presence of sodium nitrite, but turns red when acid also is added, it is probable that conditions other than the presence of nitrate-reducing bacteria are necessary to produce the discoloration of nitrated cheese.

H. J. DOWDEN.

Seguidillas bean. F. AGCAOILI (Philippine J. Sci., 1929, 40, 513—514).—The edible seed of *Psophocarpus tetragonolobus* ("seguidillas") contains water 9.74%, fat 17.04%, protein 32.81%, starch 12.50%, carbohydrates other than starch 18.7%, ash 4.01%, crude fibre 5.20%, and on extraction yields an oil having d_{20}^{20} 0.9284, n_D^{20} 1.4666, n_D^{30} (Zeiss) 62.8, saponif. value 175.6, and iodine value 82.1. These characteristics are compared with those of the soya bean, to which it is very similar.

E. H. SHARPLES.

Effect of hydrogen-ion concentration in the dyeing of cherries. R. N. JEFFREY and W. V. CRUESS (Ind. Eng. Chem., 1929, 21, 1268—1269).—The p_H values of the cherries and of the solutions used in dyeing the cherries with Erythrosin greatly affect the evenness and rapidity of dyeing and the fixation or the bleeding of the colour in dyed cherries. The dye is best applied in a dilute solution (0.3—0.4%) of sodium bicarbonate (approx. p_H 7.5), and should be fixed by dilute (about 0.5%) citric acid (or other permissible fruit acid) at approx. p_H 3.0—3.5. Leaching of Erythrosin is very slight at p_H 3.5 or less. Ponceau 3R and Amaranth bleed rapidly at all the p_H values used in the experiments reported.

E. LEWKOWITSCH.

Detection and determination of benzoic and salicylic acids. SNETHLAGE.—See III. **Cacao butter.** STRAUB and MALOTAUX. **Testing lard.** TREVTHICK and LAURO.—See XII. **Distillery residue as feed.** LÜHDER. **Hydrolysis of pectin.** PITMAN and CRUESS.—See XVIII. **Creamery wastes.** LEVINE.—See XXIII.

PATENTS.

Preservation of food [e.g., meat]. D. JASTRZEBSKI (B.P. 321,986, 24.8.29).—Meat is placed in a vessel which is then evacuated and "brine" (sodium chloride and potassium nitrate) is drawn in. The meat may finally be sealed by dipping in a solution of salt and agar-agar or melted paraffin wax.

E. B. HUGHES.

Animal foods [biscuits of special shapes]. A. E. WHITE. From ELLIS-FOSTER Co. (B.P. 321,965, 23.5.28).

Food preparations. [Custard tablets.] H. THOMPSON & Co.'s IDOLICE SPECIALITIES, LTD., and E. J. THOMPSON (B.P. 322,261, 15.9.28).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation and determination of sodium citrate. M. CATULLO (Boll. Chim. farm., 1929, 68, 1003).—The method described, which is expeditious though not highly economical, consists in dissolving 840 g. of citric acid in 840 g. of water, gradually neutralising the heated and stirred solution with sodium carbonate (to slight alkalinity), filtering, concentrating from d_{40}^{40} 1.20 to d_{80}^{80} 1.32, and allowing to crystallise. The total yield

from three crystallisations is 1075 g. of pure crystals. For the determination, 2 g. of the salt are gradually carbonised in a platinum or porcelain crucible, the flame being kept out of contact with the material and the final temperature not exceeding that of redness. The charred mass is boiled for 3 min. in a covered beaker with 50 c.c. of water and 50 c.c. of 0.5N-sulphuric acid, the solution being filtered and the residue washed with water until neutral to litmus paper. The total solution is titrated in presence of methyl-orange with 0.5N-potassium hydroxide, multiplication of the difference in volume (c.c.) between the acid and alkali by 0.04901 giving the weight (g.) of salt. T. H. POPE.

Chemical characterisation of drugs. L. ROSENTHALER (Amer. J. Pharm., 1929, 101, 784—787).—Two microchemical tests for mucus in mucous drugs are described. The sliced drug is placed in ferric chloride solution, washed, and then placed in potassium ferrocyanide solution. The mucus becomes dark blue. This test may be used for *Malvaceæ* drugs and *Semen lini*. The following test may be used for *Malvaceæ*, *Tubera salep*, and *Bulbus scillæ*: the material is placed in clear lead acetate solution for 5 min., then washed with water free from carbon dioxide, and placed in potassium dichromate solution. The mucus assumes a yellow colour. To test the mucus in slices and powder of *Tubera salep*, alkaline copper solution, which colours the mucus blue, is suitable. The examination of a number of drugs for sodium (using uranyl acetate), potassium (using copper lead nitrite), magnesium (with Pfahl's mixture of phosphates), calcium, and ammonium is described. A specific test for *Herba lobelia*: 5 g. of lobelia are distilled with 50 c.c. of water and 5 g. of sodium hydroxide so that 10 c.c. of distillate are collected. The distillate gives, with sodium nitroprusside and sodium hydroxide a deep orange to dark purple colour which on addition of acetic acid turns dirty violet; with phenylhydrazine hydrochloride or with *p*-nitrophenylhydrazine (in dilute acetic acid) immediately a strong turbidity is formed.

E. H. SHARPLES.

Manipulation of tobacco. V. P. IZVOSCHIKOV (U.S.S.R. State Inst. Tobacco Invest., Bull. 56, 1929, 27 pp.).—Studies of the curing, storage, and fermentation of tobacco show that the destruction of proteins may reach 26—31% during curing, but only 3—4% during fermentation. Amines and amino-acids increase during curing but usually decrease during fermentation. Despite considerable chemical changes there is little rise in temperature during fermentation of the Tyk-kulak type of tobacco. Chemical changes during storage and fermentation are the same.

E. HOLMES.

Content of methyl alcohol in tobacco and its manufactured products. A. SCHMUK and S. KASCHIRIN (U.S.S.R. State Inst. Tobacco Invest., Bull. 60, 1929, 11 pp.).—The amount of methyl alcohol in yellow cigarette tobacco varies from 0.4 to 0.9%, and in manufactured tobacco products from 0.2 to 0.75%. There is a relation between quality of tobacco and its methyl alcohol content: the better tobacco contains the more alcohol, but the "extra" quality contains less than the "first" quality. Different types of tobacco

such as Trebizond, Samsun, and Crimean types of American and Dubek contain nearly the same percentage.

E. HOLMES.

Content of reducing substances and their dynamics during the air-conditioned fermentation of an industrial tobacco. G. I. JURAVSKI (U.S.S.R. State Inst. Tobacco Invest., Bull. 58, 1929, 28 pp.).—There is a decrease in individual and total carbohydrates in Cuban tobaccos during air-conditioned fermentation, the amount of decomposition varying directly with the amounts originally present, but not with the heat developed. The amount of decomposition during after-fermentation or ageing is probably proportional to the original amount of decomposition and to the diminished activity of the ferment. Poly- and di-saccharides are hydrolysed to simple sugars and undergo further decomposition. Maltose and monoses in some samples, on the other hand, increased at the expense of the more complex carbohydrates. Starch and dextrins decompose more slowly during ageing than during fermentation. Anaerobic conditions during the period of intensive fermentation of bales of tobacco are unfavourable to decomposition of sucrose, but its decomposition varies with the activity of the invertase present. The dynamics of carbohydrate change are the same in air-conditioned as in ordinary fermentation. During the former there is a decrease in phenols due to oxidation accompanied by a change in the colour of the leaf blade to yellow-orange.

E. HOLMES.

Determination of nicotine as $C_{10}H_{14}N_2 \cdot HgI_2$. V. V. ZAPOLSKI (U.S.S.R. State Inst. Tobacco Invest., Bull. 52, 1929, 23 pp.).—Nicotine is precipitated from alkali carbonate solution (15 or 10% solution of potassium or sodium carbonate, respectively) by means of mercuric iodide dissolved in sodium hyposulphite, the washed precipitate reduced in alkaline solution by zinc amalgam, and the iodine determined by Volhard's method. The method is accurate in the presence of large amounts of ammonia, and of pyridine up to 0.05%.

E. HOLMES.

Rumanian coriander oil. E. KOPP (Pharm. Zentr., 1929, 70, 789—791).—Rumanian coriander seeds yield 0.4—0.9% of an almost colourless to dark greenish-brown essential oil. Five representative samples had characteristics within the following limits: d_{20}^{20} 0.873—0.888, α_D^{20} +8.7° to +10.0°, n_D^{20} 1.4642—1.4696, acid value 2.2—4.3, ester value 19.9—30.7, saponif. value 23.7—35.0, acetyl value 163.3—182.1, solubility in 70% alcohol 1 in 2—2½ vols., linalool content (from acetyl value) 51.2—58.0%, (Boulez method) 67.9—74.0%. From the green coriander plants in full bloom was obtained 0.23—0.30% of a green oil with an unpleasant odour and having d_{20}^{20} 0.872, α_D^{20} +0.9°, n_D^{20} 1.4574, acid value 17.0, ester value 35.5, acetyl value 249.9, linalool content (from acetyl value) 81.7%. A specification for coriander oil is suggested.

E. H. SHARPLES.

Hydnocarpus Wightiana oil. II. I. COLE (Philippine J. Sci., 1929, 40, 499—502).—The oil from *Hydnocarpus Wightiana* contains about 1.2% of lauric acid, and also apparently a new, optically-active, liquid fatty

acid, probably a lower homologue of hydnocarpic acid (cf. Power and Barrowcliff. B., 1905, 741).

E. H. SHARPLES.

Reduction of irritation by iodised ethyl esters of *Hydnocarpus Wightiana* oil. H. I. COLE (Philippine J. Sci., 1929, 40, 503—509).—The factors governing the reduction of irritant effects of the ethyl esters of chaulmoogra oil by the addition of 0.5—2% of iodine and the variations in these effects in the iodised esters are discussed. Too long as well as too short a time of heating of the esters with iodine leads to increase in irritant properties, and a method for the manufacture of iodised ethyl esters having minimum irritant properties is described.

E. H. SHARPLES.

Fertilisation of tobacco. EVKO. Nicotine in tobacco seeds. ILIIN.—See XVI.

PATENTS.

Anæsthetics. [Stabilisation of tribromoethyl alcohol.] I. G. FARBENIND A.-G. (B.P. 302,337, 4.9.28. Ger., 16.12.27).—A mixture of, e.g., 2 pts. of tribromoethyl alcohol with 1 pt. of amylene hydrate is claimed to be water-soluble and stable on heating.

L. A. COLES.

Manufacture of valuable photochemical [antirachitic] products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,992, 25.8.28).—Sterols dissolved in pure ethers or in solutions of ethylene or alkali hydroxides in alcohols are subjected to irradiation with ultra-violet rays. The antirachitic properties of the products are greater than when alcohol alone is used as solvent.

L. A. COLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES

PATENTS.

Sensitisation of colloid layers (gelatin, glue, etc.) by treatment with aqueous sensitising solutions, particularly dichromate solutions. N. LEBEDENKO (G.P. 455,046, 31.12.26).—After treatment with the sensitising solution, the layers, while still moist, are given a short treatment in pure water or dilute sensitising solution and dried after removal of excess water. Crystallisation at the colloid surface, which interferes with the penetration of the light, is thereby prevented.

J. W. GLASSETT.

Treatment of exposed chromate-colloidal photographic layers. C. ROEHRICH (B.P. 306,027, 18.1.29. Ger., 14.2.28).—After the copying exposure, the dichromated gelatin or glue film is moistened and given a second short, uniform exposure. [Stat. ref.]

J. W. GLASSETT.

Cleaning gelatinous surfaces [of photographic films]. TECHNICOLLOUR MOTION PICTURE CORP., and B. S. TUTTLE (B.P. 322,173, 23.8.28).—Film matrices impregnated with dye are cleansed by agitation in one or a succession of baths containing an aqueous solution of diethylamine or an aqueous alcoholic ammonia solution containing about 2% of each component.

J. W. GLASSETT.

Printing with dyestuffs photographic gelatin layers. I. G. FARBENIND. A.-G. (B.P. 304,767, 11.1.29. Ger., 27.1.28).—The dye imprint is protected from the action of water or organic solvents used in subsequent treatments by incorporating a gelatin-hardening agent, alternatively with a small quantity of a colloid such as gelatin, in the dye solution, such that the gelatin at the imprinted area is, after a short period of storage, rendered impermeable to these solvents. The following solutions are suitable: (a) para-alkali-blue 2 g., formalin 18 g.; (b) 5% aqueous para-alkali-blue containing 0.7% of gelatin 100 c.c., 10% potassium dichromate solution 32 c.c.

J. W. GLASSETT.

Photographic process printing. H. and T. WECK, and G. VAN BUGGENHOUDT (B.P. 322,007, 3.9.28).—A glass, celluloid, or zinc plate is coated first with a hardened gelatin solution containing gelatin 4 g., 10% chrome alum solution 10 g., potassium silicate 10 g., water 100 c.c., and then, after drying, with a gelatin-casein solution containing casein 300 g., hard gelatin 50 g., water 200 c.c. The dried plate is sensitised by bathing in a 2.7% solution of ammonium dichromate, and when dry exposed under a suitable screen negative, washed in running water for 30 min., and finally immersed in glycerin. Before printing, the surface is moistened with a solution containing glycerin 2 pts., water 1 pt. The surface so obtained possesses much finer depressions than the normal copying plate, and the dot formation is not detectable in the print.

J. W. GLASSETT.

Colour photography. T. T. BAKER (B.P. 322,231, 8.8.28).—Improved colour rendering and reproduction of intermediate tints are obtained in the printing of transparencies in natural colours from master multi-colour screen pictures if the characteristics of the positive film are so chosen that its average grain size is considerably smaller than that of the master film (preferably about one quarter), and its contrast only slightly greater, e.g., a gamma of 1 for the positive film and 0.8 for the master film. It is advantageous to reverse the master film to a positive image and then to project it, using parallel light at right angles to the film, upon the positive film, which in turn is developed and reversed.

J. W. GLASSETT.

Three-colour photography. T. T. BAKER (B.P. 321,998, 28.8.28).—A "tri-pack" comprises (a) a front layer of a red-sensitised, contrasty silver halide emulsion of low green-sensitivity and depressed blue-sensitivity (by addition of yellow screening dye) in front of which is placed a filter containing aesculin, filter-yellow, and mandarin-orange, which serves to absorb the ultra-violet and blue, short wave-length radiations, but transmits the violet; (b) an intermediate layer of a green-sensitised, contrasty emulsion with depressed blue sensitivity; and (c) a rear layer of a rapid, soft, unsensitised silver halide emulsion with depressed green-sensitivity. The ratio of the sensitivities of the front, intermediate, and rear layers to daylight is about 10:6:17. Both the red- and green-sensitised layers are coated very thinly (about one quarter of the normal silver per unit area), but the emulsion characteristics are so chosen that a normal degree of contrast, i.e., similar to that obtainable in the same time with the rear layer,

may be readily obtained. The amount of screening dye used on layers (a) and (b) must not lower excessively the transmission of the violet light to the rear layer.

J. W. GLASSETT.

Preparation of photosensitive photographic emulsions. W. FRANKENBURGER and G. ROESSLER, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,738,530, 10.12.29. Appl., 11.8.28. Ger., 19.8.27).—See B.P. 307,716; B., 1929, 417.

Production of negative colour-record kinematograph films. A. PILNY (B.P. 300,210, 8.11.28. Ger., 8.11.27).

Apparatus for making mosaic-screens for colour photography. H. WADE. From COMP. D'EXPLOIT. DES PROC. DE PHOTOGRAPHIE EN COULEURS L. DUFAY (VERSICOLOR DUFAY) (B.P. 322,454, 4.8.28).

XXII.—EXPLOSIVES; MATCHES.

Hydrocellulose. LEHALLEUR.—See V.

PATENTS.

Compositions for match heads and striking surfaces. I. G. FARBENIND. A.-G. (B.P. 314,852, 3.4.29. Ger., 3.7.28).—The binding agent in compositions for water-resistant match heads and striking surfaces contains a water-soluble albumin, which is rendered insoluble in water during the drying of the composition.

S. BINNING.

Damp-proof matches. W. J. SMITH and A. T. H. PERRY (B.P. 322,545, 5.10.28).—A solution of celluloid in amyl acetate is used instead of gum etc. as binder in the manufacture of matches, and the stems and heads may be dipped in a similar solution. L. A. COLES.

Wrapping up cartridges of plastic explosives and other similar material with paper or the like material. FINSKA FORCIT-DYNAMIT AKTIEB. (B.P. 303,481, 2.1.29. Swed., 4.1.28).

XXIII.—SANITATION; WATER PURIFICATION.

Fumigation with formaldehyde. D. W. HORN and A. OSOL (Amer. J. Pharm., 1929, 101, 744—778).—The yield of gaseous formaldehyde given by fifteen methods of fumigation has been examined. The methods are critically discussed as also is the problem of terminal fumigation. Horn's bleach-formalin method is the most satisfactory for routine disinfection because it combines germicidal efficiency with economy and ease of manipulation. E. H. SHARPLES.

Biological purification of creamery wastes. M. LEVINE (Ind. Eng. Chem., 1929, 21, 1223—1225).—Under anaerobic conditions the lactose present in creamery wastes produces acidities which interfere with the digestion of the protein matter. Under aerobic conditions the lactose is rapidly oxidised, producing effluents which remain neutral after 2 days' storage at 20°. Over 90% of the lactose present was removed in the upper 2 ft. of an experimental trickling filter receiving skim-milk solution containing 1700—3250 p.p.m. of lactose, an extra 0.5—1.0 ft. in depth being required to produce

similar results from cheese wastes containing a lower proportion of nitrogen. Filters from 3 to 5 ft. deep, according to the character of the waste treated, will render this suitable for admission into the public sewer.

C. JEPSON.

Cleaning of filter sand. E. K. VENTRE (J. Amer. Water Works' Assoc., 1929, 21, 1519—1520).—Sand filters may be cleaned *in situ* by treatment with caustic soda or hydrochloric acid according to the composition of the incrustant on the sand. C. JEPSON.

Washing of rapid [sand] filters [for water]. R. HULBERT and F. W. HERRING (J. Amer. Water Works' Assoc., 1929, 21, 1445—1513).—The formation of shrinkage cracks in the sand bed and other attendant filter troubles at an experimental plant at Detroit were overcome by removing a coating of alumina and clay from the sand grains and mud accumulations from the bed. The normal rate of application of wash water (24—30 in./min.) was increased to obtain 50% or more expansion of the sand layer, and this proved effective in preventing coating and shrinkage without impairing the efficiency of the filter. Old filters may be cleaned *in situ* by successive applications of caustic soda and a strong jet of water, at a cost of about 5% of that of the renewal of the sand layer, and may then be kept clean by applying the wash water at a higher rate. A sand-expansion index is suggested instead of the usual rate of vertical rise as the basis on which filter washes should be compared. The extent to which sand expands under these conditions is shown to increase as the rate of wash increases, and to decrease with increase in the temperature of the water and in the size of sand grain employed.

C. JEPSON.

Electrolysis of water. FAUSER.—See XI. **Fermentation of cellulose.** BORUFF.—See XVIII.

PATENTS.

Manufacture of face paint or cosmetic. M. FUKUI and T. MIYAGUCHI (B.P. 322,376, 4.1.29).—A fine white powder free from lead is coated with transparent or translucent matter (nitrocellulose, rubber) added in the form of a solution from which the solvent is subsequently evaporated. Gelatinous zinc hydroxide is then incorporated, and water added to give the desired consistency.

S. S. WOOLF.

Manufacture of toilet material. T. HASHIMOTO (B.P. 322,607, 2.11.28).—A mixture of india-rubber, gum tragacanth, and a volatile solvent is heated under pressure to remove the adhesive property of the gum; the usual skin toilet substances are then added.

L. A. COLES.

Composition for and method of treatment of garbage. E. H. BRUNE (B.P. 322,368, 27.12.28).—See U.S.P. 1,700,608; B., 1929, 266.

Apparatus [automatic base-exchange softener] for treatment of liquids [water]. ELECTROLUX, LTD. From AKTIEBOLAGET FILTRUM (B.P. 321,757, 4.9.28 and 1.7.29).

Base-exchange materials (B.P. 302,690 and U.S.P. 1,736,281).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEB. 14 and 21, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Calculation of tanks for high temperature and pressure. J. K. WOOD (Chem. and Met. Eng., 1929, 36, 737—740).—From a formula for tank design, in which the creep limit of the material is an important functional value, a line chart has been developed to permit a number of tanks to be calculated easily. The approximate weight of a cylindrical tank with elliptical heads is given by the value $0.889tD(l + 3D)$, in which t is the thickness of cylindrical wall, D the internal diameter, and l the length of the cylindrical portion.

C. A. KING.

Accurate measurement of high temperatures. W. E. FORSYTHE (J. Amer. Ceram. Soc., 1929, 12, 780—813).—The theory and practice of all types of high-temperature pyrometry are described, and the possible sources of error and their elimination are discussed in a comprehensive manner.

J. A. SUGDEN.

Measurement of flow of heat. A. F. DUFTON and W. G. MARLEY (Phil. Mag., 1929, [vii], 8, 841—844).—Two instruments are described for measuring the transfer of heat by convection and radiation, respectively, these being simplifications and improvements of that previously described (J. Sci. Instr., 1927, 4, 446).

C. W. GIBBY.

Drying plant. C. H. BUTCHER (Chem. Trade J., 1929, 85, 389—391, 487—488, 619—620).—For tunnel dryers and rotary dryers the safe saturation of the outgoing gases may be taken as 65%, and for chamber dryers 50%. The inlet temperature being fixed, usually by the nature of the material to be dried, the above factors fix the outlet temperature, unless internal heating is used. Velocities of 300—1000 ft./min. are used in tunnel dryers, the limit being that at which dust is entrained. For rotary dryers the maximum gas velocity for most substances is 300—350 ft./min. A table gives approximate relative rates of drying for different gas velocities. The first 80% of moisture is usually removed in 20—25% of the total drying time. Heat loss in outgoing gas is usually 30—40% of the whole unless a heat exchanger is used. Losses due to leakage of hot air in some dryers may reach 25%. The air requirements may be calculated with an error of 1—2% on the assumption that 60,000 cub. ft. of air cooling through 1° F. will evaporate 1 lb. of moisture from the wet material entering at 60° F. The further heat requirements are (a) for heating dry material to dryer temperature, (b) for heating trucks or trays, (c) for compensating for the various losses. The length of a tunnel dryer is best determined by experiment with the material to be dried. If steam is used for heating the air, 1 lb. of low-pressure steam is allowed for the heating

of 3000 lb. of air 1° F. Formulae for heat transmission between steam pipes and air are given, but in practice it is found that the surface-area efficiency of transmission increases as the pipe diameter decreases. If the air current is at right angles to the steam pipes, much greater transference is obtained than if they are parallel. If a low dryer temperature is necessary the air requirements may be very large and filtration may be necessary. This is best performed by washing with calcium chloride solution, which reduces the moisture content at the same time; or filter frames packed with small metallic rings coated with oil may be used. If flue gases are used the material should travel in the same direction as the gas. Consideration should be given to possible effects of flue dust and the alteration in the draught of the chimney concerned.

C. IRWIN.

Fundamental principles in air conditioning. C. A. BULKELEY (Chem. and Met. Eng., 1929, 36, 734—736).—For the control of atmospheric relative humidity, now a necessity in many commercial processes, the air admitted must have a constant dew point and the temperature must remain uniform. The most satisfactory method of conditioning the air supply is to pass it through a spray chamber in which such intimate contact between water and air occurs that both leave the chamber within 1° F. The air has been cleaned and is now practically saturated. Leaving the spray chamber the air is drawn through a baffle chamber and through some form of steam-heated coil from which it is distributed properly throughout the building. It is stressed that all functional changes except starting and stopping should be under automatic temperature or relative-humidity control or a combination of the two.

C. A. KING.

Design, construction, and operation of a constant-humidity room. P. H. PRIOR (Proc. Tech. Sect. Papermakers' Assoc., 1929, 9, 152—174).—The points to be considered in the design of such a room are discussed and details are given for the construction of a room 12 ft. by 15 ft. by 7 ft. 10 in. Provision is made for complete change of air four times per hour or for recirculation of the air over the controls when the room is only used for conditioning samples. Humidity and temperature control are effected by diverting part of the air over the correcting devices (humidifier, refrigerator, and heater) by means of electric fans controlled by a hair hygrometer and a bimetal strip. The controls normally operate at about 5-min. intervals and to within 1% relative humidity and 1° F.

D. J. NORMAN.

Construction materials for handling corrosive agents. A. H. COOPER (Chem. and Met. Eng., 1929, 36, 747—750).—A condensed account, compiled from

* The remainder of this set of Abstracts will appear in next week's issue.

published work, of the resistant properties of materials to the corrosive action of sulphuric, hydrochloric, nitric, and acetic acids, alkalis, and hydrogen sulphide.

C. A. KING.

Spreading of lubricants on solid surfaces. Molecular influences. Rôle of photolysis. P. WOOG (Compt. rend., 1929, 189, 977—979).—Exceptions to the author's rule (B., 1928, 290) for the spreading of lubricants on solid surfaces have been studied. The addition of 0.1% of lauric, oleic, palmitic, stearic, or similar acid to a 1:1 mixture (η_0 1.729) of saturated mineral oil and neutral sheep's-foot oil reduced the spreading power on brass, whilst on steel the long-chain acids were the most effective in this respect. Aromatic acids accelerated the spreading to an extent which was less for the toluic acids than for benzoic acid. The production of 0.1% of acid by ultra-violet or by solar photolysis increased the spreading power, and though the experiments were complicated by the change in viscosity with temperature, there is evidence that the effects of photolysis were continued after irradiation had ceased. J. GRANT.

Viscosity and plasticity. A. V. BLOM (Farben-Ztg., 1929, 35, 601—602).—A brief account of the general significance of viscosity and plasticity, the former being regarded as a limiting case of the latter. Cellulose solutions are plastics whether pigmented or not, but linseed oil does not show yield value (to whatever cooking it is subjected) unless it be pigmented. S. S. WOOLF.

Relation of adhesion tension to "liquid absorption." F. E. BARTELL and O. H. GREAGER (Ind. Eng. Chem., 1929, 21, 1248—1251).—Liquid absorption (Gardner-Coleman) and adhesion tension (cf. Bartell and Osterhof, A., 1928, 12; B., 1928, 1) values of various liquids for powdered silica, carbon black, and calcium fluoride are correlated. For all systems in which the liquid forms a zero contact angle against the solid the relation is linear, the liquid absorption varying inversely with the adhesion tension. If the liquid forms a finite angle of contact against the solid, the liquid absorption is lower than would be expected from the foregoing relation and decreases as the contact angle increases (and the adhesion tension decreases). A theoretical explanation of the observations is advanced.

E. LEWKOWITSCH.

Freezing mixture. F. NOACK (Ind. Eng. Chem., 1929, 21, 1001).—A freezing mixture composed of a mixture of ammonium chloride, sodium carbonate, and water gives a temperature drop of 31°, and is stated to be only one fourth the cost of the customary ammonium nitrate-sodium carbonate composition. Several suitable proportions of the constituents are given; the preferred composition is ammonium chloride 1, sodium carbonate 1.5, and water 3 pts.

C. A. KING.

Absorptive properties of silica gel and charcoal. OKATOV.—See VII. **Water-hammer erosion.** COOK.—See X. **Alternating-current electrolysis of water.** SHIPLEY.—See XI.

See also A., Jan., 31, **Apparatus for dispersoid analysis** (ANDREASEN and others). 55, **Laboratory mercury still** (KUENTZEL). 56, **Hot-wire viscosimeters** (RICHARDSON).

PATENTS.

Furnace port construction. L. N. McDONALD (U.S.P. 1,735,256, 12.11.29. Appl., 27.10.21).—In a regenerative furnace with a number of fuel ports, an air uptake is provided between the ports and terminates in an air port extending above and between the fuel ports.

A. R. POWELL.

Heat-exchange apparatus. F. SCHUBART (U.S.P. 1,734,274, 5.11.29. Appl., 11.6.28).—The heat is conducted from passage to passage by means of wire mesh. The walls of the passages are moulded through the meshes, and the whole is contained in a rigid frame with inlet and outlet ports which are smaller than the apertures through the mesh.

B. M. VENABLES.

Heat exchanger for recovering heat from industrial waste water. F. B. DEHN. From WHITLOCK COIL PIPE Co. (B.P. 322,366, 24.12.28).—The water or other fluid to be heated flows through tubes in a zig-zag manner generally upwards, and the dirty fluid from which the heat is to be recovered flows in an open vessel surrounding the tubes from an inlet at the top of one end to an outlet at the bottom of the other. A number of units may be used in series so as to give a more perfect countercurrent flow.

B. M. VENABLES.

Heat transferers. SCHMIDT'SCHE HEISSEDAMPF-GES.M.B.H. (B.P. 303,172, 28.12.28. Ger., 29.12.27).—A compact element for a heat exchanger is formed by taking two (or more) tubes *A* and *B* and winding *A* over a straight portion of *B*, then *B* over the remaining straight portion of *A*, the ends being joined together to form single inlet and outlet connexions.

B. M. VENABLES.

Calibration of thermostats. H. J. OSBORN and A. WRIGHT (B.P. 322,204, 23.8.28).—The external arm of the thermostat (which operates the control devices) is freed from the arbor of the thermostat proper, but restrained by a pair of set screws in its appropriate position. The thermostat is then raised to the required temperature and, when steady, the arm is clamped to the arbor.

B. M. VENABLES.

Gyratory crushers. J. E. KENNEDY (B.P. 322,690, 26.1.29).—A gyratory crusher has the head of the shaft supported by springs in such a way that, whilst it remains rigid to normal crushing stresses, it can yield to uncrushable pieces.

B. M. VENABLES.

Machines for disintegrating or acting upon materials. A. MUSKER (B.P. 322,571, 1.10.28).—The fixed impact surface of a disintegrator is formed with grooves parallel to the axis and of such a shape that the material flung centrifugally by the rotor enters and slides round them and leaves in a centripetal direction, so that a concussive action takes place between the leaving and entering streams.

B. M. VENABLES.

Pulverising mills. HARTSTOFF-METALL-A.-G. (HAMETAG) (B.P. 306,154, 15.2.29. Ger., 17.2.28).—The material, together with a current of air, enters the pulveriser tangentially to, in the plane of, and counter to the rotation of the beaters. The inlet opening is in the form of a pocket with a bar or other guard across it to intercept particles thrown by the beaters.

B. M. VENABLES.

Pulverising and treating materials. ERIE CITY IRON WORKS, Assecs. of W. J. WOHLBERG (B.P. 313,582, 5.9.28. U.S., 16.6.28).—Various fixed devices are described, suitable for pulverising coal etc. by impact while it is being conveyed in a stream of air or gas. The gas may be heated before impact.

B. M. VENABLES.

Thermal treatment of pulverulent material. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES. M.B.H. (B.P. 305,231, 1.2.29. Ger., 2.2.28).—In the heat treatment of pulverulent material when it is not desired that material should mix intimately with the gases present, it is held in a thin layer on a moving hearth or other surface by means of electric adhesion. The electric charge may be generated by friction devices on the travelling surface.

B. M. VENABLES.

Drying system. J. PÖSSNER (B.P. 321,921, 16.6.28).—A kiln suitable for drying bricks, timber, etc. is constructed with a pair of parallel walls which are preferably circular so that the kiln is annular, and is subdivided into a number of compartments by transverse walls which do not extend completely across the kiln. Fans and heaters are arranged in alternate compartments and maintain a large local circulation of air with a minor zig-zag transference. The main walls of the kiln consist substantially of hinged flaps which can be opened at suitable points for charging and discharging the material and for admitting and exhausting air. Additional heated air may be admitted to any compartment from an underground channel.

B. M. VENABLES.

Apparatus for drying broken stone, slag, etc. J. SMART (B.P. 322,274, 2.10.28).—The material is allowed to slide down a series of adjustably sloping floors composed of louvred plates or slats. Hot air is supplied to chambers below the floors and allowed to exhaust freely to atmosphere above them.

B. M. VENABLES.

Rotary drum machines for treating granular materials. H. BEHRENS, and MASCHINEN- U. WERKZEUGFABR. KABEL VOGEL & SCHEMMANN A.-G. (B.P. 322,405, 26.2.29).—A machine for purposes such as reconditioning foundry sand is constructed with a rotor having ratchet-shaped teeth which fling the material against a pair of screens behind each other, the wires being staggered so that no particle can pass straight through both screens without impact. The rotor may be built round an electric motor having an internal stator.

B. M. VENABLES.

Controlling the characteristics of spray-processed products. D. R. LAMONT, Assr. to INDUSTRIAL SPRAY-DRYING CORP. (U.S.P. 1,734,260, 5.11.29. Appl., 3.10.27).—The liquid material is sprayed into heated air and the conditions are carefully regulated so that steam is evolved while the drops are in a viscous condition, the result being formation of hollow shells.

B. M. VENABLES.

Separation of dry materials. BIRTLEY IRON CO., LTD., I. L. BRAMWELL, and C. W. H. HOLMES (B.P. 322,480, 6.6.28).—Substances, such as a mixture of coal and shale, are treated pneumatically so that one product comprises the largest particles of the heavier constituent. The remainder is then screened so that the largest

particles of the lighter constituent are obtained in the pure state, the remainder being then treated in any known way. During the process middling products may be returned to the feed.

B. M. VENABLES.

Centrifugal machine. LAUGHLIN FILTER CO., Assecs. of W. C. LAUGHLIN (B.P. 310,504, 27.8.28. U.S., 28.4.28).—The machine is of the screening type with helical scraper to remove the collected solids continuously, the shafts of the basket and scraper being concentric. The edges of the scraper blades can move outwardly by centrifugal force; they rub only on supporting ribs, and not on the screen.

B. M. VENABLES.

Continuously-operating centrifugal sludge separator. A. NYROP (B.P. 302,685, 6.12.28. Denm., 20.12.27).—The bowl has a peripheral slot for discharge of collected solids and can slide vertically on the driving hub. Below the bowl, fixed to the hub, is a dish-like member with upwardly extending flange which closes the slot in the bowl when the latter is at the lowest position. The feed channel for raw liquid is annular, but on the axis is another channel for admitting hydraulic liquid to the space between the bottom of the bowl and the dish below, so that when liquid is admitted the bowl floats and opens the peripheral slot; this same motion also permits discharge of the hydraulic liquid at the periphery so that the slot closes again ready for a fresh accumulation of sludge.

B. M. VENABLES.

Separation of solids from liquids. A. SCHUSTOV (B.P. 322,100, 1.12.28).—The liquid is settled in "flooding troughs" which are constituted by conveyer belts running against the flow of liquid, and removing the settled solid matter to a point behind the feed box, where discharge is effected by scrapers or sprays. The liquid discharge from one conveyer may fall upon another running in the opposite direction, so as to get a series of depositions. The conveyer belt may have upturned edges or may be supported on waisted rollers so that the weight of the material will cause the upper surface of the belt to be concave.

B. M. VENABLES.

Separator. F. A. BROWNE, Assr. to BARBER ASPHALT CO. (U.S.P. 1,734,324, 5.11.29. Appl., 13.2.28).—A mixture of liquid and vapours is admitted to the upper part of a closed chamber after passing through a long, narrow, spiral tube adapted to effect centrifugal separation. The upper outlet for vapours is protected by a foam separator, and the lower outlet for liquid is heated.

B. M. VENABLES.

Centrifugal dome with condensing arrangement. C. SCHMITZ, Assr. to RAMESOHL & SCHMIDT A.-G. (U.S.P. 1,734,533, 5.11.29. Appl., 3.10.28. Ger., 10.9.27).—The air currents induced by the discharge of a separated liquid through nozzles on the centrifuge cause a return circulation of fine spray and vapour inwards under the cool, domed roof of the catch chamber, between the roof and an adjacent baffle.

B. M. VENABLES.

Cleaning the discs of centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 308,828, 28.3.29. Swed., 31.3.28).—The bundle of separating discs or cones is taken out of the separating bowl and placed in an apparatus where they are subjected to a strong circulating current of cleansing fluid, so arranged that the flow is

radial between the discs. The circulation of the fluid may be effected by means of steam jets, and the apparatus may be enclosed for working under pressure.

B. M. VENABLES.

Mechanical filters. S. J. RICKS and C. E. DOUGLAS (B.P. 322,148, 2.4.29).—The whole mass of sand, or similar medium, of the filter is cleaned by a device comprising several tiers of horizontal arms which can be rotated intermittently in horizontal planes, each arm carrying a number of stirrers which rotate in vertical planes, in opposite directions in alternate tiers. The arms are hollow and terminate in water jets.

B. M. VENABLES.

Filters for liquids under pressure. G. and S. JAVITCH (B.P. 309,143, 4.4.29. Fr., 5.4.28).—A filter, of the type where a filter medium and a joint ring are clamped between two halves of a casing, is provided with a pin and slot to prevent relative rotation of the halves, and the slot is extended by a recess to allow any liquid that passes round the edge of the filter medium to leak out, thus indicating that the clamping pressure is insufficient.

B. M. VENABLES.

Apparatus for filtering liquids under pressure. H. JUNG (B.P. 322,091, 27.11.28).—The filter is composed of a number of tubular filtering elements contained in a pressure-tight casing. During the filtering period the prefilter is caused to sweep over the full length of the tubes, some prefilter being allowed to escape at the end of the casing remote from that at which it entered. After the cakes have been formed the excess prefilter is blown out and the cakes are dried by air at a pressure in excess of the filtering pressure. Lixiviation or washing may then take place by means of fluid under pressure. After a final drying by air, the bottom (inlet) end of the casing is removed and the dry cakes are blown off by reverse compressed air. The tubular filters may be constructed of a number of ceramic rings assembled round a hollow clamping bolt which forms the outlet for filtrate. They may be strengthened by sprayed metal or other substances on their abutting surfaces.

B. M. VENABLES.

Fluid segregator. D. SAMIRAN and P. MELVILLE (B.P. 322,654, 17.12.28).—The apparatus comprises a float chamber in which water may settle out of, e.g., petrol (or for similar purposes); the needle of the float valve controls the outlet of water, whilst the current of petrol flowing in and out may be continuous. Devices are incorporated to prevent a sudden rush of water from choking the supply of petrol, for adjusting the float to the correct buoyancy, and to afford a resilient connexion between the float and needle valve.

B. M. VENABLES.

Cleaning of filters or strainers. E. FOLTA (B.P. 308,974, 28.3.29. Czechoslov., 2.4.28).—A vacuum cleaner device which travels over the filtering surface is described.

B. M. VENABLES.

Protection of boilers against scale formation. SIEMENS-SCHUCKERTWERKE A.-G. (G.P. 455,983, 12.12.25).—The water is not only heated by the usual methods for steam raising, but also locally by the introduction of hot gases or vapours or by means of an electric arc working under its surface and remote from the side walls of the boiler.

A. R. POWELL.

Distillation of liquid materials. J. PINTSCH A.-G. (B.P. 297,122, 14.9.28. Ger., 16.9.27).—The distillation of a liquid (e.g., hydrocarbon oil) is effected by a constant circulation of a carrier gas (e.g., the permanent gas derived from the oil) which is all heated by passing through one half of a regenerator; then the greater part passes through a distillation chamber preferably situated in a hollow of the wall between the halves of the regenerator where the oil to be distilled is added to the stream. The circulating mixture passes to a first separator where remaining liquid is caught, and then to a column where the desired fraction is condensed; the cool carrier gas is pumped back to the same half of the regenerator (until it becomes too cold), and since an excess of gas will be produced by the distillation, this excess is passed to the other half of the regenerator and burned therein with air, heating it up until the next change-over. The distillation being effected in the presence of a large proportion of gas, the partial pressure of the vapours is low, thus giving a result comparable with the use of vacuum or steam.

B. M. VENABLES.

Separation of liquids by distillation. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 304,756, 12.12.28. Belg., 26.1.28).—A mixture of liquids, one of which is very soluble in water and the other only slightly so, is distilled in a column which is provided with a water spray at the top, the water being supplied at the temperature of the azeotropic mixture of the slightly soluble liquid and water. The heat is supplied at the bottom of the column, being preferably assisted by heating the feed which is supplied to the middle of the column. The very soluble liquid is withdrawn from the bottom in the form of a solution diluted with water. The vapours from the top of the column are condensed and settle into two layers, the lower of which, mostly water, is returned to the column. The upper layer will be the slightly soluble constituent mixed with a little water, but will not contain any of the very soluble constituent. It is claimed that mixtures of ethyl acetate, alcohol, and water; acetal, alcohol, and water; isopropyl ether and isopropyl alcohol, can thus be separated. (Cf. B.P. 305,528; B., 1930, 135.)

B. M. VENABLES.

Degasification of liquids. W. S. ELLIOTT (U.S.P. 1,734,515, 5.11.29. Appl., 19.11.23).—The liquid is sprayed over heated tubes and falls to a separating space below; the vapours and permanent gases pass through a surface condenser which is cooled by the raw liquid. The latter may be further preheated in a separate heater before admission to the degasifier proper.

B. M. VENABLES.

Gas and liquid separator. L. L. WESTLING and M. R. HICKMAN (U.S.P. 1,734,507, 5.11.29. Appl., 23.10.28).—The mixed fluids are caused to flow upwards through a vertical pipe. Whirling is produced by vanes at the bottom, and the top is surmounted by a dome from the extreme top of which the gas is withdrawn, the liquid exhausting smoothly through a number of holes lower down. If the flow is too slow to produce a vortex, separation will be effected by simple settlement.

B. M. VENABLES.

Method of absorption [of gas in liquids]. J. A. CAMPBELL (U.S.P. 1,736,070, 19.11.29. Appl., 19.4.27).

—The liquid and gas are continuously introduced through spray jets into the lower part of a reaction vessel so as to form a voluminous froth which is broken down by a paddle device at the top of the reaction vessel.

A. R. POWELL.

Introducing liquid or semi-liquid materials into high-pressure vessels. S. R. BOYCE, W. R. TATE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 322,645, 7.12.28).—Material such as a mixture of oil and coal is pumped by two plunger pumps in series, and between them is an intermediate receiver comprising a cylinder with differential piston, to the smaller side of which the paste is admitted, whilst the larger side is subjected to the pressure of a gas, which may be the same as that in the high-pressure vessel to be charged.

B. M. VENABLES.

Dust filters. BÜHLER BROS. (B.P. 317,078, 25.7.29. Ger., 10.8.28).—The filter is of the bag type with mechanical shaking. The lower ends of the bags are not secured to anything but are weighted; the upper ends are supported from a frame which at intervals is raised by a cam and allowed to fall on a spring cushion.

B. M. VENABLES.

[Stream-line gas] filters. F. E. SQUIRE. From MIDWEST STEEL & SUPPLY CO., INC. (B.P. 322,684, 23.1.29).—The filter comprises a number of spaced plates all bent equally into hyperbolic shapes, resulting in a stream-line flow, with a certain amount of expansion and contraction between them. The outlet edges of the plates are provided with collecting pockets which extend (transversely to the gas currents) outside the casing so that the settled dust may be collected without stopping the action. The pockets are preferably formed by bending back the edges of the plates.

B. M. VENABLES.

Solidification of gases. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 301,741, 3.12.28. Ger., 2.12.27).—The process when used for the solidification of carbon dioxide is worked as follows: Liquid carbon dioxide at cold-water temperature and 58 atm. is passed through one passage, say the inner, of three heat-interchangers (*A*, *B*, *C*) in series. Between *A* and *B* a portion (about one third) of the liquid is expanded to a pressure just above the triple point, and the cold gas is passed through the outer of *B*, thus reducing the temperature of the liquid remainder to just above the f.p. (-50°); after further cooling in *C*, the liquid is expanded through a valve into the freezing chamber at 1 atm., where the kinetic energy of the issuing carbon dioxide is utilised to cause the solid to form in lumps. The gas at atmospheric pressure passes through the outer of *C* and *A* to atmosphere. The gas (at 7 atm.) from the outer of *B* also passes through a separate outer of *A* and passes to the second or third stage of the compressor. The theoretical (adiabatic) yield of solid is increased to 61.5% from 39% by the ordinary process. A further reduction of power can be effected by working the final expansion not to atmosphere, but to a pressure slightly below the triple point, and returning the exhaust gases to the compressor. Several freezing chambers, used intermittently, are preferably provided for one set of heat exchangers, used continuously.

B. M. VENABLES.

Pressure vessel for transport and evaporation of liquefied gases of low b.p. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 322,702, 25.2.29).—The vessel is constructed of an austenitic, non-magnetic, alloy steel containing such components as manganese, chromium, or nickel in such small amounts that the crystals are of a single type. Such steel, in addition to its already known low contraction, is found to resist impact and transpiration.

B. M. VENABLES.

Apparatus for [cooling and] conditioning air. L. HELMER (U.S.P. 1,735,611, 12.11.29. Appl., 31.1.29).—Cold water is sprayed into the stream of air in a dehumidifying chamber with spray trap and collecting sump for the water; the latter is drawn into an evaporator, where it is cooled by a high vacuum produced by a mechanical pump and steam-jet augments. The vapours may be condensed and, together with the cooled water, used again.

B. M. VENABLES.

Apparatus wherein gases are passed over solids. W. R. WOOD (B.P. 322,601, 29.10.28).—In a rotating heat recuperator of the type where comminuted solid matter is contained in an annular chamber subdivided into sectors, through some of which a hot gas is passed axially while simultaneously through others a gas to be heated is passed, the sectors are provided with radial guides which do not extend to the outer and inner circumferential walls, and the heat-absorbing material does not completely fill the sectors. Thus, on rotation, the material will turn over and over, thereby freeing itself from dust which leaves through perforations provided preferably through the inner circumferential wall.

B. M. VENABLES.

Controllably feeding gases [from containers containing them in liquid phase]. J. M. ROWLAND, ASSR. to HOOKER ELECTROCHEM. CO. (U.S.P. 1,736,509, 19.11.29. Appl., 7.1.27).—The gas is withdrawn from the bottle as a liquid and vaporised in a coil submerged in a bath held at a constant temperature by a thermostat. The bottle is also submerged in a bath of which the temperature is raised as the gas is withdrawn, so that the pressure is held constant.

B. M. VENABLES.

Measuring the turbidity of liquids. W. PATERSON. From D. J. MIDDENDORP (B.P. 322,461, 4.9.28).—A trough has its vertical walls lined with mirrors which are not quite parallel. A constant source of light is situated behind a transparent aperture in one side near one end, and a view is taken through a hole in the opposite side a little further along the trough. A number of virtual images of the aperture will appear on the mirror opposite this hole, and in the presence of a turbid liquid these images will gradually get fainter, the number of images remaining visible being inversely as the turbidity of the liquid.

B. M. VENABLES.

Anti-freezing mixtures for use in radiators. GES. F. KÄLTECHEMIE GES.M.B.H., and II. SAUTIER (B.P. 322,859, 2.11.28).—An aqueous solution containing aluminium and calcium chlorides is claimed.

H. ROYAL-DAWSON.

Production of artificial fog. HANSEATISCHE APPARATEBAU-GES. VORM. L. VON BREMEN & CO. (B.P. 298,980, 11.9.28. Ger., 18.10.27).—Suitable organic

substances, *e.g.*, formic or oxalic acid, are brought into contact with the fog-forming acid (*e.g.*, sulphur trioxide dissolved in chlorosulphonic acid), whereby decomposition and dehydration occur immediately, the gas is liberated, and the necessary pressure produced to atomise the materials.

H. ROYAL-DAWSON.

Heat-exchanging device. C. McNEIL (U.S.P. 1,740,192, 17.12.29. Appl., 11.12.28. U.K., 11.7.28).—See B.P. 313,780; B., 1929, 739.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coking tests on coals from Western Canada.

R. E. GILMORE and R. A. STRONG (Canada Dept. Mines Fuel Invest., 1927, No. 696, 12—31).—Nine bituminous coals (20—30% of volatile matter) were examined. The investigation was conducted in two stages, *viz.*, "box" coking tests in full-scale by-product ovens, followed by carbonisation tests in a special laboratory apparatus. It was found that three of the nine coals examined were suitable, individually, for the production of high-quality coke, gas, and by-products. The remaining six coals each produced a satisfactory domestic coke when blended with a high-quality (imported) coking coal; for use in plants where no blending facilities are available a considerable further reduction in ash content is necessary before any of these (six) coals can be utilised. All nine coals were characterised by low sulphur contents (0.4—0.9%) and comparatively high ash-fusibility temperatures.

C. B. MARSON.

Coke as a domestic heating fuel. P. NICHOLLS and B. A. LANDRY (Fuel, 1929, 8, 588—600).—Coke at present forms about 6% of the total solid fuel used in the United States for domestic purposes. The general characteristics of coke as a fuel for central-heating are discussed. Tests carried out with an experimental furnace of the type used for central-heating have shown (a) that the draught required to give a fixed rate of burning decreases rapidly with increase in the size of the coke pieces; (b) that the draught required increases almost linearly with the rate of burning; (c) that the carbon monoxide content of the gases rising from the fuel bed, averaged over the period between firings, and consequently the secondary air required, decreases with increase in the size of the coke pieces. With small coke ($\frac{1}{2}$ —1 in.) it is easier to start the fire, which is also less likely to go out; the furnace is more easily controlled (by adjustment of the dampers) and firings need not be so frequent as with larger coke. On the other hand a greater draught is required, and a hotter fire may be obtained, with consequent clinker trouble; moreover, the efficiency is lower. Low-temperature coke ignites and burns more readily and requires less draught than high-temperature coke. It requires, however, more secondary air for complete combustion. The efficiency with low-temperature coke tends to be lower on account of the higher temperature of the flue gases and the tendency for unburnt volatile matter to pass to the chimney.

A. B. MANNING.

Use of gas and by-product cokes for domestic heating purposes. E. S. MALLOCH and C. E. BALTZER (Canada Dept. Mines Fuel Invest., 1927, No. 696, 5—11). The chief differences between the two cokes in common

use, *viz.*, gas and by-product cokes, are outlined. Types of domestic central-heating furnaces most frequently used in Ontario and Quebec are described, and instructions are given for the utilisation of coke in these appliances.

C. B. MARSON.

Production of a strong semi-coke from a poorly caking or non-caking coal by the addition of ordinary or oxidised low-temperature tar. F. FISCHER, A. PRANSCHKE, and H. SUSTMANN (Brennstoff-Chem., 1929, 10, 480—487).—A hard, coherent coke has been obtained by carbonising a non-caking coal (Brassert), mixed with its own low-temperature tar, at 500—600°. It was necessary to mix the tar with the finely-ground coal as uniformly as possible in the proportion of at least 1 : 1, and to avoid a too rapid rate of heating over the range 300—400°. About 86% of the tar was recovered; this was much richer in lighter-boiling constituents than the original tar, and had lost the power of forming a coherent coke with the coal. This power, however, was restored by gentle oxidation, *e.g.*, by blowing air through the tar at 60—70°, or preferably at higher temperatures.

A. B. MANNING.

Determination of mineral matter in coke. K. HRADSKÝ (Paliva a Topeni, 1929, 11, 30—32).—The relation between the true mineral content of coke and the composition of the ash is discussed. If the coke contains little sulphur and no sulphate the ash is free from sulphate. Sulphide-sulphur may be removed by treatment with acid. Sulphate in the ash is not derived from organic sulphides. The sulphur in the ash is unchanged by addition of lime to the coke. The coke (0.5—1 g.) is treated in a porcelain boat with dilute acetic acid (3—5 c.c.), dried, and ashed in air or oxygen at 700—800°; the ash is reduced with dry hydrogen at the same temperature, and cooled in hydrogen. After further treatment with acetic acid and reduction in hydrogen the ash is weighed. The iron in coke is present as the element or as ferrous sulphide.

CHEMICAL ABSTRACTS.

Use of a portable calorimeter in the retort house. G. G. BUTTERY (Gas J., 1929, 188, 840—841).—For control of individual streams of coal gas which are subsequently mixed, a small water-flow calorimeter is used. A gas sample is taken into a large aspirator, and 0.1 cub. ft. is employed for the calorific value determination. The whole outfit can be made portable and is easily operated; examples are given of its applications.

R. H. GRIFFITH.

Destructive distillation of coconut shells and oil palm nut shells. C. D. V. GEORGI and T. A. BUCKLEY (Malayan Agric. J., 1929, 17, 398—402).—Coconut shells give a higher yield of charcoal than oil palm nut shells and these, in turn, than hardwoods. Both shells yield hard, brittle charcoals, palm nut charcoal having the glossier fracture and a remarkably high sp. gr. (d 1.47; coconut charcoal has d 1.17—1.21). Coconut shells form a valuable source of supply of acetic acid, creosote, and wood spirit, recoverable from the pyro-ligneous acid.

E. LEWKOWITSCH.

Regeneration of norit. S. S. KUTZEV and M. R. KOTLYARENKO (Zhur. Sakh. Prom., 1928, 2, 236—238).—Exhausted norit (ash 17.5%, mineral oils 2.03%) was

treated with ether to remove the oils, and washed with 2% sodium hydroxide solution (6 times) and 2% hydrochloric acid (once); the decolorising power was restored to 69% of the original. Regenerated norit, after being heated in carbon dioxide and washed once each with sodium hydroxide solution and hydrochloric acid, had a decolorising power 88% of the original.

CHEMICAL ABSTRACTS.

Complete gasification plant at Mill Hill. F. J. PEARCE (Gas J., 1929, 188, 580—585).—Modifications of an existing water-gas plant are described which made it available for gasification of coal. A new plant evolved from it has been constructed and successfully operated for several months, using coal only. It consists of a generator with superimposed carbonising chamber, carburettor, and superheater, and is fitted with the back-run device and with a circulating fan. The coal is carbonised by the sensible heat of superheated steam mixed with water-gas; maintenance of the optimum temperature in the cracking vessels and in the circulated gas is very important and requires more supervision than is given to the ordinary water-gas set. No difficulties were encountered in enrichment of the gas by means of oil, but the quality of the tar produced is uncertain and the ammoniacal liquor formed is too weak to be of any use. Figures are given for a variety of working conditions, and it is stated that 172.2 therms of gas are obtained per ton of coal; this is compared with 162 therms in a normal two-stage process, but no data for steam consumption are included. The capacity of a plant giving 1 million cub. ft. per day with coke is reduced to about $\frac{2}{3}$ million with coal.

R. H. GRIFFITH.

Gas drying and producer practice. F. FIRTH (Gas J., 1929, 188, 657—658; cf. B., 1926, 474).—A rough calculation shows that to dehydrate gas saturated at 20° to a dew point of 2° by refrigeration would require about 21 tons of ice per 5000 cub. ft. The most suitable arrangement for carrying out the process consists of a washer filled with a mineral oil, *e.g.*, gas oil, having a low viscosity at low temperatures, and cooled by refrigeration. The naphthalene in the gas is thereby removed at the same time. The quality of the gas from a producer of the open step-grate type improved with increased velocity of gas through the fuel bed, but remained unchanged when the depth of the fuel bed was decreased by allowing the clinker to accumulate to a depth of 2 ft. The carbon dioxide content of the gas is high immediately after clinkering, but diminishes as a layer of clinker forms; this improvement in quality of the gas is attributed to the increased velocity of the air passing through the interstices of the clinker. Some further effects of clinker formation on the performance of a gas producer are briefly described.

A. B. MANNING.

Recovery of by-product nitrogen and sulphur in the coke-oven and gas industries. F. MUHLERT (Brennstoff-Chem., 1929, 10, 487—490).—The following processes for the removal of hydrogen sulphide and/or ammonia from coal gas are briefly discussed: (1) the "Seaboard," "Ferrox," "Thylox," and "Nickel" processes of the Koppers Co. (cf. B., 1928, 357; 1929, 8 *et seq.*); (2) the processes of the Ges. f. Kohlentechnik

(cf. Ferbers, B., 1929, 5); (3) Bähr's process (B., 1928, 354); and (4) Petit's process (cf. Lorenzen, B., 1929, 766).

A. B. MANNING.

Economics of benzol recovery at gas works. F. FIRTH (Gas J., 1929, 188, 595—596).—The thermal aspect of stripping benzol from coal gas and, if necessary, replacing it by oil gas is discussed. A hypothetical cost sheet has been prepared and the various items involved are inspected.

R. H. GRIFFITH.

Determination of sulphur in spent [coal] gas-purification material. A. RINCK and E. KAEMPF (Gas- u. Wasserfach, 1929, 72, 1269—1270).—The material (10 g.) is extracted with carbon disulphide and the extract made up to 100 c.c.; 10 c.c. of this are heated on a water-bath until all the solvent is removed, and the residue is heated at 100° for 2 hrs. with 15 g. of caustic potash and 20 c.c. of water. The whole is then diluted to 200 c.c., filtered, and one tenth of it is mixed with excess of bromine water; the sulphuric acid formed is determined as barium sulphate.

R. H. GRIFFITH.

Carburettng process in water-gas manufacture. A. G. GRANT (Gas J., 1929, 188, 590—593).—The types of oil available for enrichment of water-gas are described and the manner in which cracking conditions affect operating results is shown in a review of recent work on the subject. The difficulty of exact measurements on the plant is emphasised, but it is shown that the efficiency of oil cracking is easily under control in practice. Possible future sources of oils for enrichment are surveyed and the general trend of plant design is discussed.

R. H. GRIFFITH.

Tar works' emulsions. R. G. W. EADIE (Gas J., 1929, 188, 658—660).—Emulsions are discussed under the headings: (a) those formed between the crude tar and liquor, and in the extraction of the tar acids; (b) creosote emulsions used as disinfectants; and (c) road-tar emulsions. The water content of MacLaurin tar can be reduced from 10% to 2% by centrifuging. The agents producing the emulsions formed during the extraction of the tar acids are probably the free carbon in the tar, calcium carbonate in the caustic soda, and other finely-divided solids. Creosote emulsions are more effective as disinfectants than the solutions obtained when excess of soap is used, probably because the bacteria are adsorbed on the particles of the emulsion and thereby exposed to the action of the concentrated creosote. Some advantages of the use of an emulsified pitch, such as the French preparation "Arcite," for road-surfacing purposes are discussed.

A. B. MANNING.

Assay of bituminous sands. R. E. GILMORE, A. A. SWINNERTON, and G. P. CONNELL (Canada Dept. Mines Fuel Invest., 1927, No. 696, 83—103).—A number of laboratory tests for the examination of bituminous sands have been examined and the following methods are recommended: (1) Determination of bitumen.—A sample ($\frac{1}{4}$ — $\frac{1}{2}$ lb.) is air-dried to constant weight; the water content of the air-dried sample is determined by the toluene method, in which 100 g. of the sand are weighed into a 500-c.c. round-bottom flask, fitted with reflux and graduated receiver, 150 c.c. of toluene

are added, and heating is continued until no more water is collected. The extraction with solvent is effected by centrifuging 0.5–1 kg. of the sample with benzol in a Dulin–Rotarex centrifuge; further quantities of benzol are added until the solvent comes out colourless; the sand is dried to constant weight at 105°, and, after being kept overnight, the solvent, with its extracted material, is decanted, the sediment is transferred to a filter paper, washed with benzol, dried, added to the main bulk of the sand, and weighed. The total loss by extraction minus the moisture content represents the bitumen content. (2) Determination of sulphur.—A 50-g. sample is placed in a glass-stoppered graduated cylinder and the volume made up to 250 c.c. with benzol; after shaking, the sandy material is allowed to settle and 10 c.c. of the supernatant liquid are transferred to a Parr peroxide bomb, from which the solvent is evaporated by placing the bomb in a specially adapted steam-bath; the potassium chlorate accelerator is added, mixed with the bitumen, sodium peroxide is then added, the bomb is exploded, and the sulphur determined in the usual way. (3) Destructive distillation of bitumen.—1 kg. of bituminous sand is extracted with benzol as in (1), the solvent-free bitumen is weighed and transferred to a cast-iron retort (capacity 1000 g.) which is connected to condensers, receivers, and a gas-meter. Distillation is stopped when no further oil distils off; the distillate is weighed, the water separated from the oil, and the yield of dry oil obtained; the pitch residue is also weighed and the weight of the gas calculated from its volume and analysis. Then 100 c.c. of the distillate are subjected to fractional distillation in a 250-c.c. Engler flask, temperature readings are taken at every 5% distilled over, and the receiving vessels changed at 150° and 300°. The amount of pitch remaining is weighed. The three fractions are examined for sp. gr. and for unsaturated compounds by treatment with sulphuric acid. C. B. MARSON.

Asphalt emulsions for highways. H. NOVÁK (Paliva a Topeni, 1929, 11, 4–11).—Laboratory tests of asphalt made by the cold process are recorded.

CHEMICAL ABSTRACTS.

Testing of parquet asphalt. H. NOVÁK (Paliva a Topeni, 1929, 11, 17–20).—The deflexion test for asphalt to be used as a base for parquet floors is described.

CHEMICAL ABSTRACTS.

[Preparation of] anti-knock gasoline by cracking various products at the atmospheric pressure. V. F. GERR (Neft. Choz., 1928, 15, 343–351).—When cracked at atmospheric pressure, a mixture of dimethyl-, trimethyl-, and ethyl-benzene (fraction b.p. 122–200°) gave good results; toluene was more resistant and formed carbon. With copper as catalyst, more tar was produced. Green oil and paraffin wax, m.p. 60°, gave a low yield, whilst Surakhani fuel oil and crude oil behaved similarly to kerosene distillate. Cracked kerosene, b.p. 150–300°, gave slightly lower yields than normal kerosene; the unsaturated hydrocarbons were readily converted into aromatic compounds. Over the temperature range 670–700°, the highest yields of aromatic compounds were obtained when the charging rate was kept within certain limits. The gasolines were

of high anti-knock quality, containing up to 25% of aromatic hydrocarbons. As compared with cracking under increased pressure, the quantity of gas obtained was trebled.

CHEMICAL ABSTRACTS.

Rectification of gaseous products from crude oil cracked in the vapour phase. M. B. MARCOVICH (Neft. Choz., 1929, 16, 67–73).—Gasoline obtained in the "blau gas" cracking process had d_{40}^{25} 0.725 and absorbed up to 60 vol.-% of gas; it contained propylene 2, butylene 19, C_5 hydrocarbons (up to 90% unsaturated) 28, C_6 hydrocarbons (at least 85% unsaturated) 22, benzene 17, higher polymerides 12%, and some divinyl and butane. The composition of the absorbed and permanent gases is recorded. CHEMICAL ABSTRACTS.

Sakhalin crude oil from Katangli. S. S. NAMETKIN and L. N. ABAKUMOVSKAYA (Neft. Choz., 1929, 16, 527–529).—The characteristics of the oil are recorded.

CHEMICAL ABSTRACTS.

Vacuum distillation of the typical Baku fuel oils. V. GURVICH and B. KAMINER (Azerbeid. Neft. Choz., 1929, No. 5, 64–70).—Distillation data for paraffin-, naphthene-, asphalt-, and mixed-base crude oils are recorded.

CHEMICAL ABSTRACTS.

Oxygen values of asphaltenes and resins in crude oils. S. S. NAMETKIN and V. G. PUTZILLO (Neft. Choz., 1929, 16, 230–235).—The discrepancies in the coefficients of unsaturation determined by iodine and oxygen values, respectively, are due to asphaltenes. Perbenzoic acid, but not iodine, reacts with the resins and asphaltenes. The results are unaffected by the small amounts of sulphur compounds present in the oil.

CHEMICAL ABSTRACTS.

Action of sulphuric acid on gas from cracked kerosene. V. F. GERR and M. M. TKHOMIROVA (Neft. Choz., 1929, 16, 411–414).—The gas (heavy hydrocarbons 33.6, CH_4 49, H 17.4%), after treatment with sulphuric acid, contained: heavy hydrocarbons 24, CH_4 56, H 20%, 0.3 g. being absorbed from each litre. Acid recovered from sludge was more active than pure acid. The composition of the oil layer formed on the acid and its behaviour on fractionation are described.

CHEMICAL ABSTRACTS.

Cylinder oils from heavy bottoms. L. GUKHMAN (Azerbeid. Neft. Choz., 1929, No. 5, 70–72).—Excessive polymerisation in the treatment of heavy lubricating oil bottoms with acid is avoided by dilution with gas oil. The characteristics of the oil fractions and of asphalts from the acid sludge are recorded.

CHEMICAL ABSTRACTS.

Utilisation of acid sludge from lubricating oil bottoms. S. N. OBYADCHIKOV (Neft. Choz., 1929, 16, 80–82).—The acid in the sludge from Grozni lubricating oil is partly removed by settling; the remainder is neutralised with lime and the mixture is extracted with gasoline at 50–80°. Alternatively, the sludge is blown with air for 15 min. at 185–218°, affording a brittle product having softening point 113° and being suitable for use as pulverised fuel.

CHEMICAL ABSTRACTS.

Asphalt tar from Grozni mixed-base crude oil. A. I. VORONOV and V. S. TVERTZIN (Neft. Choz., 1929, 16, 242–246).—Heavy residues (characteristics recorded) were agitated with superheated steam (320°) and heated

at 350—375°. The properties of the asphalt obtained (optimal d_{20}^{20} 1.003) and of products recovered are recorded.

CHEMICAL ABSTRACTS.

Determination of sp. heat of crude oil products at elevated temperatures. V. I. TIKHOMIROV and V. P. ZHUSE (Nef. Choz., 1929, 16, 74—79).—The following values were obtained: kerosene distillate, b.p. 26.84—144.10°, 0.4418—0.5936; gas oil, d 0.8649, 29.67—204.00°, 0.434—0.6293; gas oil, d 0.8845, 24.41—196.32°, 0.4226—0.6367; gas oil, d 0.8916, 24.13—221.94°, 0.4307—0.6603.

CHEMICAL ABSTRACTS.

Prevention of knocking of gasoline. Y. NAGAI (J. Fuel Soc. Japan, 1929, 8, 109—111).—The addition of small amounts of anti-knock agents to a gasoline raises the temperature of flame propagation in its vapour, and narrows the inflammable limits. These effects are considered sufficient to explain the successful use of these substances with higher compression ratios.

R. H. GRIFFITH.

Amyl acetate from aviation gasoline. A. DOBRYANSKI and E. GUREVICH (Nef. Choz., 1928, 15, 532—559).—The pentane fraction (about 20%) of Krasnodar aviation gasoline, after redistillation between 31° and 38° (11.5—12.5% of gasoline, d 0.6332, mol. wt. 69—70.5), was chlorinated in artificial light; the highest yields of monochloride were obtained by chlorination in the gas phase with dry chlorine at 60—70°. A yield of about 90% of crude amyl acetate was obtained by esterification with dry sodium acetate at 230° in sealed tubes for 4—8 hrs.

CHEMICAL ABSTRACTS.

Standards for Diesel fuel. N. I. TSCHERNOZHUKOV (Izvest. Teploekh. Inst., 1927, No. 9, 78—87).—The Russian standards are recorded.

CHEMICAL ABSTRACTS.

Preparing petrolatums. N. I. TSCHERNOZHUKOV (Nef. Choz., 1929, 16, 240—241).—The preparation of petrolatums of high or low viscosity is described. For medicinal petrolatums only the paraffins of high m.p. are removed.

CHEMICAL ABSTRACTS.

Technical and medicinal petrolatums from Grozni mixed-base fuel oil. B. K. TARASOV (Nef. Choz., 1929, 16, 236—239).—The fuel oil was treated with sulphuric acid, neutralised with sodium silicate, filtered through silica gel, and the resulting oil was distilled with steam. For the preparation of medicinal petrolatum the technical product was dissolved in gasoline, treated twice with fuming sulphuric acid, separated from sludge, neutralised, washed, dried, and treated with fuller's earth. Russian and American petrolatums, respectively, have d 0.866, 0.832, and contain paraffin wax (18.9%, 30%) of m.p. 56—69.5°, 58.5—62°.

CHEMICAL ABSTRACTS.

Oil analysis. KETTLE.—See XII.

See also A., Jan., 57, **Oxidation of petroleum hydrocarbons** (BERMEJO). **Structure of bog-head coal** (STADNIKOV and KASCHTANOV). 80, 2:3:6-Trimethylantracene (MORGAN and COULSON).

PATENTS.

[Coal-]distillation apparatus. G. V. MCINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS CO. (U.S.P. 1,733,747, 29.10.29. Appl., 19.10.22).—A compact

masonry body is formed with a series of vertical retorts of small horizontal cross-section, and a series of separate vertical channels parallel to and between the retorts. Ports connect the retorts with some of the channels so that they serve as flues for distillation gases, whilst others act as heating flues for the retorts.

W. G. CAREY.

[Coal-]distillation apparatus. E. PIROU (U.S.P. 1,733,750, 29.10.29. Appl., 3.12.26).—An endless conveyor in the distillation chamber is fitted with scrapers to remove the residue remaining thereon after discharge.

W. G. CAREY.

Regenerative coke ovens. W. MUELLER (B.P. 322,303, 3.11.28. Addn. to B.P. 308,994; B., 1929, 464).—Each of the twin heating flues or single heating flues (cf. the prior patent) is connected directly with one of a number of adjacently disposed gas and air regenerators, situated underneath each oven chamber and extending the whole length thereof.

A. B. MANNING.

Utilisation of coke ovens. N. V. SILICA EN OVENBOUW MIJ., and W. HIBY (B.P. 322,341, 3.12.28).—A non-caking coal is carbonised together with a caking coal in a coke oven or horizontal chamber oven by first charging the caking coal into the chamber in such a manner that a free space of triangular section is left at the front end of the oven, into which the non-caking coal is then charged. When the coking process is complete and the oven door is opened, and the coke from the non-caking coal is discharged automatically by gravity and can be removed prior to the expulsion by the ram of the normal cake of coke. The non-caking fuel may be introduced into the oven in the form of briquettes, if desired.

A. B. MANNING.

Gasification of earthy, moist, raw, brown coal, without ammonia recovery. ALLGEM. VERGASUNGS-GES.M.B.H. (G.P. 455,066, 1.6.23).—The air used for gasification is preheated to above 300° so that the fuel introduced into the upper part of the producer is pre-dried by the sensible heat of the gas current passing through it.

A. B. MANNING.

Removing distillation products from the lower part of coke ovens or retorts. K. BEUTHNER, M. and M. KLÖNNE (B.P. 322,351, 11.12.28).—The bottom of the oven is slightly extended in cross-section in such a manner that the fuel does not completely fill the extension, but, as a result of its natural angle of repose, leaves a space suitable for conducting away the distillation products. The off-take from this space is provided with cleaning means and is connected with an upper off-take pipe. Steam is introduced into the oven through the lower off-take towards the end of the coking period for the production of water-gas.

A. B. MANNING.

Improving the economy of operating a coke-oven plant by running a Bergius hydrogenation plant in co-operation therewith. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 630,328, 5.3.27. Ger., 19.11.26).—The coke-oven gas poor in hydrogen, produced in the first stages of distillation, is mixed with the rich gas from the hydrogenation process, and distributed as illuminating gas in long-distance transmission

systems. The gas rich in hydrogen evolved in the later stages of distillation is supplemented with hydrogen made by utilising the coke, and used in the hydrogenation plant. A poorer gas coal is used in the coke-oven plant than in the hydrogenation plant.

A. B. MANNING.

Production of liquid hydrocarbons by hydrogenation of coal. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 630,324, 5.3.27. Ger., 30.7.26).—The high-boiling products of the hydrogenation of coal at elevated temperatures and under high pressures are reintroduced into the pressure apparatus, the carbonaceous material being thereby further transformed into liquid and soluble products.

A. B. MANNING.

Production of briquetted fuel. IMPERIAL CHEM. INDUSTRIES, LTD., and R. E. SLADE (B.P. 322,194, 31.7.28).—A paste of coal and oil is hydrogenated under conditions, *e.g.*, at 410° and under 200 atm., which result in only a partial hydrogenation of the coal with the formation of a liquid product which solidifies on cooling to the ordinary temperature. This is used as a binding agent for briquetting fuels, particularly friable semi-coke.

A. B. MANNING.

Briquetting of fuels. CHEM.-TECHN. GES.M.B.H. (B.P. 308,579, 14.2.29. Ger., 23.3.28. Addn. to B.P. 300,195; B., 1929, 546).—The mixing of the blended fuel described in the prior patent is carried out rapidly in a disintegrator or similar device, in such a way that the heat interchange of the differently heated fuels occurs during and after the mixing, or mainly after the mixing. The pressure treatment is preferably carried out during the equalisation of the temperature of the mixture, at the time when its caking capability is at a maximum. Pitch or other binding agent may be added, if desired, during the mixing. Grooves or holes may be provided on or in the briquettes for the escape of the gases evolved during the pressure treatment.

A. B. MANNING.

Carbonaceous briquette and its manufacture. H. F. RIPPEY, G. DAVIDSON, C. N. CONE, I. F. LAUCKS, and H. P. BANKS (U.S.P. 1,735,506, 12.11.29. Appl., 3.5.26).—Coal, coke, or anthracite fines are bonded with a mixture of 5 pts. of soya-bean flour, 10 pts. of wheat flour, 5 pts. of molasses, 3 pts. of slaked lime, and 4 pts. of sodium carbonate, and briquettes formed therefrom under pressure are dried at 40–80°.

A. R. POWELL.

Preparation of absorptive carbon. A. B. RAY, ASSR. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,736,051, 19.11.29. Appl., 28.10.21).—Carbon for water filtration is prepared by mixing finely-divided activated carbon with 2–3 times its weight of commercial glucose, moulding the mixture under pressure into bricks, and heating these slowly up to 450–500°.

A. R. POWELL.

Improving the electrical and thermal conductivity of active charcoal. METALLBANK U. METALLURGISCHE GES. A.-G. (G.P. 454,855, 16.2.24).—Previously activated charcoal is heated above 600°, preferably at about 750°.

L. A. COLES.

[Carbon] pigment. W. O. SNELLING (U.S.P. 1,730,389, 8.10.29. Appl., 9.10.24).—In order to produce the deep black colour of carbon black obtained by the

incomplete combustion of hydrocarbons, carbonaceous material, *e.g.*, coal or charcoal, is ground wet in a colloid mill, and dyed with a complementary colour. *E.g.*, a brown-black charcoal is dyed with a blue-black dye.

F. G. CLARKE.

Gas-producing apparatus. Making mixed water-gas and coal gas. Gas-producing process. J. J. O'CONNOR (U.S.P. 1,733,621—3, 29.10.29. Appl., [A] 12.10.21, [B] 14.4.23, [C] 8.4.29. Renewed [B] 8.4.29).—(A) In an arrangement of duplicate generators and a gas receiver each pair of generators has a chamber for gas-yielding material and duplicate upper and lower conduits for the gas, one of each being common to the two chambers, and the other pair leading to the receiver; a system of separate valve control for the various conduits is described. (B) Separate charges of coal or other carbonaceous material are contained in duplicate gas generators and one charge is heated to incandescence, steam passed through, and the resultant water-gas passed while hot directly through the second charge and thence to a gas outlet. The direction of flow of the steam is reversed periodically without altering the order in which the two masses are attacked thereby. (C) The material as in (B) is treated so that one charge is subjected to partial combustion, and the gas obtained passed directly through the second charge without substantial change or decrease in its sensible heat and thence to a gas outlet. The flow of gas through the first charge is reversed frequently.

W. S. E. CLARKE.

Production of rich gas in a water-gas plant. K. WOLINSKI (G.P. 454,873, 13.12.25).—A current of hot gas is introduced into the distillation chamber of the producer. This gas is preheated by making use of the sensible heat of the gas produced, which has been raised to a high temperature in order to fix the low-temperature tar vapours it contains.

A. B. MANNING.

Removal of sulphuretted hydrogen from gases. KALI-IND. A.-G., C. T. THORSSELL, and A. KRISTENSSON (B.P. 297,114, 31.8.28. Ger., 16.9.27).—The gases are washed with a suspension of basic ferric salts in a ferric salt solution, obtained by treating a solution of the corresponding ferrous salt with a strong oxidising agent, *e.g.*, nitric acid. The reaction, which may be represented by the equation: $11\text{FeCl}_2 + \text{FeCl}_3 \cdot 6\text{Fe}(\text{OH})_3 + 9\text{H}_2\text{S} = 18\text{FeCl}_2 + 9\text{S} + 18\text{H}_2\text{O}$, proceeds quantitatively and the sulphur is readily recovered in a pure form.

A. B. MANNING.

Odorised [coal] gas production. J. H. JAMES, ASSR. to C. P. BYRNES (U.S.P. 1,732,516, 22.10.29. Appl., 16.7.26).—To prevent accidental poisoning by coal gas or carbon monoxide a small quantity of an odorant gas is added. This gas is obtained by the incomplete oxidation of aliphatic or naphthenic hydrocarbons in the presence of a catalyst, *e.g.*, molybdenum trioxide, at 450–500°, and consists of a mixture of aldehyde derivatives.

A. R. POWELL.

Preparation of a hard pitch, combining high binding power with high softening point. CONTINENTALE PRODORIT A.-G. (G.P. 453,712, 5.12.24).—Tars of low free carbon content, or similar materials, are distilled until the residue possesses a softening point

(Krämer-Sarnow) of 100–160° or higher. The distillation may be carried out under reduced pressure and, in the later stages, if necessary, also in a current of superheated steam. The product is used especially in the manufacture of concrete compositions.

A. B. MANNING.

Treatment of bitumen for production of new products. F. W. NICHOLLS (B.P. 323,060, 6.10.28).—Glue is melted in half its weight of gas oil, the solution (5%) is added to a mixture of bitumen (20%) and gas oil (75%) heated to about 150°, and the whole is emulsified in a colloid mill.

H. ROYAL-DAWSON.

Blending of [creosote] oils. T. COX and A. S. KNOWLES, ASSRS. to M. L. REQUA (U.S.P. 1,735,421, 12.11.29. Appl., 10.8.25).—To produce creosote oil of predetermined gravity, an intimate mixture of predetermined amounts of petroleum oil and finely-crushed coal is coked from below and upwards at a temperature sufficiently low to blend the mixture chemically and liberate a maximum quantity of vapours and a minimum of gas therefrom, which are condensed. F. R. ENNOS.

Manufacture of bituminous emulsions. L. KIRSCHBRAUN (U.S.P. 1,733,493—4, 29.10.29. Appl., [A] 7.5.24, [B] 25.5.25).—(A) A coating composition comprises an emulsion containing a pigment, a protective colloid to prevent inversion, and a bituminous material solid at the ordinary temperature. An emulsifying agent is used, which normally gives an emulsion that is unstable when brushed or rubbed, in the presence of a pigment. (B) A flocculating agent having an acid reaction is added to an aqueous vehicle containing a clay-like emulsifying agent, and the vehicle then used for emulsifying a bituminous material. F. G. CLARKE.

Manufacture of aqueous [bitumen-pitch] dispersions. L. KIRSCHBRAUN (U.S.P. 1,733,495—8, 29.10.29. Appl., [A] 25.3.27, [B] 30.7.27, [C] 8.8.27, [D] 2.1.28).—(A) After a bitumen-pitch material has been dispersed in a medium which has been modified by the addition of an acidic substance, more of the latter is added to reduce the p_H value to less than 4. (B) A bitumen-pitch material is dispersed at a predetermined p_H value, the viscosity of the dispersion is reduced, and then further quantities of the bitumen-pitch is dispersed therein, while maintaining a p_H value different from that of the first dispersion. (C) An aqueous dispersion of a bitumen-pitch material is subjected to a beating action to counteract the action of an electrolyte which is added in sufficient quantity, normally to produce flocculation. (D) An alkaline earth is used as the electrolyte and, in addition to beating the dispersion, a protective colloid is added.

F. G. CLARKE.

Apparatus for treating [cracking] oils. W. R. HOWARD, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,715,066, 28.5.29. Appl., 31.3.24. Renewed 2.6.27).—Hydrocarbon oil is raised to cracking temperature, e.g., 400°, in a heating coil and passed into a partitioned expansion chamber in such a way that reflux from the dephlegmator flows into the bottom chamber. Vapours from the dephlegmator pass to the usual condenser and receiver, whilst the residuums from the expansion chamber are drawn off separately, that from the inner and upper chamber being passed to a still connected

with a condenser and receiver. Residuum from this still may be stored or circulated to the heating coil as desired.

R. BRIGHTMAN.

Combined process of distilling, cracking, and gas-making, and apparatus therefor. A. SCHWARZ (U.S.P. 1,733,135, 29.10.29. Appl., 5.1.27).—The apparatus described comprises within a furnace a combustion chamber, a series of heating zones connected thereto, gas retort, oil still, steam superheater, boiler, and oil and water preheaters. In order to produce volatile hydrocarbon distillate and gas from crude petroleum oil, separate streams of preheated oil and steam are passed in countercurrent flow out of direct contact with flue gases, and the steam is superheated. The oil is vaporised at points where the flue gases are at sufficiently high temperatures, and the oil vapours are removed and condensed. The residual oil is treated with the superheated steam at a point in the flow of the flue gases where the temperature is highest and able to convert a substantial portion of this oil into gas. W. S. E. CLARKE.

Heat treatment of hydrocarbon oils. E. C. D'YARMETT, ASSR. to FRACTIONATOR Co. (U.S.P. 1,732,805, 22.10.29. Appl., 3.11.28).—Preheated oil, pumped into the bottom of a vertical cylindrical still through a tangential pipe, is thus caused to move upwards with a rotary motion. This motion is accelerated by means of a series of rotating discs with radial fins so that a thin film of oil is formed on the heating surface. In this way overheating during the cracking operation is avoided and the light oils produced contain the maximum content of gasoline and the minimum of unsaturated hydrocarbons. Addition of hydrogen or methane to the cracking vessels improves the yield still further.

A. R. POWELL.

Treatment of heavy hydrocarbon oils. C. A. JENSEN. From JENKINS PETROLEUM PROCESS Co. (B.P. 319,040, 13.6.28).—Hydrocarbon oils, in which a finely-divided absorptive material has been intimately mixed, are treated under pressure in a closed still, comprising an upper, longitudinal drum superimposed over a bank of tubes expanded into front and rear transverse drums, the latter being connected to the upper drum by suitable sluiceways in one of which is installed a positive circulatory device. This consists of a balanced propeller having a flywheel adapted to force the entire liquid contents of the still through its cyclic channel at a velocity sufficiently great to maintain the liquid in a uniform condition as to temperature and dispersion of solids. The cracking stock is continuously introduced into the still, cracked vapours being led to a dephlegmator and residual stock withdrawn. H. S. GARLICK.

Method of distillation. [Cracking of hydrocarbons.] C. P. TOLMAN, ASSR. to PETROLEUM CONVERSION CORP. (U.S.P. 1,733,143, 29.10.29. Appl., 22.11.24).—Saturated mercury vapour, at a temperature not much in excess of the desired conversion temperature, is passed into physical contact with hydrocarbon compounds in a reaction zone. The mercury vapour provides the heat required at the substantially uniform temperature of its condensation, the products are withdrawn from the reaction zone, and the hydrocarbons of low mol. wt. recovered.

W. S. E. CLARKE.

Converting high-boiling hydrocarbons into low-boiling hydrocarbons. H. TERRISSE and L. DUFOUT (B.P. 305,981, 21.1.29. Ger., 13.2.28).—High-boiling oils are cracked by heating at 200–500° and under 5–40 kg./cm.² in the presence of hydrogen, water-gas, or like gases. Ferrous chloride is added as a catalyst and to act as a desulphuriser. The reaction is accelerated by the addition of active carbon, brown coal, lignite, or peat.

W. S. E. CLARKE.

Cracking of petroleum products. A. SACHANEN [SACHANOV] and M. TILITSCHYEW [TILITSCHEEV] (B.P. 322,760, 11.9.28).—To overcome the disadvantage of coke formation which takes place in cracking plants fitted with reaction vessels, while the oil is held in a quiescent state at high temperature, the present process is so designed that the oil, after leaving the heating coil, is passed through a long series of reaction coils, of larger diameter, where it is held at cracking temperature while in a condition of turbulence. The duration of heating is more definite under these conditions than when the oil forms part of a big bulk of which it is only possible to fix an average reaction period. After leaving the reaction tube the oil is expanded into a vaporising chamber at atmospheric pressure, from which coke may be easily removed. Two such expansion chambers are used so that the process need not be interrupted for cleaning purposes.

T. A. SMITH.

Cracking or destructive hydrogenation of oils. F. HOFMANN and C. WULFF (B.P. 301,395, 27.11.28. Ger., 29.11.27).—The process is carried out in the presence of a halide of boron or of an addition product thereof, such as $\text{Et}_2\text{O} \cdot \text{BF}_3$. In the presence of such a catalyst only small amounts of gases (e.g., methane) or of carbon deposits are produced. Owing to the catalysts being gases or liquids, a good distribution of them throughout the material is obtained. A crude mineral oil, boiling above 300°, in the presence of the halide (5%) and hydrogen at 150 atm. and 380° gave 75% of oil boiling below 250° and 15% of a highly viscous lubricating oil. With the double compound (10%) at 400° and 1 atm. a yield of 60% of light oil was obtained.

T. A. SMITH.

Lubricating oils. H. M. WELLS and J. E. SOUTHCOMBE (B.P. 320,899, 19.7.28).—Synthetic hydrocarbons of low viscosity, manufactured by hydrogenation of carbonaceous material, are mixed with small quantities (1% or less) of organic acids containing more than five carbon atoms (e.g., oleic acid).

W. S. E. CLARKE.

Manufacture of viscous [lubricating and insulating] oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,935, 21.1.29. Addn. to B.P. 305,553; B., 1929, 274).—Liquid products, prepared by the process of the prior patent and having a viscosity greater than that desired, may be blended with lighter hydrocarbon oils to give lubricating oils etc. of any desired viscosity.

T. A. SMITH.

Reclaiming lubricating oil. W. C. RATH (U.S.P. 1,735,546, 12.11.29. Appl., 20.3.29).—Used lubricating oil from internal-combustion engines etc. is treated with a current of hot water-gas or producer gas, direct from the generator, to remove volatile oils and enrich the gas, and the heavier oils are then filtered while still hot,

the filtrate being suitable for use again as lubricating oil.

A. R. POWELL.

Purifying oils. M. LUTHER and K. PIEROH, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,371, 22.10.29. Appl., 29.6.27. Ger., 5.7.26).—Mineral and vegetable oils are purified by agitation with methyl formate, which extracts the coloured impurities and sulphur compounds.

A. R. POWELL.

Refining of petroleum oils. Treatment of hydrocarbons. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,733,619 and 1,733,620, 29.10.29. Appl., [A] 9.12.25, [B] 24.10.27).—(A) Lighter-gravity distillation products from the cracking process are treated with an aqueous solution of an acid sulphate. (B) An alkaline-earth metal soap is introduced into the residual oil from the cracking process, and the whole is subjected to a homogenising treatment.

W. S. E. CLARKE.

Refining of crude paraffin wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,921, 19.7.28).—Crude wax is treated in the liquid form with hydrogen at high temperature and pressures. The duration of treatment and the temperature are regulated so that the wax suffers no cracking. A catalyst consisting of oxides of at least one of the metals of groups III, IV, or VI may be added, and the addition of small quantities of iron, nickel, cobalt, or their oxides has an activating effect.

W. S. E. CLARKE.

Manufacture of white petrolatum. A. HOLMES, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,733,597, 29.10.29. Appl., 27.10.25).—The material yielding petrolatum is treated in the liquid form with fuming sulphuric acid at a high temperature, the residual liquid containing suspended carbonaceous products is treated with a solvent for the petrolatum, and the purified petrolatum is recovered from the solution.

W. S. E. CLARKE.

Preparation of mixtures of carbon monoxide and hydrogen from hydrocarbons. L. CASALE (U.S.P. 1,734,559, 5.11.29. Appl., 14.4.27. Italy, 20.4.26).—See B.P. 299,492; B., 1929, 17.

Safety device for gas burners. K. BECK (B.P. 300,966, 19.11.28. Ger., 21.11.27).

Fuel burners for steam generators, metallurgical furnaces, etc. A. E. YOUNG and G. P. DENNIS (B.P. 322,808, 14.9.28).

Thermal treatment of pulverulent material (B.P. 305,231). **Separation of dry materials** (B.P. 322,480). **Fluid segregator** (B.P. 322,654). **Distillation of liquids** (B.P. 297,122). **Introducing materials into high-pressure vessels** (B.P. 322,645).—See I. **Cleaner for fabrics** (U.S.P. 1,733,842).—See VI. **Hydrogen** (U.S.P. 1,736,065). **Nitrogen and hydrogen** (B.P. 298,190).—See VII. **Organic acids** (B.P. 321,399). **Unsaturated fatty acids** (B.P. 322,437). **Products resembling wax** (B.P. 320,854).—See XII. **Plastic masses** (B.P. 322,496 and G.P. 455,551).—See XIII.

III.—ORGANIC INTERMEDIATES.

Determination of impurities in naphthalene. M. COQUETTE (Bull. Soc. chim. Belg., 1929, 38, 387–391).—The setting points of mixtures of pure naphthalene and

of various quantities of oil, b.p. 175—380°, d 1.05, isolated in the industrial purification of naphthalene, were determined and a curve was constructed. The oil content of naphthalene could thus be determined from the setting point by reference to the curve.

A. I. VOGEL.

[Acetic acid etc. from] nut shells. GEORGI and BUCKLEY. Amyl acetate from gasoline. DOBRYANSKI and GUREVICH.—See II. Aldehyde-oxidation reactions for phenols. FULTON.—See XX. By-products of saccharin manufacture. HERZOG.—See XXI.

See also A., Jan., 80, Synthesis of anthracene homologues (MORGAN and COULSON). 82, Anomalous diazo-coupling with unsaturated phenols (QUILICO and FREMI). 87, Electrolytic reduction of aromatic acids (SOMLO). 96, Polycondensed heteronuclear systems (CHARRIER).

PATENTS.

Production of carbon disulphide. I. G. FARBEN-IND. A.-G. (B.P. 300,579, 1.11.28. Ger., 15.11.27).—Carbon uniformly heated in a retort is treated with sulphur vapour superheated in a separate retort to a temperature regulated according to the quantity of carbon disulphide required. The retorts, which may be arranged, *e.g.*, in blocks of three comprising two carbon retorts with an intermediate sulphur superheater, are so fired that their lower ends are heated the most strongly. (Cf. B.P. 237,716 and 282,049; B., 1925, 759, 1928, 262.)

L. A. COLES.

Production of concentrated acetic acid. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 308,210, 7.3.29. Ger., 19.3.28).—Aqueous acetic acid is run slowly into an acetic acid solvent, preferably a chlorinated hydrocarbon, both liquids being cooled so that the temperature of the mixture does not exceed about -25° . Ice crystals which separate are removed by centrifugal means, and glacial acetic acid is recovered from the residual liquid by fractional distillation. The ice crystals and the cold residual liquid are used for precooling fresh supplies of the liquids, or in the condenser of a refrigerator.

L. A. COLES.

Manufacture of organic bases [from acetylene and ammonia or amines]. I. G. FARBENIND. A.-G. (B.P. 302,939, 22.12.28. Ger., 23.12.27. Addn. to B.P. 283,163 and 296,423; B., 1929, 275, 747).—In the process of the prior patents the product boiling at 120—300° is used to scrub the vapours leaving the catalyst chamber, thereby removing liquid products, acetylene, and ammonia; the latter are recovered on boiling the solution, and are returned to the reaction chamber.

C. HOLLINS.

Manufacture of keten. H. J. CLARKE and C. E. WARING, ASSRS. to EASTMAN KODAK Co. (U.S.P. 1,723,724, 6.8.29. Appl., 13.4.28).—Acetone etc. is passed through a copper tube heated to 650—670° and packed with copper or other turnings of a metal, m.p. above 700°, coefficient of heat conductivity > 0.150 c.g.s. unit, the period of contact being less than 5 sec. and the tube and packing free from iron and nickel.

C. HOLLINS.

Increasing the wetting property of treatment liquids. H. T. BÖHME A.-G. (B.P. 307,397, 21.2.29.

Ger., 6.3.28).—Esters of adipic acid or of the cheaper substituted adipic acids are used as wetting-out agents; they dissolve in water in presence of solubilising agents (soap, sulphonated oils, alkylated naphthalenesulphonic acids).

C. HOLLINS.

Separation of organic liquids from mixtures containing same. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 305,528, 19.11.28. Belg., 6.2.28).—For the separation of a liquid *A* from a lower-boiling liquid *B* in aqueous mixtures of the two, water is first removed by the azeotropic method, using as entrainer either *A* or a more volatile liquid *E*, and the anhydrous mixture of *A* and *B* is then resolved by distillation in the ordinary manner. A continuous process for separating *iso*amyl and *isobutyl* alcohols from wet fusel oil, ethylene chlorohydrin from *cyclohexanol* and water, or acetic acid from *cyclohexyl* acetate and water is described. (Cf. B.P. 304,756; B., 1930, 126.)

C. HOLLINS.

Substituted dithiocarbamates and their manufacture. S. M. CADWELL, ASSR. to NAUGATUCK CHEM. Co. (U.S.P. 1,726,646—8, 3.9.29. Appl., 2.3.28).—(A) Polynitroaryl-, (B) benzylidenebis-, and (c) 4-chloro-2:6-dinitrophenyl-dialkyldithiocarbamates are prepared by the usual methods.

C. HOLLINS.

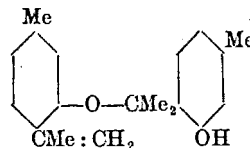
Manufacture of halogenated anilines. W. J. HALE and G. H. CHENEY, ASSRS. to DOW CHEM. Co. (U.S.P. 1,729,775, 1.10.29. Appl., 23.8.26. Renewed 26.7.28).—Polyhalogenated benzenes, or their mixtures, are heated with anhydrous ammonia in presence of a copper compound. *E.g.*, dichlorobenzene in alcohol is heated with 3 mols. of ammonia at 150—250° in presence of metallic copper and 0.1 mol. of cuprous chloride.

R. BRIGHTMAN.

Carrying on catalytic reactions. [Manufacture of phthalic anhydride from naphthalene.] F. A. CANON and C. E. ANDREWS, ASSR. to SELDEN RES. & ENG. CORP. (U.S.P. 1,735,951, 19.11.29. Appl., 12.6.22).—A mixture of air and naphthalene vapour is conducted over a vanadium pentoxide catalyst at 425°, and the heat of the reaction is controlled by cooling the reaction vessel with an alloy (b.p. 425°) of 30% Pb, 30% Sn, and 40% Hg. The mercury vapour passes into a reflux air condenser from which the condensed metal passes back to the container holding the alloy, the composition and b.p. of which are thus kept constant.

A. R. POWELL.

Alkylcoumarans and their manufacture. H. JORDAN, ASSR. to CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (U.S.P. 1,679,664, 7.8.28. Appl., 20.6.27. Ger., 29.6.26).—The condensation product from *m*-cresol and acetone (annexed formula) gives on distillation at 300° 3:6-dimethylcoumaran, b.p. 98°/11 mm., together with 4-isopropenyl-*m*-cresol. The product from *p*-cresol and acetone similarly yields 3:5-dimethylcoumaran, b.p. 102°/11 mm., and 3-*isopropenyl-p*-cresol. The dimethylcoumarans are solvents for lacquers, and the *isopropenyl*cresols may be hydrogenated to thymols and menthols.



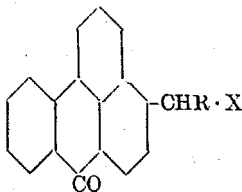
Manufacture of alkylisopropylphenols and their hydrogenation products [thymol, menthol]. CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (B.P. 273,685, 15.6.27. Ger., 29.6.26).—The isopropenyl-*m*- and -*p*-cresols of B.P. 273,686 (B., 1928, 236) are di- or octahydrogenated at 140–160° or 170–180° in presence of nickel to give thymol, 3-isopropyl-*p*-cresol, menthol, or 4-methyl-6-isopropylcyclohexanol. C. HOLLINS.

Manufacture of hydrogenation products. SCHERING-KAHLBAUM A.-G. (B.P. 293,001, 23.6.28. Ger., 29.6.27. Addn. to B.P. 276,010 and 280,956; B., 1929, 237).—The process of the prior patents is applied to coumarans (3:6-dimethylcoumaran gives thymol or menthol or their isomerides) and to the condensation product from α -naphthol and acetone (giving 4-isopropyl- α -naphthol etc.). C. HOLLINS.

Manufacture of hydrogenated aromatic carboxylic esters and acids. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 286,201, 7.1.29).—Aromatic esters (other than acylaminonaphthoic esters) are hydrogenated at 30–40 atm. and 120–150° in presence of a catalyst, comprising nickel or a mixture of nickel and a metal of the iron group, prepared by the method of B.P. 255,884 (B., 1927, 528). Examples are: ethyl hexahydrobenzoate; ethyl cyclohexylacetate; methyl hexahydrovalerate, b.p. 96–103°/8 mm.; ethyl hexahydrophthalate; methyl hexahydroterephthalate, b.p. 133–139°/13 mm.; ethyl 5:6:7:8-tetrahydro-1-naphthoate, b.p. 156–159°/12 mm.; ethyl 5:6:7:8-tetrahydro-3-hydroxy-2-naphthoate, b.p. 170°/17 mm.; and ethyl 5:6:7:8-tetrahydro-3-amino-2-naphthoate, m.p. 55–58° (free acid, m.p. 178°). C. HOLLINS.

Manufacture of nitrated aromatic amino-compounds. BRIT. CELANESE, LTD., D. H. MOSBY, H. C. OLPIN, and G. H. ELLIS (B.P. 319,296, 18.6.28).—Arylsulphonyl derivatives of arylamines are nitrated with nitric acid of 60–70% concentration, and are subsequently hydrolysed by means of concentrated sulphuric acid. *p*-Toluenesulphon-*o*-anisidide gives at 20–40° 5-nitro-*o*-anisidine, and at 40–50° 3:5-dinitro-*o*-anisidine, m.p. 182°. C. HOLLINS.

Manufacture of condensation products of the benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,593, 24.3. and 7.8.28).—Benzanthrones having a free 4-position are condensed at 10–30° in presence of caustic alkali and an organic solvent with compounds of the type $R \cdot CH_2 \cdot X$, where X is CN, CO·NH₂, or COR', and R is hydrogen, or an alkyl, aryl, or carboxylic ester group. The products have the annexed formula. Those from benzanthrone with acetone (m.p. 189–190°), with acetophenone (m.p. 205–206°), with acetonitrile (m.p. 192–194°), with propionitrile (m.p. 150–152°), and with phenylacetonitrile (m.p. 208–210°), and from 3-chloro-benzanthrone with acetone are described.



C. HOLLINS.

[Alkylation and aralkylation of] amino-derivatives [especially of vat dyes and their interme-

diates]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 319,805, 30.4.28).—Aminoanthraquinones etc. are converted into sulphamic acids, *e.g.*, by means of pyridine-sulphuric anhydride in pyridine, and these are alkylated or aralkylated, *e.g.*, by means of methyl sulphate and alkali. Examples are the preparation of 2-methylaminoanthraquinone, 3-chloro-2-methylaminoanthraquinone (m.p. 234–235°), 1-amino-4-methylaminoanthraquinone, methylaniline, and methylaminodibenzanthrone. C. HOLLINS.

Condensation product [of a phenol], containing sulphur. P. VIRCK, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,736,063, 19.11.29. Appl., 7.11.25. Ger., 14.11.24).—See B.P. 242,974; B., 1926, 596.

Hydrogenation of naphthalene. (A) W. SCHRAUTH, and (B) G. SCHROETER and W. SCHRAUTH, ASSRS. to NEWPORT MANUF. Co. (U.S.P. 1,733,908–9, 29.10.29. Appl., 29.8.21. Ger., [A] 13.5.16, and [B] 1.8.16).—See G.P. 299,012 and 324,863; B., 1920, 743 A; 1921, 253 A.

Production of homonuclear aminoalkylaminoanthraquinonesulphonic acids. K. WEINAND, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,735,147, 12.11.29. Appl., 18.8.26. Ger., 21.8.25).—See B.P. 280,637; B., 1928, 83.

Manufacture of 1-phenylbenzanthrone compounds. W. TRAUTNER, B. STEIN, and R. BERLINER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,736,061, 19.11.29. Appl., 16.9.26. Ger., 23.11.25).—See B.P. 297,129; B., 1928, 847.

Separation of liquids (B.P. 304,756).—See I. Fatty acids (B.P. 322,437 and G.P. 456,353).—See XII. Vulcanisation accelerators (B.P. 303,827).—See XIV.

IV.—DYESTUFFS.

Zeiss scale-photometer. KÜNTZEL.—See XV.

See also A., Jan., 53, Determination of minute amounts of copper (CALLAN and HENDERSON). 79, *s*-Triphenylbenzene dye (VÖRLANDER and others). 81, Cleavage of azo-dyes by sulphite (ENGEL). Chrome dyes: chromotrope 2R (ROSENHAUER and others). 96, Cyanine dyes (HAMER).

PATENTS.

Manufacture of sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 319,860, 18.7.28).—The addition of 4–7 pts. of cyclohexanol or other hexahydrophenol to the sulphide melt results in a purer dye, especially in the case of sulphurised indophenols or leucoindophenols. Examples are the sulphurisation of carbazole-leucoindophenol, of 4-anilino-4'-hydroxydiphenylamine, and of 4-amino-4'-hydroxy-3-methyldiphenylamine, in presence of hexahydrocresol. C. HOLLINS.

Indanthrene compound and its manufacture. M. S. THOMPSON, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,731,800, 15.10.29. Appl., 24.7.24).—2-Aminoanthraquinone is added to fused sodium and potassium hydroxides at about 220° in presence of potassium chlorate or other oxidising agent and an alkali phenoxide, *e.g.*, sodium phenoxide or cresoxide. The *N*-dihydro-1:1':2:2'-anthraquinoneazine (Ponsol

Blue R) obtained in increased (15–20%) yield is more resistant to bleaching than Indanthrene Blue RS.

R. BRIGHTMAN.

Manufacture of [azo] dyes [for cellulose ester materials]. BRIT. CELANESE, LTD., D. H. MOSBY, H. C. ORPIN, and G. H. ELLIS (B.P. 319,308, 18.6.28).—3:5-Dinitro-derivatives of *o*-toluidines, *o*-anisidines, or *o*-phenetidines are diazotised and coupled with non-sulphonated coupling components, particularly α -naphthylamines and their hydroxyalkyl derivatives. The dyeings on acetate silk are readily discharged. Examples are: 3:5-dinitro-*o*-anisidine \rightarrow *N*- γ -chloro- β -hydroxypropyl- α -naphthylamine (bright blue) or α -naphthylamine (violet); 3:5-dinitro-*o*-toluidine \rightarrow 4-nitro-*m*-phenylenediamine (yellow). (Cf. B.P. 319,390; B., 1930, 139.)

C. HOLLINS.

Hair dye. M. A. MINGUS (B.P. 319,946, 11.10.28).—A suitable synthetic or vegetable dye is mixed with a large excess of tartaric acid.

C. HOLLINS.

Manufacture of isodibenzanthrones. A. LÜTTINGHAUS and H. WOLFF, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,725,590, 20.8.29. Appl., 16.10.25. Ger., 28.2.25).—See G.P. 436,533; B., 1927, 386.

Amino-derivatives of dyes (B.P. 319,805).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Composition of sisal hemp from different countries. ANON. (Bull. Imp. Inst., 1929, 27, 441–449).—The chemical composition and behaviour of samples of sisal hemp from the following countries are described: Tanganyika (9 samples, av. cellulose content 78.9%; underwent smallest losses on hydrolysis, water washing, and acid purification), Kenya (4 samples, av. cellulose content 77.3%; suffered greater losses on water washing and acid purification), Portuguese East Africa (4 samples, av. cellulose content 76.3%, slightly inferior to Kenya samples), Mexico (10 samples, av. cellulose content 73.1; mostly inferior to the East African).

E. H. SHARPLES.

***Eucalyptus saligna* as a source of wood pulp for paper and artificial silk.** ANON. (Bull. Imp. Inst., 1929, 27, 449–452).—The soft, rather coarse-grained wood (moisture 9.3%, ash 0.5%, cellulose 56.9%) from a six-year-old tree of *Eucalyptus saligna* grown in Zululand gave a 55% yield of dry, unbleached pulp under comparatively mild treatment with caustic soda and produced a fairly soft, opaque, pale brown paper of good strength, but it required a concentrated bleaching solution to bleach it to even a moderately pale colour. To determine its suitability for artificial silk manufacture the dry bleached pulp (42% of the original wood) was examined. It had moisture 8.2%; α -, β -, and γ -cellulose 85.0%, 10.2%, 4.8%, respectively (on moisture-free material); ash (on dry pulp) 0.33%; copper number 2.04%; phloroglucinol absorption value 1.6; soda absorption value 209; acetone extract 0.46%. Probably, by submitting the wood to a more drastic treatment, the content of α -cellulose could be increased and the β -cellulose content and phloroglucinol absorption value reduced.

E. H. SHARPLES.

Identification and evaluation of unbleached chemical wood pulps. G. A. HALL (World's Paper

Trade Rev., 1929, 91, 1340–1354, 1396, 1442, 1444, 1484–1492, 1530–1536, 1588, 1630–1638, 1676–1688, 1738–1750, 1818–1834, 1906–1916).—A detailed discussion of the relation between the treatment and properties of unbleached chemical wood pulps, particularly unbleached sulphite and sulphate pulps.

CHEMICAL ABSTRACTS.

Acetate silk. D. KRÜGER (Papier-Fabr., 1930, 28, 1–8).—A review.

Preparation of [rosin] size [for paper] by a cold process. W. DOLIWO-DOBROWOLSKI and I. KAHAN (Zellstoff u. Papier, 1929, 9, 836–838).—The process described, in which a solution of sodium hydroxide is circulated over pieces of rosin, is similar to the Delthirna process. The most suitable concentration of sodium hydroxide is 0.5%; higher concentrations give too alkaline a size. Most rapid dissolution of the rosin occurs at 22–24°, so that the temperature of 15° recommended by Delcroix (B., 1928, 851) is not the most satisfactory one. The highest circulation velocity of the liquor consistent with good dissolution of the rosin is used (up to 6 cm./min.), and pieces of rosin 5–6 cm. in size are better than smaller ones, which cause blocking of the liquor channels. Experiments show that in sizing paper a 35% saving in rosin, compared with that when white size is used, may be obtained, but there is an increase in the proportion of alumina to rosin necessary. Comparison is made between sizing results obtained by the new and old processes from the point of view of cost and the properties of sized writing and printing papers. The following advantages of the new process are claimed in addition to saving of materials: reduction of steam consumption by 80%, economy of personnel, limitation of running replacements, decreased cleaning of plant, and avoidance of the disadvantages of the old soda-boiling process.

B. P. RIDGE.

Constant-humidity room. PRIOR.—See I. **Calcium sulphate content of tower liquors.** SCHMIDT and HÖNN.—See VII.

See also A., Jan., 10, **Spectrography of cellulose derivatives** (RASSOW and AEHNELT). 21, **Fibre X-ray diagrams of copper-alkali-cellulose** (TROGUS and HESS). 28, **Sorption of water vapour by cellulose and its derivatives** (SHEPPARD and NEWSOME). 46, **Action of light on cellulose nitrate** (DEVORE and others). 72, **Cellulose ethers** (NIETHAMMER and KÖNIG).

PATENTS.

Production of fibre articles. M. M. FROST (U.S.P. 1,735,426–9, 12.11.29. Appl., 20.8.27. Renewed [A, C, D] 1.5.29).—A pulp formed of the fibre in finely-divided condition with water is mixed with an adhesive such as rosin soap (A–C), or with casein and borax (D), and, after expressing the water in a suitable die under high pressure, the moulded article is dried and impregnated with (A) a solution of pine oil and banana oil; (B) 6 pts. of sulphur, 3 pts. of rosin, and 1 pt. of paraffin; (C) shellac and borax; (D) formaldehyde.

F. R. ENNOS.

[Fibrous] material for making gears and similar objects. W. H. ADAMS (B.P. 322,345, 7.12.28).—

A blank from which gears etc. may be cut is made by winding fibrous, hard, twisted cord in layers, the strands being saturated and bound together with a synthetic resin which is applied in the fluid state and subsequently polymerised.

F. R. ENNOS.

Treatment of silk-containing wool materials.

D. C. CHISHOLM and J. A. HANNUM, ASSRS. to G. E. COLLINGS (U.S.P. 1,735,420, 12.11.29. Appl., 12.5.26).—In order to destroy the silk in a wool-silk mixture, the latter is sprayed with 30% hydrochloric acid at ordinary temperature, wrung out, neutralised by spraying with 5% sodium hydroxide, again wrung out, washed with water, and dried.

F. R. ENNOS.

Treatment of waste spun or woven textile material.

A. L. BURLIN (B.P. 322,608, 2.11.28 and 21.2.29).—Textile waste containing natural or artificial fibres is cleansed by means of aqueous detergents and, before or after treatment in an opening carder, is subjected to the action of a weak acid (boric acid) and dextrin or soluble starch solution, with sodium sulphate solution, and also, if much artificial silk is present, with a solution of acid ammonium phosphate.

F. R. ENNOS.

Treatment [lubrication] of fibrous material of vegetable, artificial, or animal origin. J. G. KASTNER (U.S.P. 1,730,430, 8.10.29. Appl., 20.7.28. Ger., 27.8.26).—A highly viscous aqueous extract of carob-bean kernels is added to emulsions, oils, and fats used for wetting and softening fibres, *e.g.*, cotton and jute, before spinning. The kernels are finely ground and the embryos removed before they are extracted.

F. G. CLARKE.

Pulping of raw cellulosic material. Production of sulphite pulp. Production of cellulose pulp. G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,735,013—5, 12.11.29. Appl., [A] 1.6.28, [B] 20.7.28, [C] 23.8.28).—(A) Wood is digested under pressure at temperatures above 100° with a solution of the usual fibre-liberating chemicals in 90% glycerin containing 3% of sodium carbonate. The waste liquor is steam-distilled to recover glycerin, the aqueous solution evaporated to dryness, and the residue burned to remove organic matter. (B) Wood is digested under pressure with a solution of sodium and barium or strontium hydrogen sulphites. After releasing the pressure and pumping off the liberated sulphur dioxide the fibres are screened off from the finely-divided barium sulphite precipitate. (C) The digestion is carried out in a tall, cylindrical vessel with a screen at the bottom, which is provided with a pump for continuously removing screened liquor and circulating it through an indirect heater which is connected with a second pump for returning the liquor to the top of the digester.

A. R. POWELL.

Manufacture of cellulose [from sugar-cane fibre].

J. J. DE LA ROZA (B.P. 322,763, 30.6.28).—Cane fibre is treated with dilute sulphurous acid solution to prevent growth of fungus etc., and in this condition may be stored. After thorough washing with water the material is heated with dilute (15–20%) sodium or potassium hydroxide solutions, with or without pressure, and subsequently bleached by known methods.

A. G. POLLARD.

Cyclic process of [cellulose] fibre liberation. G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,730,383, 8.10.29. Appl., 19.5.28).—Cellulosic material is digested with a sulphurous acid solution of borax, at the usual temperature and pressure, and the liberated fibre separated from the spent liquor. The latter is neutralised, evaporated, and calcined to recover the alkali salts for re-use.

F. G. CLARKE.

Production of artificial silk by the cuprammonium stretch-spinning process. J. P. BEMBERG A.-G. (B.P. 299,038 and Addn. B.P. 322,425, 11.9.28. Ger., [A] 20.10.27).—The still plastic threads, after leaving the spinning funnel, where they have been preliminarily coagulated and stretched, are (A) subjected to a second considerable stretching by suitable means and are finally hardened by treatment with acid, or (B) treated with acid and, while still moist with acid, are subjected to a second considerable stretching.

F. R. ENNOS.

Production of artificial filaments [by the dry or evaporative method]. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 322,557, 19.9.28).—After leaving the spinning cell, the filaments are propelled at a speed greater than that of their extrusion by means of a gaseous or other fluid, *e.g.*, compressed air, which passes through the annular space between two conical tubes while the filaments pass through the inner tube where they are stretched and reduced in denier.

F. R. ENNOS.

Working-up of cellulose derivatives. A. EICHENGRÜN (B.P. 291,386, 31.5.28. Ger., 31.5.27).—The cellulose ester or ether is treated in the cold with a non-solvent, and after removal of the excess of liquid the swollen material is converted into a homogeneous plastic mass by heating and/or addition of small amounts of a solvent or a non-solvent, together with a plasticiser, if desired. The product is then worked up into the desired form in any suitable known manner.

F. R. ENNOS.

Manufacture of products having a basis of cellulose derivatives. C. DREYFUS and G. SCHNEIDER, ASSRS. to CELANESE CORP. OF AMERICA (U.S.P. 1,732,330, 22.10.29. Appl., 21.11.25. U.K., 24.12.24).—Powdered cellulose acetate is made into a paste with water and the paste is thoroughly incorporated with a mixture of benzene and triacetin. The product is dried and moulded.

A. R. POWELL.

Manufacture of artificial cork masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,752, 10.9.28).—Cork meal is impregnated with less than 30% of its weight of an unhardened resinous condensation product of urea (or its derivatives) and formaldehyde, with a softening, plasticising, or binding agent (*e.g.*, glycerin) and is subsequently pressed.

A. G. POLLARD.

Adhesive for binding waxed paper. F. SICHEL KOMM.-GES. (G.P. 455,014, 9.10.25).—The product obtained by heating starch with water and subsequently treating the paste with oxidising agents, *e.g.*, chlorine, ozone, is mixed with toluene, xylene, etc., capable of forming stable emulsions without the addition of protective colloids.

L. A. COLES.

Utilisation of stem fibres. C. E. BAHRÉ (U.S.P.

1,739,491, 17.12.29. Appl., 9.12.27).—See B.P. 302,052; B., 1929, 202.

Treatment of fibrous vegetable materials [flax]. O. D. LUCAS, Assr. to VICKERS, LTD. (U.S.P. 1,739,683, 17.12.29. Appl., 11.12.28. U.K., 8.12.27).—See B.P. 309,211; B., 1929, 593.

Manufacture of sheet-like articles and the like from cellulose. Cellulose precipitating agent. E. CZAPEK and R. WEINGAND (U.S.P. 1,740,226—7, 17.12.29. Appl., 31.8.26. Ger., 1.9.25).—See B.P. 257,924—5; B., 1927, 745,699.

Felt and felt-like materials [containing silk residue etc.] and hats etc. or materials made therefrom. M. J. L. SOBRINHO (B.P. 303,017, 22.12.28. Brazil, 23.12.27).

Pumping of artificial silk-spinning solutions. BRIT. CELANESE, LTD., J. BOWER, and W. YORKE (B.P. 322,778, 12.7.28).

Treatment of cellulose derivatives (B.P. 291,765).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Basic cotton dyeing. R. HALLER and K. ECKARDT (Kolloidchem. Beih., 1929, 30, 1—82).—The theoretical and practical aspects of basic cotton dyeing and mordanting are discussed. The tannin-potassium antimonyl tartrate reaction product used as a mordant consists of antimonyl digallate and has the composition $(C_{14}H_5O_9)_2SbOH$; it forms a colloidal solution which is readily taken up by cotton fibre. The reaction product of tannin and methylene-blue consists of methylene-blue tetragallate and its colloidal properties have been investigated. An examination of the three lakes formed by methylene-blue with tannin and potassium antimonyl tartrate shows that in each case the components are present in the same stoichiometric proportion, but are stereoisomerides, and this difference gives rise to differences in the degree of dispersion. The D-lake is less highly dispersed than the F-lake; the former corresponds to true printing and the latter to dyeing; a difference in structure is recognisable by microscopical examination of the fibre. Some colloid-chemical properties of katanol have been investigated.

E. S. HEDGES.

Mothproofing. M. G. MINAEFF and J. H. WRIGHT (Ind. Eng. Chem., 1929, 21, 1187—1195).—The mothproofing powers of different substances have been examined by exposing treated and untreated samples of woollen material to attack by mature larvæ under controlled conditions. Many nitrogenous substances are found to be valueless. Urea derivatives of good mothproofing power contain sulphur and have at least one amino-group intact, whereas those of no value either contain no sulphur or are thio-derivatives having substitution radicals in both amino-groups. The alkalis possess some proofing power, but it is considered insufficient; so also has oleic acid, but it is destroyed by the presence of alkaloids such as quinidine, caffeine, etc. Compounds containing fluorine differ widely in the amount of protection they afford. Silicofluorides are the most effective, and sodium silicofluoride is better

than sodium fluoride even when the latter is present in twice the concentration. The addition of certain salts (chlorides or sulphates) in limited proportion enhances the protective power of substances containing fluorine; mild acidity is also of assistance, but in higher concentrations acids have a retarding action. Sodium silicofluoride gives the best results when aluminium ions are also present, and materials treated with this substance retain their resistance to larval attack after rinsing in water to a much greater extent than do fabrics treated with sodium fluoride. Analysis of silicofluoride and fluoride solutions before and after the immersion of woollen fabrics shows that the former readily become exhausted, whereas the latter change but little in concentration. Undyed wool absorbs silicofluoride more readily than does dyed material. The acceleration of the absorption by aluminium ion is a function of the concentration of the latter, whilst, in equivalent concentrations, chlorides give greater acceleration than do sulphates.

B. P. RIDGE.

PATENTS.

Treatment of artificial fibres with liquids. HERMINGHAUS & Co. G.M.B.H., Assees. of H. RATHERT (B.P. 297,000, 10.9.28. Ger., 10.9.27).—Artificial viscose or cuprammonium fibres are sprayed with appropriate liquids (for desulphurising, de-coppering, bleaching, acidifying, oiling, or dyeing) while wound in a twisted condition on perforated or non-perforated bobbins made of or coated with porous material.

F. R. ENNOS.

Treatment of materials made of or containing cellulose esters. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 319,420, 26.6.28).—Acetate silk etc. is weighted and partially hydrolysed (preferably in that order) and then acquires, in addition to its affinity for basic and insoluble dyes, an affinity for direct, mordant, sulphide, and vat dyes. In particular, the treated material is suitable for production of discharge effects. The weighting may be performed according to prior patents B.P. 258,874, 259,899, 260,290, 277,602, 280,094, 281,084, 284,798, 285,941, and 309,876 (B., 1928, 288; 1927, 295; 1928, 477, 154, 296, 227, 444, 330; 1929, 596); for hydrolysis dilute sodium hydroxide and soap are used.

C. HOLLINS.

Coloration of materials comprising cellulose [ester or ether] derivatives. BRIT. CELANESE, LTD., D. H. MOSBY, H. C. OLPIN, and G. H. ELLIS (B.P. 319,390, 18.6.28. Cf. B.P. 319,308; B., 1930, 137).—The dyes of the prior patent are applied to acetate silk etc. or may be produced on the fibre, e.g., by padding with α -naphthylamine and developing with the diazotised amine.

C. HOLLINS.

Production of fast prints [with azo chrome dyes] on vegetable fibres. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,457, 7.8.28).—For printing cotton with prechromed azo dyes an organic acid (acetic) and a metallic mordant (chromium acetate) are added to the printing paste.

C. HOLLINS.

Cleanser [for fabrics]. F. S. VIVAS, Assr. to INTERNAT. FIREPROOF PRODUCTS CORP. (U.S.P. 1,733,842, 29.10.29. Appl., 30.6.26).—Gasoline is chlorinated in the presence of 7.5% of ammonium carbonate and 2%

of aluminium (or molybdenum) chloride until 35% of its weight of chlorine has been used; the product is then washed with soda and distilled; the chlorinated compounds obtained may be used for the removal of grease etc. from fabrics.

A. R. POWELL.

Dyeing mixed textile goods made of wool and silk fibres. H. WAGNER, H. EICHWEDE, and E. FISCHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,739,879, 17.12.29. Appl., 17.3.27. Ger., 22.3.26).—See B.P. 267,985; B., 1928, 669.

Production of aniline-black on textile fibres. L. A. LANTZ and R. WATSON (U.S.P. 1,739,908, 17.12.29. Appl., 18.7.27. U.K., 20.7.26).—See B.P. 279,164; B., 1928, 11.

Production of pattern effects upon textile goods. G. HEBERLEIN, Assr. to HEBERLEIN PATENT CORP. (U.S.P. 1,739,965—6, 17.12.29. Appl., 11.11.26. Ger., [A] 27.3.26, [B] 30.3.26).—See B.P. 268,389; B., 1928, 332.

Proofing cellulosic, animal, and other substances against insects, animals, and organisms. S. W. KENDALL (U.S.P. 1,739,840, 17.12.29. Appl., 10.8.25. U.K., 14.8.24).—See B.P. 247,242; B., 1926, 314.

Means [coloured paper sheets] for tinting of liquids [for laundry purposes]. L. CRUMP and E. R. MITFORD (B.P. 322,295, 25.10.28 and 16.5.29).

Wetting-out agents (B.P. 307,397).—See III. **Cleansing preparation** (B.P. 322,868).—See VII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Adsorptive properties of silica gel [and charcoal]. A. P. OKATOV (J. Appl. Chem. Russia, 1929, 2, 21—84).—The adsorptive properties are independent of the coagulating temperature and the concentration of silica in the sol. Above 25° it is difficult to obtain sols containing more than 12.5% SiO₂. Rapid coagulation hinders the development of internal structure. Monobasic acids increase the rate of coagulation. Syneresis is a good measure of the maturity of the gel. Excess of ammonia tends to produce gels with a slow rate of adsorption, but gels produced by incomplete neutralisation with ammonia are unsatisfactory. The internal structure is independent of the water content up to 80%. Treatment with water increases the rate of adsorption, but not the adsorptive capacity. The adsorptive properties of gels not treated with activating solutions are almost independent of the temperature changes during drying and activation. The adsorption of benzene, ether, and ethyl alcohol, but not that of water vapour, accords with Freundlich's law. More heat is evolved in the adsorption of vapours of organic substances by charcoal than by silica gel. Of chlorine adsorbed by charcoal 11% is retained even after heating at 260—280° in a current of air; charcoal poisoned with chlorine loses some of its adsorptive capacity. The hydrogen and oxygen present in charcoal are active substances which are indispensable constituents of its colloidal particles.

CHEMICAL ABSTRACTS.

Cause of deformation and breaking of stirrers in mechanical pyrites burners. K. SHABALIN (J.

Chem. Ind. Russia, 1928, 5, 521—522).—The metal becomes almost completely converted into ferrous sulphide and covered with a layer of a mixture of this substance with pyrites.

CHEMICAL ABSTRACTS.

Converter for the oxidation of ammonia with pure oxygen. J. Y. YEE (Ind. Eng. Chem., 1929, 21, 1024—1026).—The converter was designed for the production of liquid oxides of nitrogen as a step in the direct manufacture of highly-concentrated nitric acid. It is made of aluminium, in two parts, each water-cooled. A series of sharp edges on both castings are in contact with the gauze and conduct away surplus heat. Conduction direct to the converter surface is minimised to avoid unequal temperature distribution on the gauze. The oxygen-air mixture is passed through a baffle chamber and thence through very small ports to prevent the transmission of an explosion backwards from the gauze. The best volume ratio was found to be 7 vols. of oxygen to 4 vols. of ammonia, good efficiencies (up to 94%) being thereby obtained. Photomicrographs are given showing the effect on the structure of an activated gauze of overheating. The temperature of the hottest part of the gauze in the converter described was found to be 850—900°.

C. IRWIN.

Stabilising hydrogen peroxide solution with "nipagin" [methyl *p*-hydroxybenzoate]. E. BÖHM (Pharm. Ztg., 1929, 74, 1670—1671).—A solution of hydrogen peroxide containing 0.1% of "nipagin" and kept in a corked flask at room temperature, after periodical examination from 1925 to 1928, had only decreased in strength from 3.0% to 2.7% in that time. Under similar condition, solutions preserved with oxalic acid and urea had decreased in strength to 0.1% and nil, respectively.

E. H. SHARPLES.

Dehydration of Glauber salt by aqueous ammonia. G. YAKOVKIN (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 8, 5—13).—When the concentration of ammonia is about 15 g. per 100 g. of water, temperature variations have no effect on the solubility of sodium sulphate, and the heat of dissolution is zero; at lower concentrations of ammonia the solubility of the sulphate decreases slightly with rise of temperature, and the heat of dissolution of the anhydrous salt is positive, whilst in solutions containing 15—35 g. it increases with rise of temperature, indicating a negative heat of dissolution. In general, the influence of temperature is small in comparison with that of concentration of ammonia. As a dehydrating agent ammonia lowers the solubility to a greater extent than does methyl or ethyl alcohol; the expense of heat is approximately the same.

CHEMICAL ABSTRACTS.

Preparation of alkali cyanides from their carbonates, carbon, and nitrogen. N. A. FLEISHER (Trans. State Inst. Appl. Chem. Moscow, 1928, No. 10, 5—27).—A wet mixture of sodium carbonate 1, charcoal (70.7% C) 0.7, and reduced iron 0.17 pt. was briquetted, dried, and heated (*e.g.*, at 990° for 1.5 hrs.) in an iron tube in a current of nitrogen. Briquetting improves the yield (*e.g.*, 58%) of sodium cyanide; moisture is detrimental, and the addition of manganese (G.P. 176,080) is superfluous. With potassium carbonate (1.3 pts.) the yield (*e.g.*, 55% for 990°, 2.75 hrs.) is affected by the state of

division of the iron. Better yields (64%, 68%) were obtained at 1010° at atmospheric pressure, using reduced iron and potassium ferrocyanide, but not the latter alone. Potassium sulphate is detrimental. The iron was reduced by various processes.

CHEMICAL ABSTRACTS.

Preparation of barium chloride from wastes in the production of lithopone. F. N. STROKOV (Trans. State Inst. Appl. Chem., Moscow, 1928, No. 10, 28—35).—The solid residue remaining after extraction of barium sulphide from the product of reduction of barytes with carbon contains barium silicate and carbonate. By treatment with hydrochloric acid, igniting, dissolving in water, filtering, and crystallising, barium chloride containing BaCl_2 85.16, H_2O 14.93%, CaCl_2 trace, Fe and SiO_2 none, was obtained.

CHEMICAL ABSTRACTS.

Conversion of barium sulphide into barium chloride by the action of chlorine. F. N. STROKOV (Trans. State Inst. Appl. Chem., Moscow, 1928, No. 10, 36—56).—Barium sulphide was extracted with hot water, and the cold solution filtered from precipitated hydroxide and hydrosulphide; rapid or prolonged treatment with chlorine increases the amount of sulphate formed. Treatment of a filtered, hot solution (preferably containing 160—270 g. per litre) with chlorine (of which 80—94% reacted), the liquid being filtered from sulphur, gave yields of 95% of barium chloride and 90% of sulphur, of which 27% was present as barium sulphate. Complete conversion of barium sulphide into chloride is possible by passing chlorine over the dry substance heated above 60°, the exothermic reaction maintaining the temperature for a time at 90—100°; further heating to 120° is necessary, and is followed, with distillation of sulphur monochloride, by rise of temperature to 200°. After 3 hrs. the mass is washed with benzene to remove sulphur monochloride, extracted with hot water, and the filtered solution is crystallised. Over 70% of sulphur chloride is recoverable.

CHEMICAL ABSTRACTS.

Determination of the calcium sulphate content of tower liquors. E. SCHMIDT and C. HÖNN (Papier-Fabr., 1929, 27, 813—815).—Raschig's benzidine method (B., 1903, 883, 1066) has been found very suitable for rapid process-control determinations of calcium sulphate in presence of sulphur, as benzidine sulphate is completely precipitated at p_{H} 2 as in tower liquors. Thus 5 g. of benzidine are shaken with 100 c.c. of water, 4 c.c. of concentrated hydrochloric acid are added, and the solution is diluted to 2 litres. Tower liquor (50 c.c.) is then run into 100 c.c. of this reagent, the precipitate being collected and washed on a filter with 10 c.c. of cold water only. It is then washed off the filter paper by shaking with water warmed to 50° and titrated with caustic soda and phenolphthalein. When neutrality is approaching, the solution is boiled. The method gives very exact results, which are quite unaffected by sulphur dioxide concentration. It is not suitable for cooker liquors, as ligninsulphonic acids also give a precipitate with benzidine hydrochloride. C. IRWIN.

Producing alumina by acid and electrothermal processes. J. D. EDWARDS and R. B. MASON (Chem.

and Met. Eng., 1929, 36, 730—733).—Methods which have been proposed and in many instances protected for the production of alumina from ores, clays, etc. by means of acid processes are reviewed. Contrary to alkaline processes, iron and titanium are dissolved in quantity and a complete separation is practically always difficult and expensive. Electrothermal processes aim to reduce the impurities in the raw material to the metallic state and to separate them in that form from an upper layer of molten alumina. A modification of Hall's process has been operated commercially, by which a mixture of siliceous and ferruginous bauxites, properly proportioned, is reduced and the impurities are tapped out as a ferrosilicon-aluminium-titanium alloy, together with a small quantity of aluminium. The molten alumina (at 2500°) may be cast into blocks, or is preferably blown by high-pressure air or steam into small hollow granules. The process is completed by leaching the granules with dilute acid to remove lime and about half of the remaining oxides of iron, silicon, and titanium; the purity may then attain 99.75%. Another modification of Hall's process lowers the fusing point of alumina by the introduction of ferrous sulphide, which yields aluminium oxide embedded in a matrix of aluminium sulphide; this is then decomposed by water, chlorine, or hydrochloric acid. Other "nitride" dry processes as represented by the Serpek method have failed to materialise into useful commercial application owing to the difficulty of forming the nitride on a large scale, and also because of the numerous problems involved in its subsequent decomposition. C. A. KING.

Manufacture of alumina by a wet process. J. SÉAILLES (Compt. rend., 1929, 189, 1276—1279).—Interaction in solution of lime and bauxite in the proportions Al_2O_3 : 4CaO for 1½—8 hrs. at 4—8 kg. pressure in the autoclave, followed by treatment with sodium carbonate, filtration of the sodium aluminate, and precipitation with carbon dioxide, gives a 90% yield of iron-free alumina containing 0.15—3% SiO_2 . There is evidence for the formation in the autoclave of the compound $\text{Al}_2\text{O}_3 \cdot 3\text{CaO}$ (cf. Travers and Schnoutka, A., 1928, 1124). Low silica contents are ensured by the absence of free alkali before the carbonate treatment. The precipitate of ferruginous calcium carbonate is suitable for inclusion in Portland cement pastes. J. GRANT.

Preparation of pure alumina from Tikhvin bauxites. J. LILEEV (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 8, 14—38).—Packard's process gave good results with bauxite containing SiO_2 17.4, Al_2O_3 40.6, and Fe_2O_3 23.5%, but was inapplicable to samples containing, respectively, 7.6, 63.5, 14.0%, or 5.0, 74.5, 5.7%, since the reactions taking place are: $\text{Na}_2\text{CO}_3 + \text{Al}_2\text{O}_3 = 2\text{NaAlO}_2 + \text{CO}_2$ and $\text{Fe}_2\text{O}_3 + \text{Na}_2\text{CO}_3 = 2\text{NaFeO}_2 + \text{CO}_2$. It is necessary to use equimolecular ratios of sodium carbonate and alumina and ferric oxide, respectively, to adjust the ratio Na_2CO_3 : CaCO_3 to 1.5 : 2, to calcine in an oxidising atmosphere at 875—925°, and to wash with water at 90°. Under the conditions employed 85—90% of the alumina is extracted. CHEMICAL ABSTRACTS.

Reduction of tricalcium phosphate by carbon: effect of silica and alumina on the reaction. K. D.

JACOB, D. S. REYNOLDS, and W. L. HILL (Ind. Eng. Chem., 1929, 21, 1126—1132).—Silica has a marked effect in increasing the rate of reduction. Volatilisation of phosphorus begins at 1050°, and under favourable conditions is more than 90% complete in 1 hr. at 150° or in 10 min. at 1350°. As the silica content of the charge was increased there was a progressive increase in the total phosphorus volatilised; e.g., at 1200° the percentage of phosphorus volatilised increased from 45.1 with a mol. ratio of $\text{SiO}_2:\text{CaO} = 1.3$ to 65.8 with a ratio of 4.3. The effects of quartz and silica gel were similar. Alumina also accelerates the reduction, though to a less pronounced degree than silica. Rapid reduction of aluminium phosphate by carbon occurs at 1100°, and the presence of silica exerts little influence on this reaction. It is suggested that in the presence of silica carbon monoxide acts as a reducing agent towards tricalcium phosphate, or probably towards phosphorus pentoxide vapour formed by dissociation.

C. A. KING.

Gaseous ammonia for superphosphate. M. M. HALE (Chem. and Met. Eng., 1929, 36, 741).—Treatment of phosphatic fertilising materials with ammonia solution may be a new factor, but the use of gaseous ammonia has been suggested since 1867. When wet acid phosphate is treated with ammonia the phosphoric oxide reverts to insoluble form owing to a reaction between calcium sulphate and ammonium mono- or di-hydrogen phosphate. This reaction does not occur if the moisture content does not exceed 6%, neither does it revert on storage. When a pure salt was treated with ammonia a triple combination existent as a single salt, e.g., calcium ammonium phosphate, was indicated.

C. A. KING.

Utilisation of natural aluminium phosphate on the Pacific coast. Y. KIDA (Proc. III Pan-Pacific Sci. Cong., 1926 [1928], II, 1999—2000).—(1) The material was decomposed with sulphuric acid and either extracted with alcohol or acetone and treated with ammonia to give ammonium phosphate, or treated with a sulphate and lime or calcium phosphate to yield an alum and monocalcium phosphate. (2) The material was treated with sodium hydroxide solution under different conditions.

CHEMICAL ABSTRACTS.

Determination of lead peroxide. II. A. V. PAMFILOV and E. G. IVANČEVA (Z. anal. Chem., 1929, 79, 186—195; cf. B., 1929, 850).—Lead peroxide may be determined by heating until dissolved with excess of 0.15—0.20*N*-ferrous chloride solution to which 5 c.c. of concentrated hydrochloric acid per 25 c.c. have been added, then titrating with dichromate, using ferricyanide as indicator. Diphenylamine may be employed as internal indicator if only 10 c.c. of 4*N*-hydrochloric acid per 25 c.c. of ferrous chloride solution are added. Lux's method is unsatisfactory for the determination of either lead peroxide or manganese dioxide.

R. CUTHILL.

Anti-corrosive construction materials. COOPER. **Freezing mixture.** NOACK.—See I.

PATENTS.

Concentration of sulphuric acid. W. BÜSCHING (B.P. 322,977, 22.3.29).—Impure acid which has been

pretreated in a dephlegmator is fed by means of a pipe to the bottom of a closed cast-iron vessel, which is heated and fitted with a stirring apparatus. Compressed air is introduced into the stirred acid to increase the oxidising effect.

W. G. CAREY.

Purification of caustic soda. A. H. HOOKER and W. J. MARSH, Assrs. to HOOKER ELECTROCHEM. CO. (U.S.P. 1,733,879, 29.10.29. Appl., 12.8.26. Renewed 14.3.29).—A solution containing 40—46% NaOH is saturated with sodium chloride at ordinary temperature, then diluted with water, and cooled below the point at which it becomes saturated with respect to $\text{NaOH}\cdot 3.5\text{H}_2\text{O}$.

W. G. CAREY.

Dehydration of caustic [alkali]. J. F. WAIT, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,734,699, 5.11.29. Appl., 31.12.24).—Caustic alkali solution is heated under reduced pressure in a closed iron vessel only 40% of the volume of which is filled. As evaporation proceeds more solution is run in to maintain the volume nearly constant. When the temperature rises to 300° addition of solution is stopped, the pressure is reduced to 2 lb., and dehydration finished above 400°.

A. R. POWELL.

Cleansing preparation. J. ANDERSON and T. D. HEFFERNAN (B.P. 322,868, 15.11.28).—An aqueous solution containing caustic soda or potash, ammonium bicarbonate, potassium carbonate, and borax or baking soda.

H. ROYAL-DAWSON.

Production of ammonium sulphate. I. G. FARBER-IND. A.-G., Assces. of F. ACKERMANN and E. SCHUBERT (G.P. 453,686, 25.2.26).—Ammonium bisulphite dissolved in alcohol of at least 60% concentration is oxidised by treatment with air or other gases containing oxygen.

L. A. COLES.

[Manufacture of] ammonium sulphate. T. NISHIGAWA (F.P. 630,426, 7.3.27).—A mixture of ammonia, sulphur dioxide, and oxygen or gas containing oxygen enters at the base of a cylindrical tower containing filling material in the form of shells with perforated bases. A fan-type atomiser is arranged in the upper part of the tower and distributes either water or dilute aqueous solutions of ammonium sulphite or sulphate. Laminar atomisers are arranged between layers of the filling material in order to distribute liquid falling from the filling.

J. S. G. THOMAS.

Production of ammonium phosphates. J. GUILLISSEN, and UNION CHIM. BELGE Soc. ANON. (B.P. 322,700, 22.2.29).—A solution of phosphoric acid containing impurities, such as ferric salts, alumina, and lime, is partially saturated with ammonia until the impurities are precipitated, whereon the mother-liquor is separated and treated with excess of ammonia until the bulk of the triammonium phosphate is precipitated. The latter is pure and may be thermally converted into mono- or di-ammonium phosphate. The original phosphoric acid solution is preferably diluted so that ammonium phosphates are not precipitated with the impurities.

S. K. TWEEDY.

Purifying fused salt baths. W. J. MERTEN and C. T. GAYLEY, Assrs. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,736,457, 19.11.29. Appl., 10.10.25).—Impurities may be removed from fused chloride

annealing baths by the addition of a mixture of boric acid with 10–25% of powdered charcoal. Sulphates, phosphates, carbonates, and particles of slag and scale are thus removed as a spongy dross which floats on the surface of the bath and is readily skimmed off.

A. R. POWELL.

Refining of borates. W. A. GALE and C. F. RITCHIE, ASSRS. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,732,806, 22.10.29. Appl., 27.1.25).—Borate solutions containing phosphate, such as are obtained in the mother-liquors in the refining of Searles Lake borates, are purified by treatment with sufficient milk of lime to precipitate all the phosphoric acid as calcium phosphate without appreciably precipitating the boric acid.

A. R. POWELL.

Manufacture of calcium sulphite liquors. R. BÖHLIG (G.P. 455,681, 10.9.25).—Sulphur dioxide and lime water are sprayed simultaneously into an absorption tower from jets located at the bottom thereof, whereby a solution of $d 1.036$ is obtained containing calcium sulphite and excess sulphur dioxide.

A. R. POWELL.

Production of pure alumina. SALZWERK HEILBRONN A.-G., K. SCHMIDT GES.M.B.H., and K. FLOR (G.P. 455,472, 27.2.25).—The dross obtained in melting aluminium is sieved to remove coarse aluminium prills and digested with hydrochloric acid to obtain a relatively pure solution of aluminium chloride, which is then saturated with hydrogen chloride, whereby $AlCl_3 \cdot 6H_2O$ is precipitated. This is washed with concentrated hydrochloric acid and calcined to obtain alumina and hydrochloric acid for use again.

A. R. POWELL.

Treatment of phosphate rock. G. SINGLETON and R. P. THORNTON (U.S.P. 1,736,553, 19.11.29. Appl., 17.12.23).—Native aluminium phosphate (wavelite) is ground and moistened with ammonia or potassium carbonate containing sufficient alkali to convert all the phosphate into alkali phosphate. After keeping for several days the mass is dried, ground, and used directly as a fertiliser.

A. R. POWELL.

Separation of titanium dioxide hydrate from hydrolysable solutions of titanium salts. J. BLUMENFELD (B.P. 310,949, 28.1.29. Czechoslov., 5.5.28).—An inhibition period occurs in the thermal hydrolysis of titanium solutions in mineral acid during which amicroscopic nuclei serving as accumulation centres for hydrate particles are formed. It is proposed to hasten the hydrolysis by treating the solutions with a liquor containing nuclei of titanic hydroxide (to the extent of 1% of the total titanium), which nuclei are still capable of being dispersed wholly or partially by dispersing agents towards or into a condition of apparently homogeneous solution. The liquor is made by hydrolysing titanium compounds (e.g., a portion of the main solution to be hydrolysed) under conditions of low acidity (p_H 2–7, preferably 4–4.5), ripening at a moderate temperature (70–80°), and, if necessary, cooling. The conditions must be nicely adjusted to obtain a liquor having highly active nuclei, but a correctly prepared liquor maintains its activity for weeks.

S. K. TWEEDY.

Preparation of coagulating sols. R. A. HENRY (B.P. 322,798, 15.6.28).—Sols to coagulate suspensions

or emulsions in water are made by adding amylaceous material or living cells, e.g., starch, aquatic weeds, vegetables, etc., together with potassium, sodium, or calcium hydroxides to the water after neutralising its acidity with lime. The amylaceous material is maintained in suspension by a pump working in a closed circuit, and is mixed with the hydroxide in a channel where the speed of the common flow and the length of the path travelled can be adjusted so as to govern the duration of the reaction.

W. G. CAREY.

Manufacture of colloidal metallic salts. R. LORENZ and H. HEINZ (G.P. 456,188, 30.7.24).—Such salts are produced by reaction between one or more components present in the colloidal state, the remaining components being present in the massive form. The resulting solution is evaporated and solid reversible colloids are obtained.

J. S. G. THOMAS.

Production of hydrogen. R. WILLIAMS, ASSR. to DU PONT AMMONIA CORP. (U.S.P. 1,736,065, 19.11.29. Appl., 5.8.26).—A mixture of steam and a gaseous hydrocarbon, e.g., methane, is passed through a series of reaction vessels containing a number of superimposed trays containing a suitable catalyst, and the heat of the reaction is maintained by admitting small quantities of air to the free spaces just below every tray so that only a small part of the mixture is converted into hydrogen and carbon monoxide at every stage of the treatment, thus avoiding overheating and undesired side-reactions.

A. R. POWELL.

Production of nitrogen and hydrogen. KALI-IND. A.-G., and C. T. THORSELLE (B.P. 298,190, 24.9.28. Ger., 5.10.27. Addn. to B.P. 288,154; B., 1929, 719).—Coke-oven, blast-furnace, lignite-distillation gases etc. containing a proportion of carbon dioxide too high for satisfactory use in the reduction stage of the prior process are rendered suitable by being passed over coke at a temperature such that in the treated gases the ratio $CO_2 : CO$ does not exceed 0.45 and the ratio $CO : CH_4$ is at least 1.5. Alternatively, a portion only of the gas is treated and mixed with the remaining gas in proportion such that the mixture conforms to the required standard.

L. A. COLES.

Manufacture of mixtures of nitrogen and hydrogen for the synthesis of ammonia. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,701, 22.2.29. Addn. to B.P. 307,529; B., 1929, 393).—Part of the air added before the exposure of the carbonaceous material to the electric arc is replaced by carbon dioxide, which latter is converted by the arc with liberation of oxygen into carbon monoxide (from which the requisite carbon dioxide is produced, e.g., by passing with steam over a nickel catalyst). The nitrogen and hydrogen in the resulting gas mixture are more nearly in the proportion of 1:3 than in the mixture obtained by the process previously described.

S. K. TWEEDY.

Purification of nitrogen, hydrogen, or mixtures thereof. SOC. D'ETUDES MINIERES & INDUSTRIELLES (B.P. 302,306, 16.11.28. Fr., 14.12.27).—Nitrogen and hydrogen containing oxidisable impurities, e.g., carbon monoxide, and/or hydrocarbons, and also reducible impurities, e.g., sulphur or phosphorus compounds, are humidified and mixed with water vapour and with

sufficient oxygen and hydrogen to combine with the oxidisable and reducible impurities, respectively, and then passed under pressure over charcoal at not above 600° (except in the case of nitrogen), the conversion products of the impurities then being removed by known means.

W. G. CAREY.

Recovery of iodine. E. KUREK (U.S.P. 1,731,309, 15.10.29. Appl., 29.11.22).—Iodine present in limestone or dolomite is recovered by passing the gases, liberated in the burning process, through a condenser and then into water or an aqueous alkaline solution.

F. G. CLARKE.

Production of absorbent material. T. P. HILDITCH and H. J. WHEATON, Assrs. to H. N. HOLMES (U.S.P. 1,739,305, 17.12.29. Appl., 13.8.23. U.K., 14.8.22).—See B.P. 206,268; B., 1924, 22.

Production of soluble lead reagents. D. W. PARKES, Assr. to H. W. ROBINSON (U.S.P. 1,740,312, 17.12.29. Appl., 31.10.28. U.K., 17.2.28).—See B.P. 305,827; B., 1929, 322.

Apparatus for converting carbon dioxide or other material into a solid. J. E. MORTIMER (B.P. 322,807, 14.9.28).

Solidifying gases (B.P. 301,741). **Anti-freezing mixture** (B.P. 322,859).—See I. **White lead** (U.S.P. 1,732,490).—See XIII. **Fertilisers** (B.P. 291,461 and 322,424). **Herbicide** (B.P. 322,438).—See XVI.

VIII.—GLASS; CERAMICS.

Relationship between chemical composition and thermal expansion of glasses. S. ENGLISH and W. E. S. TURNER (J. Amer. Ceram. Soc., 1929, 12, 760).—Errors in calculation are pointed out by the authors, in their previous results (B., 1928, 262) for the cubical expansion factors of certain oxides. The true figures should be zinc oxide 2.1, alumina 0.42, baryta 4.2.

J. A. SUGDEN.

Density factors for soda-lime glasses. S. R. SCHOLES (J. Amer. Ceram. Soc., 1929, 12, 753—755).—Density factors calculated in terms of Na_2O , 2SiO_2 , CaO , SiO_2 , MgO , SiO_2 , SiO_2 , and Al_2O_3 have given better agreement with the observed densities of a series of glasses than have the factors based on the individual oxides. (Cf. English and Turner, B., 1921, 891 A.)

J. A. SUGDEN.

Relation between the density and refractive index of silicate glasses, with application to the determination of imitation gem-stones. F. A. BANNISTER (Min. Mag., 1929, 22, 136—154).—Data taken from various sources, e.g., Peddle (B., 1920, 545 A, 783 A; 1921, 216 A, *et seq.*), Wright (B., 1921, 10 A, 487 A), are plotted in various ways. It is found that when $(n - 1.460)/(d - 2.21)$, in which 1.460 and 2.21 are the refractive index and density of silica glass, respectively, is plotted against the refractive index n , there is a wider separation of the different glass families of various chemical compositions. In doubtful cases a further aid is given by a determination of the optical dispersion. New data are given for thallium glasses and a glass with the composition of emerald.

L. J. SPENCER.

Determination of strain in glass by a radiation method. L. R. INGERSOLL (J. Amer. Ceram. Soc.,

1929, 12, 756—759).—In the ordinary optical (polarised light) test for strain, the eye has been replaced by a thermopile. Certain glasses which are too opaque to be tested with visible radiation pass sufficient infra-red rays to be tested with the modified apparatus. It is also possible to test rough-surfaced diffusing glasses. The apparatus can be made suitable for factory use.

J. A. SUGDEN.

Greater efficiency in tunnel dryers. W. D. RICHARDSON (J. Amer. Ceram. Soc., 1929, 12, 761—764).—Decreased efficiency is due to a cold bottom in the exit end of the tunnel, and to the hot air rising immediately to the top and drying only the upper part of the ware, thus emerging from the dryer but partially saturated. The improved dryer is provided with a hot-air duct throughout its whole length, and with a series of baffle plates and fans in order to circulate the air between the bottom and top.

J. A. SUGDEN.

Tunnel kiln installation for porcelain insulators. E. H. FRITZ (J. Amer. Ceram. Soc., 1929, 12, 711—715).—Certain details of equipment are described, the object of which is to give uninterrupted operation and more perfect control of the kiln.

F. SALT.

Oil firing of refractories. R. C. ZEHR (J. Amer. Ceram. Soc., 1929, 12, 742—745).—Data are presented on the construction and operation of round, down-draught kilns firing refractories with fuel oil.

F. SALT.

Chemical changes in clay and clay-lime mixtures on firing. C. J. FRANKFORDER and J. N. DETRICK (J. Amer. Ceram. Soc., 1929, 12, 771—779).—A Nebraska clay to which lime had been added was fired at 1000°. The lime appeared to combine with the various constituents of the clay, and there was also reaction between the iron oxide and silica.

J. A. SUGDEN.

Glost warping of white wall tile. T. D. TEFFT (J. Amer. Ceram. Soc., 1929, 12, 765—770).—A simple apparatus for measuring the degree of warping is described, with which the influence of glaze composition on warping has been studied. The warping seems to be increased by the addition of an oxide which renders the glaze more fusible. Zinc, barium, and magnesium oxides and excess of lime increase the glost warping.

J. A. SUGDEN.

Chrome [ore] and diasporite mixtures. H. G. JONES (J. Amer. Ceram. Soc., 1929, 12, 732—737).—An investigation was conducted to determine the softening points of various mixtures of chromite and diasporite, the workability of mixtures made by different processes, the porosity, firing and reheating shrinkage, and the resistance to slag action and spalling. All the mixtures stood up to cones 33—34. The dry-press method was the most suitable for making the test pieces. The addition of 50% of chromite reduced the shrinkage of raw diasporite to about one third. Chromite and diasporite have nearly the same absorption, and this was not changed in the mixtures. The addition of a small amount of finely-ground chromite reduced the action of slag on the bricks. The addition of diasporite to chromite increased its resistance to spalling, and the addition of fine chromite to diasporite also increased its resistance to spalling.

F. SALT.

X-Ray study of firebrick. A. E. R. WESTMAN (Univ. Ill. Eng. Exp. Sta. Bull., 1929, No. 193, 16 pp.).—In all the bricks examined mullite lines and one cristobalite line were present, the quartz lines being masked or faint. The scope of the method is discussed.

CHEMICAL ABSTRACTS.

Transverse breaking strength of refractories. C. A. VINCENT-DAVISS (J. Amer. Ceram. Soc., 1929, 12, 738—739).—Furnace equipment described by Hartmann and Koehler (B., 1921, 771 A) has been modified so as to give continuous operation of the transverse crushing test at temperatures up to 1350° and 1500°. Two pairs of furnace units are arranged side by side, each unit being mounted on a base fitted with four wheels which run on rails. While one unit of a pair is being heated up the other is cooling.

F. SALT.

Moulding sand. TAKAHASHI.—See X.

See also A., Jan., 31, **Apparatus for dispersoid analysis** (ANDREASEN and others). 55, **Porcelain evaporating basins etc.** (VOLKMANN).

PATENTS.

Production of heat-absorbing glasses and batches therefor. CORNING GLASS WORKS, Assees. of W. H. RISING (B.P. 312,311, 17.4.29. U.S., 24.5.28).—The addition of small amounts of tin or its compounds to glasses containing ferrous iron, a reducing agent, and one or more elements (at. wt. 20—140) of group II enables the iron to be much more completely reduced. This complete reduction to the ferrous form imparts to the glass a blue coloration and the property of absorption of the visible red, infra-red, and ultra-violet, together with relatively high transmission for the remainder of the spectrum.

J. A. SUGDEN.

Manufacture of glass transparent to ultra-violet rays. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY ET CIREY (B.P. 291,468, 2.6.28. Fr., 4.6.27).—From 0.05 to 0.5% of manganese oxide is incorporated in the batch, and melting is performed in a reducing atmosphere, subsequent "solarisation" being thus prevented.

A. COUSEN.

Manufacture of strengthened glass. H. D. MURRAY and D. A. SPENCER (B.P. 322,598, 25.10.28).—The discoloration of the layer of celluloid in "safety glass" can be avoided (i) by mixing with the celluloid a substance such as (a) quinine sulphate or æsculin, to convert the ultra-violet light into a light of less active wave-length, or (b) sodium silicate or glycerin, to inhibit the decomposition of the celluloid or to form colourless compounds with the coloured decomposition products thereof; or (ii) by using glass elements impermeable or less permeable to ultra-violet light.

J. A. SUGDEN.

Matting the interior surface of hollow glass bodies. ELEKTR. GLÜHLAMPENFABR. "WATT" A.-G. (B.P. 302,582, 14.12.28. Austr., 17.12.27).—The two processes of etching and consolidating after-etching are carried out simultaneously by the application of a solution of ammonium hydrogen fluoride, calcium chloride, water, and hydrofluoric acid, which has been rendered viscous by the addition of finely-ground barium sulphate. The etching action generates heat which

serves to liberate, by hydrolysis, the hydrofluoric acid for after-etching.

J. A. SUGDEN.

Vitrified compositions and abrasive objects manufactured therefrom. Grinding wheels and similar abrasive articles. C. J. BROCKBANK (B.P. 322,448—9, 3.9.28).—(A) A mixture of felspar 70 pts., china clay 25 pts., and titanium dioxide 5 pts. is fritted at cone 15—20 and finely ground. The abrasive or other material is mixed with 15% of the frit and a small amount of dextrin, dried, and fired at cone 13. The process of moulding and firing may be completed in 24 hrs., and gives a strong and tough material. (B) Ethylene glycol monoethyl ether is used as the solvent in the bonding of abrasive materials with shellac, rubber, resins, etc. The moulding can be carried out in the cold, after which the article is baked at 140—220°.

J. A. SUGDEN.

Production of lepidolite enamel. C. TRENZEN, ASSR. to C. SCHWARTZ and G. G. G. HUNTER (U.S.P. 1,734,698, 5.11.29. Appl., 9.7.26. Ger., 10.7.25).—Iron goods may be enamelled directly without an under-glaze coat by covering them with a paste of powdered lepidolite and water, drying, and firing just below 1000°. The resulting enamel is white, glossy, and opaque.

A. R. POWELL.

Drying and firing of ceramic ware in the same kiln. F. HAGER (G.P. 447,970, 26.7.25).—The hot gases introduced into the kiln for drying the ware are charged with water vapour by passing over a vessel of water in the entry flue, and when the drying is completed the vessel is removed so that dry gases are available for firing purposes.

L. A. COLES.

Refractory crucibles. C. J. BROCKBANK (B.P. 322,476, 3.9.28).—Titanium dioxide, silicate, or aluminate is added to the clay bond used in the manufacture of graphite and carborundum crucibles. When fired at cone 5—10 a very strong mass is produced having a glazed surface which prevents the oxidation of the graphite etc.

J. A. SUGDEN.

Production of heat-absorbing glasses and batches therefor. W. H. RISING, ASSR. to CORNING GLASS WORKS (U.S.P. 1,737,686, 3.12.29. Appl., 24.5.28).—See B.P. 312,311; preceding.

Light-weight ceramic material and its manufacture. R. ERICSON, ASSR. to UNITED STATES GYPSUM CO. (Re-issue 17,523, 17.12.29, of U.S.P. 1,702,076, 12.2.29).—See B., 1929, 284.

Method and apparatus for producing sheet glass. A. E. WHITE. From MISSISSIPPI GLASS CO. (B.P. 322,759, 11.9.28).

Leading-in wire for glass (U.S.P. 1,734,900).—See X.

IX.—BUILDING MATERIALS.

Method of studying the reactions in a Portland cement kiln. W. N. LACEY and H. WOODS (Ind. Eng. Chem., 1929, 21, 1124—1126).—In order to sample material in any part of a rotary kiln without the necessity of stopping the kiln, a cup provided with an extended lip to prevent the collection of material until the desired moment and to protect it after sampling is mounted on

an iron rod which may be inserted to any position along the length of the kiln. Results of analyses of samples drawn in this manner indicate that comparatively little calcination of limestone occurs in the first 45 ft., calcination is rapid in the next 25 ft., and extensive recombination of lime takes place after this stage. The progress of calcination was followed by observing the loss on ignition and the uncombined calcium oxide, the latter being determined by titration of the lime with ammonium acetate in absolute alcohol solution, the ammonia formed being expelled by heating and therefore allowing the use of phenolphthalein as indicator. C. A. KING.

Action of sulphates on Portland cement. II. Steam-curing of Portland cement mortar and concrete as a remedy for sulphate (alkali) action. III. Effect of addition of silica gel to Portland cement mortars on their resistance to sulphate action. T. THORVALDSON, V. A. VIGFUSSON, and D. WOLOCZOW (Canad. J. Res., 1929, 1, 259—384, 385—399).—II. The resistance of mortar and concrete to the action of sulphate solutions is very considerably increased by curing in steam for 24 hrs. or more. Curing at 100° reduces considerably the expansion caused by sodium or calcium sulphate and retards that caused by magnesium sulphate, but the curing process produces mortars of low tensile strength and does not preclude the possibility of failure after prolonged exposure to sulphate solutions. Much more satisfactory tensile and compressive strength coupled with a greater resistance to sulphate action is obtained by steam-curing under pressure at 125—175° for 24 hrs. After this treatment the expansion of mortar or concrete in sodium or calcium sulphate solutions is almost entirely eliminated and expansion in magnesium sulphate solution greatly reduced. The behaviour of cements of varying composition after this treatment in sulphate solutions of various concentrations has been examined and the results are reproduced in a series of 19 tables.

III. Replacement of part of the Portland cement in cement-sand mortars by silica gel up to 20% of the total increases slightly the resistance of the steam-cured mortar to the disintegrating action of sodium sulphate or calcium sulphate solutions (see above), but decreases the resistance to magnesium sulphate. Lime-silica gel-sand mortars behave similarly in sulphate solutions to the corresponding cement mortars. During the setting of either type of mortar to which the gel has been added the silica combines with the free lime liberated, in the case of cement mortars, during hydrolysis of the basic silicates present; the product is itself a gel which crystallises only very slowly. Steam-curing of cements free from silica gel also causes the free lime to disappear, with the formation of an isotropic variety of tricalcium aluminate which is not easily hydrolysed by sulphate solution. Apparently it is only the common hexagonal plates and needles of tricalcium aluminate found in ordinary cement mortar after setting which are attacked by sulphate solutions, thus causing expansion and disintegration of the mortar. A. R. POWELL.

Formation of two different calcium sulphato-aluminates from gypsum in cement. P. MECKE (Tonind.-Ztg., 1929, 53, 681—684; Chem. Zentr., 1929,

ii, 340).—Interaction of lime, alumina, and the compound $3\text{CaO}, \text{Al}_2\text{O}_3, 3\text{CaSO}_4$ affords a second sulphatoaluminate according to the equation: $2(3\text{CaO}, \text{Al}_2\text{O}_3, 3\text{CaSO}_4) + 3\text{CaO}, \text{Al}_2\text{O}_3 = 3(3\text{CaO}, \text{Al}_2\text{O}_3, 2\text{CaSO}_4)$. In the formation of the second salt the whole of the sulphate is precipitated, even from dilute solutions.

A. A. ELDRIDGE.

Emulsions. EADIE; NOVAK.—See II.

See also A., Jan., 31, **Apparatus for dispersoid analysis** (ANDREASEN and others).

PATENTS.

Manufacture of concrete. W. O. ZIMMERMANN (G.P. 448,030, 31.7.26).—Mortar prepared from 1 pt. of quick-lime and 16—18 pts. of sand is ground and mixed with 1 pt. of cement before it has set completely.

L. A. COLES.

Solidification of sandy masses permeable by water. TIEFBAU- U. KALTEIND. A.-G., SIEMENS-BAU-UNION GES.M.B.H. KOMM.-GES., and H. JOOSTEN (B.P. 322,182, 24.5.28).—Porous sandy rock, constructional work, etc. is treated with an alkali silicate or silicic acid solution and then with an acid or sodium chloride solution. The material may also be pretreated with water, soap solution, etc., and finally treated with organic salt or acid solutions etc. [Stat. ref.]

L. A. COLES.

Impregnated wood and process of treating wood. Fireproofed wood. J. R. COOLIDGE, ASSR. to MONTAN, INC. (U.S.P. 1,734,714—5, 5.11.29. Appl., [A] 5.4.27, [B] 13.12.26. Renewed [A] 13.4.29).—(A) Wood is impregnated with a solution of zinc chloride or borax together with aluminium sulphate and then treated with a soap solution to precipitate an insoluble aluminium soap in the outer layers to act as a seal for the preservative salts within. (B) A solution of ammonium chloride and borax is used as the impregnating and fireproofing agent and, after kiln-drying, the wood is treated with soap solution to seal the pores. A. R. POWELL.

Liquefier for preparing stone for bitumen coating. S. S. SADTLER, ASSR. to AMIESITE ASPHALT CO. OF AMERICA (U.S.P. 1,739,652, 17.12.29. Appl., 18.8.27).—See B.P. 309,634; B., 1929, 520.

Drying system (B.P. 321,921). **Drying stone, slag, etc.** (B.P. 322,274).—See I. **Hard pitch** (G.P. 453,712). **Bituminous emulsions and dispersions** U.S.P. 1,733,493—8).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Effect of various factors on the open-hearth steel process investigated by large-scale tests. A. HERBERHOLZ (Arch. Eisenhüttenw., 1929—1930, 3, 173—184; Stahl u. Eisen, 1929, 49, 1579—1581).—A summarised account is given of the results of a series of tests, extending over a year and including 2288 charges, designed to determine the effect of varying the calorific value of the gas and the time occupied in charging the furnace in the open-hearth steel process. When the calorific value of the gas fell below 4150 kg.-cal./m.² the heat consumption of the furnace increased by 5—7% compared with normal practice. An increase or decrease

of $\frac{1}{2}$ hr. in the time of charging a 30-ton furnace decreased or increased the heat consumption by about 8%.

A. R. POWELL.

Relation between α -veining and the A3 transformation point. E. AMMERMANN and H. KORNFELD (Arch. Eisenhüttenw., 1929—1930, 3, 307—311; Stahl u. Eisen, 1929, 49, 1581; cf. Rawdon and Berglund, A., 1928, 939).—Annealing experiments with a mild steel containing 0.13% C and with electrolytic iron with 0.03% C showed that the appearance of α -veins throughout the ferrite grains on etching with Fry's reagent occurs only when the annealing temperature exceeds the A3 point. Annealing between the A1 and A3 points produces local veining in those ferrite grains which are in close proximity to cementite or pearlite fields, probably owing to a local reduction of the A3 point caused by segregation of the carbon content. Deformation below the A3 point does not produce veining unless the metal is subsequently annealed above this point, in which case the deformed metal shows the veining more markedly than metal which has been simply annealed without deformation.

A. R. POWELL.

Inhibitors in the action of acid on steel. F. H. RHODES and W. E. KUHN (Ind. Eng. Chem., 1929, 21, 1066—1070).—The measure of inhibiting action was taken as the percentage decrease in the rate of formation of hydrogen by the action of dilute acid on iron due to the presence of organic materials, which in these experiments were chiefly heterocyclic compounds of nitrogen. In compounds of this type the inhibiting effect increased with the mol. wt. and also with the introduction of a methyl, phenyl, or amino-group. Thus in such a series as pyridine-quinoline-acridine the increase in the number of rings in the molecule was accompanied by a regular increase in effect, and in the series pyridine-lutidine-picoline-collidine the introduction of a single additional methyl group increased the inhibiting action of the compound by nearly 25%. Hydroxyl groups appeared to decrease inhibiting action, and the most efficient of all the compounds studied were those containing several alkyl and amino-groups substituted in an acridine nucleus; thus 3:7-diamino-2:8-dimethyl-acridinum ethochloride and -acridine hydrochloride. The addition of a specific inhibitor to an acid electrolyte increased the film resistance, and it is indicated that the protective action is due to some specific property of the observed film and not solely to the increased resistance offered.

C. A. KING.

Thermal analysis of quenched carbon steels. S. SATO (Sci. Rep. Tôhoku Imp. Univ., 1929, 18, 303—316).—When quenched plain-carbon steels containing 0.2—1.8% C are heated very slowly an evolution of heat takes place in the range 100—170° with a poorly-marked maximum at 150° corresponding with the transformation of α - into β -martensite. At 260—300° the latter decomposes into troostite with the evolution of a fair amount of heat; at the same time any residual austenite is transformed rapidly through α - and β -martensite into troostite again with the evolution of heat. Further evolution of heat occurs in the ranges 300—400° and 450—600°; the former is attributed to the crystallisation of very fine cementite and the latter,

which is very small, to recrystallisation of α -iron. Retained austenite is transformed into the two modifications of martensite at the temperature of liquid air, especially when subjected to magnetic agitation at this temperature.

A. R. POWELL.

Gravimetric determination of chromium in steel. W. F. POND (Chemist-Analyst, 1929, 18, 11).—The steel (0.5—1.5 g.) is dissolved in hydrochloric (25 c.c.), nitric (5 c.c.), and sulphuric (1 c.c.) acids (all 6N), the solution evaporated to 10 c.c., and heated for a few minutes with 100 c.c. of hot water. After cooling and cautious addition of sodium peroxide (2 g. in excess), the solution is heated for 30 min. below the b.p. and filtered; the filtrate and (hot water) washings are boiled for 30 min., acidified with acetic acid, and the chromium is precipitated as lead chromate.

CHEMICAL ABSTRACTS.

Detection and determination of cobalt in special steels. M. E. BERTRAND (Bull. Soc. chim. Belg., 1929, 38, 364—371).—Cobalt may be detected by treating 1 g. of steel with 20 c.c. of concentrated hydrochloric acid, making ammoniacal, and adding 3.5 c.c. of 30% tartaric acid. On then adding 2% potassium ferricyanide solution cobalt gives a reddish-brown colour. Interference by nickel and copper can be prevented by adding potassium cyanide until the blue colour produced by ammonia just disappears. Experimental details are given for the separation of cobalt from nickel by precipitation in boiling solution with bromine in the presence of excess of zinc oxide and zinc salts, and for the electrolytic determination of the cobalt.

C. W. GIBBY.

Solvent action of ferrous sulphate solution on oxidised copper ore. W. G. MURRAY (Chem. Eng. Min. Rev., 1929, 22, 52—54).—Leaching tests have been made on an oxidised copper ore carrying 6% Cu as malachite, chrysocolla, and cuprite, using a 5% solution of commercial ferrous sulphate crystals in sea-water or in fresh water. In the absence of air and in a neutral solution very little copper was dissolved; with free access of air a maximum of 56% of the total copper dissolved readily, although 98% was easily dissolved by 2N-acetic acid. The solvent action of ferrous sulphate is due to its oxidation to ferric sulphate, which behaves as a weak acid, the iron being precipitated slowly as a basic salt by the copper minerals. Malachite is far more active in this respect than chrysocolla.

A. R. POWELL.

Surface dezincification of brass and tombak. E. BEUTEL and A. KUTZELNIGG (Z. Metallk., 1929, 21, 412—413).—Six methods of obtaining a clean copper surface on brass and tombak are briefly described. Quenching tombak from a red heat in 10% sulphuric acid produces a bright red matt surface and the solution contains copper, but no zinc, sulphate; heating and quenching in water results in the formation of an outer layer of cupric and cuprous oxides above a layer of almost pure copper, and the sulphuric acid simply dissolves the outer layer, leaving the copper film exposed. A bright copper coating on brass is obtained by immersing the brass in 20% ferric chloride solution, washing to remove soluble salts without breaking the cuprous chloride film, then heating at 120°, whereby the zinc in the brass reduces this film to metallic copper. By

more prolonged immersion in ferric chloride solution a rose-red film is produced on brass by dezincification of the outer layer. This film is produced more rapidly and rendered thicker by placing an electronegative metal in contact with the alloy during this treatment, which is applicable both to tombak and brass. A. R. POWELL.

Application of the potentiometer to the determination of chlorides in zinc dross. N. JOASSART and E. LECLERC (Bull. Soc. chim. Belg., 1929, **38**, 360—363; cf. A, 1929, 785).—A potentiometric method, using the authors' gelatin electrode, gives satisfactory results even in the presence of zinc, tin, chromium, aluminium, manganese, and copper. C. W. GIBBY.

Effect of copper and zinc in cyanidation with sulphide-acid precipitation. E. S. LEAVER and J. A. WOOLF (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 250, 23 pp.).—A proposed process for the cyanidation of gold-silver ores containing less than 0.5% of cyanide-soluble copper is based on the regeneration, by a combination of sulphide and acid precipitation, of about 80% of the cyanide used in dissolving the copper. The resulting solution is made alkaline with lime and the gold is precipitated with zinc dust.

CHEMICAL ABSTRACTS.

Effect of small quantities of third elements on the ageing of lead-antimony alloys. E. E. SCHUMACHER, G. M. BOUTON, and L. FERGUSON (Ind. Eng. Chem., 1929, **21**, 1042—1044).—The normal rate of precipitation of antimony in a lead-antimony alloy due to ageing is influenced greatly by the presence of minute quantities (0—0.01%) of a third element. Changes were observed by means of resistance measurements on wires of a lead alloy containing 1% Sb and a third element. The rate of precipitation of antimony was increased first by the presence of arsenic, being followed closely by manganese, copper, and silver, though nickel retarded the effect after three days. Quantities of impurity greater than 0.01% did not produce proportionately increasing effects.

C. A. KING.

Determination of non-sulphide lead. H. K. HANSEN (Chemist-Analyst, 1929, **18**, 10—11).—For the determination of lead present as sulphate, oxide, or carbonate the finely-divided sample (2.5—5 g.) is heated to incipient boiling for 5 min. with 20 c.c. of concentrated ammonium acetate solution containing 5 c.c. of acetic acid; the mixture is filtered and 1—2 c.c. of saturated sodium sulphide solution is added to the filtrate. After boiling, the precipitate is collected, washed with cold water, treated with nitric acid and potassium chlorate, and evaporated to fuming with sulphuric acid (5—7 c.c.), the determination being completed in the usual manner. In an alternative procedure the sulphuric acid is added to the first filtrate. Directions for the determination of zinc and iron are also given.

CHEMICAL ABSTRACTS.

Metals of the tungsten and tantalum groups. C. W. BALKE (Ind. Eng. Chem., 1929, **21**, 1002—1007).—An account is given of the preparation and uses of metals of this group. As the grain size of a sintered ingot of tungsten is of the utmost importance in the later mechanical processes, and is affected by the original

purity of the material, the density of the oxide, the rapidity of reduction, and the conditions of heat treatment, great care has to be taken in the purification of the crude tungstic acid which is dissolved in ammonia and evaporated to obtain ammonium paratungstate. This salt is treated with hydrochloric and nitric acids in proper proportions to reproduce tungstic acid, and this cycle must usually be repeated five times to reduce the total impurities present, e.g., iron, calcium, aluminium, etc., to negligible quantity. The reduction of tungstic acid in hydrogen may be controlled so as to vary the product from a light fluffy powder (apparent d_{15}) up to a very coarse-grained powder (apparent d_{100}). The preparation of molybdenum is essentially identical with that of tungsten, taking into account the lower m.p. (2620°) of the metal. Tantalum and niobium are usually associated in ores found only in pegmatite dikes, and are separated by recrystallisation of the double fluorides of potassium, the successful production of these metals depending on the purification of the metal powder from which the ingots are sintered. Both metals exhibit great avidity for hydrogen, oxygen, and nitrogen, and any heat treatment can be conducted only in specially constructed vacuum furnaces.

C. A. KING.

Inclusions in [Japanese] moulding sand. E. TAKAHASHI (Sci. Rep. Tôhoku Imp. Univ., 1929, **18**, 329—340).—Sand from the Kunimitôge district of Japan contains small rounded grains of a volcanic glass which contain small gas bubbles. This causes them to decrepitate at high temperatures, and consequently when the sand is used for moulds the castings have a poor skin and are subject to surface deformation. Satisfactory castings were obtained in moulds made from sand that had been heated at 600° and mixed with 10% of clay bond.

A. R. POWELL.

Water-hammer erosion. S. S. COOK (Proc. Univ. Durham Phil. Soc., 1929, **8**, 88—100).—Calculation of the water-hammer pressure set up by a jet of water impinging on a metal surface in a high vacuum, such as arises by the condensation of steam by expansion, shows that this pressure may frequently exceed the elastic limit of the metal, in which case serious erosion occurs. This effect may also be caused in turbines by the impact of condensed drops of water on a rapidly rotating blade. Experiments with a rotor containing blades of various metals run for 18½ hrs. at 8800 rev./min. and setting up water-hammer pressures of 12—20 tons/in.² showed that under these conditions staybrite steel, monel metal, and mild steel with elastic limits of 16—18 tons/in.² were badly eroded, whereas Hecla A.T.V. steel and tungsten steel with elastic limits of 31—40 tons/in.² were scarcely attacked. The action of collapsing cavities is to intensify the water-hammer effect; some experiments with the water-hammer cone devised by Parsons showed that it was possible to puncture a brass plate 0.03 in. thick in one blow by the collapsing cavity effect, which must therefore have set up a pressure of about 140 tons/in.²

A. R. POWELL.

Reducing and oxidising agents and lime consumption in flotation pulp. L. KLEIN, C. R. KING,

T. F. MITCHELL, O. E. YOUNG, F. H. MILLER, L. M. BARKER, and O. C. RALSTON (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 224, 17 pp.).—Atmospheric oxygen is absorbed by pyritic flotation pulps made alkaline with lime, sulphide minerals then giving rise to soluble sulphide and thiosulphates. Lime is consumed by interaction with sulphide and gangue minerals, probably by surface adsorption, and by interaction with oxidation products, atmospheric carbon dioxide, and constituents of the water. CHEMICAL ABSTRACTS.

Significance of wetting and adsorption in flotation process. E. BIERBRAUER (Z. tech. Physik, 1929, 10, 139—141; Chem. Zentr., 1929, ii, 344).—The flotation of apatite and cassiterite with sodium palmitate is considered. A. A. ELDREDGE.

Chromium-plating experiments. C. M. ALTER and F. C. MATHERS (Amer. Electrochem. Soc., Sept. 1929. Advance copy. 7 pp.).—The effect of certain special factors on the deposition of chromium from the standard type of plating bath has been examined. Under conditions giving a good current efficiency of chromium deposition at a copper cathode comparatively low efficiencies were obtained at cathodes of platinum, palladium, or Acheson graphite. The deleterious effect of cobalt salts in the plating bath is very marked, 0.5 g. of cobalt chromate per 100 c.c. preventing the deposition of chromium altogether, whereas copper, zinc, cadmium, ferric iron, nickel, molybdenum, vanadium, or aluminium had little effect up to 5 g. per 100 c.c. The action of cobalt is regarded as due to its catalysing the reduction of chromic acid, and thus reducing the cathode potential below that necessary for chromium deposition. Periodic reversal of the current during electrolysis or periodically raising the cathode from the bath greatly reduced the current efficiency, often to zero, thus supporting the view that chromium deposition occurs from a layer of partially reduced solution formed at the cathode surface. Adding silica gel to the bath produced no definite effect on the current efficiency, and did not improve the throwing power (cf. U.S.P. 1,705,954; B., 1929, 401). The current efficiency of chromium deposition was materially raised by surrounding the anodes with porous cups, but the deposited metal was soft, dark, and non-adherent. H. J. T. ELLINGHAM.

Anti-corrosive construction materials. COOPER.—See I.

See also A., Jan., 19, **Analysis by X-ray emission spectra** (SCHREIBER). 22, **X-Ray investigations of copper amalgams** (KATON). 43, **Equilibria in reduction, oxidation, and carbonisation processes in iron** (SCHENCK and others). 45, **Electrolysis of metallic oxides** (ANDRIEUX). 47, **Extraction of germanium** (PUGH). 53, **Separation of lead and bismuth** (COLLIN). 54, **Separation of platinum metals and alloys** (WADA and SAITŌ).

PATENTS.

Roller-hearth furnace. R. M. HORTVET, Assr. to MAHR MANUF. Co. (U.S.P. 1,735,968, 19.11.29. Appl., 28.9.28).—A furnace for the heat treatment of rounded

steel articles comprises a longitudinal furnace chamber provided with transverse I members above the refractory bottoms, these members carrying a number of rollers of varying diameter so as to support castings of different sizes and to prevent buckling while they pass through the furnace. A. R. POWELL.

Briquetting of blast-furnace or other ferruginous dust. G. WEBER (F.P. 628,408, 2.2.27).—The materia is ground to destroy the spherical shape of the particles, mixed with carbonaceous material, ferruginous limestone, cement, tar, and other fluxes or binders, and the mixture briquetted in known manner.

A. R. POWELL.

Production of cast iron in a coke-fired blast furnace. M. NEUMARK (F.P. 629,277, 18.2.27).—The charge is smelted under such conditions that the slag obtained contains less than 10% SiO_2 , more than 30% Al_2O_3 , and is rich in lime. This is effected by adding bauxite to the fluxes, and including a proportion of ilmenite in the charge; the titania content of the latter replaces the usual silica in the slag. The resulting iron contains less than 0.5% Si, less than 0.4% Mn, not more than 0.05% S, and about 4% C. The slag is suitable for the manufacture of cement.

A. R. POWELL.

Preparation of open-hearth steel. J. P. ROBERT (F.P. 629,508, 19.2.27. Spain, 20.2.26).—Steel turnings are melted in the open-hearth furnace and a mixture of scrap steel and cast iron is melted in a cupola. The metal from the latter furnace is tapped directly into the former, and the refining finished as usual. By adding carbon to the charge in the cupola the quantity of cast iron required can be appreciably reduced.

A. R. POWELL.

Hardening of steel. S. I. SALZMAN (U.S.P. 1,732,244, 22.10.29. Appl., 29.3.28).—Thin bands of steel, such as is used for making safety-razor blades, are drawn successively through a furnace in which they are heated to the hardening temperature, a water-cooled tube in which they are cooled rapidly, a second furnace in which they are tempered, and a second water-cooled tube. In the last three treatments pressure is applied to prevent deformation.

A. R. POWELL.

Method and apparatus for annealing. O. TANGRING, Assr. to AMER. STEEL & WIRE Co. OF NEW JERSEY (U.S.P. 1,734,697, 5.11.29. Appl., 2.4.27).—An apparatus for annealing iron or steel wire in coils comprises an annular pot filled with a mixture of fused sodium chloride and calcium carbonate into which is placed a second annular pot containing the wire coils packed in sand or similar inert material. The whole is fired in a cylindrical furnace provided with several gas burners so as to provide an even heat throughout.

A. R. POWELL.

Preservation of metallic surfaces. J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,734,949 and 1,734,950, 5.11.29. Appl., [A] 8.3.28, [B] 18.4.28).—Inhibitors for baths for pickling iron and steel comprise (A) an unsymmetrically substituted thiocarbamide, e.g., phenyl-*p*-tolylthiocarbamide, or (B) the reaction product of an aldehyde-ammonia and a substituted thiocarbamide. A. R. POWELL.

Inhibitor [for steel-pickling baths]. L. J. CHRISTMANN, Assr. to AMER. CYANAMID CO. (U.S.P. 1,734,560—1, 15.11.29. Appl., 18.9.29).—To prevent dissolution of the iron or steel during pickling a small quantity of (A) thiobenzamide or (B) a condensation product of o-toluidine and sulphur chloride is added to the bath.

A. R. POWELL.

Selectively controlling metal-pickling baths. J. H. GRAVELL, Assr. to AMER. CHEM. PAINT CO. (U.S.P. 1,736,118, 19.11.29. Appl., 16.7.27).—A pickling bath for removing scale from iron and steel comprises 3% sulphuric acid containing 0.5% of thiourea and 0.1% of gelatin.

A. R. POWELL.

Alloying molybdenum with iron and steel. A. KISSOCK (U.S.P. 1,736,120, 19.11.29. Appl., 15.11.27).—A pig iron containing 4–50% Mo, formed by addition of calcium molybdate or of molybdenite to the blast-furnace charge, is claimed as an intermediate agent for adding molybdenum to iron or steel.

A. R. POWELL.

Nickel-chromium-iron alloy and articles made therefrom. W. ROHN (U.S.P. 1,736,053, 19.11.29. Appl., 16.9.27. Ger., 23.11.25).—A rustless alloy for surgical instruments comprises 80–50% Ni, 10–40% Cr, 1–40% Fe, 0.2–12% Mo, and less than 0.05% C. In addition part of the above constituents may be replaced by cobalt, tungsten, and/or aluminium, so that the final alloys contain 0.2–15% W, 2–20% Co, and 0.5–6% Al.

A. R. POWELL.

Leading-in wire for glass vessels. E. FRIEDERICH, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,734,900, 5.11.29. Appl., 29.10.24. Ger., 10.2.16).—A metal wire having a coefficient of expansion nearly the same as that of glass comprises an alloy of 80% Fe, 10% Co, and 10% Mo; part of the iron may be replaced by manganese and all or part of the molybdenum by tungsten.

A. R. POWELL.

Continuous copper-melting furnace. H. S. LUKENS and R. P. HEUER (U.S.P. 1,733,419, 29.10.29. Appl., 19.12.25. Renewed 16.8.29).—The furnace comprises a well-shaped hearth provided with an inclined flue through which the copper to be melted is fed into the hearth and in which it is preheated by the hot gases without being appreciably contaminated with sulphur. The hearth contains molten copper under a layer of slag, above which an oxidising atmosphere is maintained. As the slabs to be melted reach the end of the flue they fall through the slag into the hot metal below and melt immediately without contamination. The lower layer is tapped occasionally into an electric refining furnace where it is treated with a slag adapted to dissolve its oxide content.

A. R. POWELL.

Ore-flotation apparatus. H. D. HUNT (U.S.P. 1,732,893, 22.10.29. Appl., 6.8.27).—The apparatus comprises a V-shaped cell with wide, central tubes closed at the upper end and passing nearly to the bottom. Air is passed downwards into the cell through narrower tubes centrally disposed in the wider tubes and extending slightly below them so that the uprising streams of air bubbles suck the oiled pulp up through the central tubes into very short, downwardly and outwardly directed tubes from which the mixture of

pulp and air bubbles falls back into the main body of pulp outside the central tube, thus ensuring efficient aeration. Protective screens prevent the froth which forms from coming into contact with the cascading aerated streams.

A. R. POWELL.

Flotation process [for copper-zinc ores]. S. P. WARREN (U.S.P. 1,735,190, 12.11.29. Appl., 19.4.28).—In the flotation treatment, when pine oil and xanthate are used in an alkaline circuit, the addition of small quantities of copper ammonium salts inhibits flotation of the blende but accelerates flotation of the copper and precious-metal minerals.

A. R. POWELL.

Treating [sulphide] ores. Chlorinating [sulphide] ores. T. A. MITCHELL, Assr. to L. M. HUGHES (U.S.P. 1,736,659—1,736,660, 19.11.29. Appl., 11.10.26).—(A) Pyritic ores containing copper and zinc are suspended in a solution of calcium and manganese chlorides and chlorine is passed through the mixture until the sulphides are dissolved with liberation of sulphur, the manganese chloride acting as a carrier for the chlorine. After filtration the solution is treated with zinc carbonate to precipitate iron, then with zinc carbonate and chlorine to recover the manganese as dioxide. The filtrate from the latter process is treated with powdered limestone, which regenerates zinc carbonate and calcium chloride for use again. The chlorine is obtained by electrolysis of part of the recovered calcium chloride solution. (B) Lead sulphide ores are treated with chlorine in a solution of ferric and manganese chlorides, the filtered liquor is treated with litharge and chlorine to remove manganese and iron, and the lead chloride recovered by dilution and cooling after decomposition of the tetrachloride present.

A. R. POWELL.

Treatment of ores containing zinc carbonate. P. MOUTONNIER (F.P. 628,810 and 628,811, 31.1.27).—The finely-divided ore is leached with feebly acid solutions of alkali chlorides or hypochlorites or with an acid solution containing free chlorine, and the purified solution is electrolysed for the recovery of zinc and regeneration of the leaching solution.

A. R. POWELL.

[Nickel-copper-silicon] alloy and its heat treatment. M. G. CORSON, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,732,327, 22.10.29. Appl., 19.11.26).—An alloy of 40–80% Ni, 1.5–10% Si, and remainder copper is claimed. The Si: Ni ratio should be 1:7–28. The preferred composition is 70% Ni, 26.5% Cu, and 3.5% Si. After quenching from 900° the alloy age-hardens at 500–700°.

A. R. POWELL.

Soldering of chain and ring-mesh fabrics. E. THOMA, Assr. to E. G. BEK (U.S.P. 1,732,317, 22.10.29. Appl., 4.9.24. Ger., 10.9.23).—The chain or mesh is plated with successive layers of the constituents of the desired solder, e.g., with copper, silver, and zinc or cadmium, then heated slowly to allow these layers to diffuse into one another and finally to melt the solder into the joints.

A. R. POWELL.

Metal alloy [silver-copper] for electrical contacts. H. M. FREDRIKSEN (U.S.P. 1,732,839, 22.10.29. Appl., 31.5.27).—The alloy comprises 80% Ag and 20% Cu, the copper being used in the form of the nodules which

form on the cathode during the electrolytic refining of copper. A. R. POWELL.

Purification of aluminium and its alloys. ELEKTRIZITÄTSWERK LONZA (F.P. 628,495, 5.2.27. Ger., 8.2.26).—Aluminium containing calcium, carbides, or the like is treated with a slag containing alumina, which reacts with the impurities. A. R. POWELL.

Solder for aluminium and its alloys. E. D. DEHAESE (F.P. 628,802, 24.4.26).—An alloy of 45% Zn, 45% Sn, 3.5% Cu, 3.5% Sb, and 3% Pb is claimed. A mixture of this alloy with 5% of sodium chloride and 3% of stearin is rubbed on to the cleaned parts which have been preheated to 250°. A. R. POWELL.

[Aluminium alloy] piston and its manufacture. G. D. WELTY, ASSR. to ALUMINUM CO. OF AMERICA (U.S.P. 1,732,573, 22.10.29. Appl., 2.8.24).—An alloy of aluminium with 10% Cu, 0.25% Mg, and 1.25% Fe is cast into shape and annealed, without cold-working or quenching, at 150–315° for 2–60 hrs. A. R. POWELL.

Cyanide process of treating ores containing precious metals. P. C. SCHRAPS (U.S.P. 1,734,306, 5.11.29. Appl., 16.1.25).—The ore pulp is subjected to diminished pressure to remove dissolved air, cyanide is added, and the mixture either aerated with oxygen or treated with a compound which evolves oxygen readily in alkaline solution. The method is especially suitable for ores containing silver sulphide. A. R. POWELL.

Electroplating with chromium on other metals, especially iron. A. E. HARDY (F.P. 629,423, 30.4.26).—The metal to be plated is first coated with a thin deposit of tin, cadmium, or lead, or an alloy of these metals, and then plated as usual with chromium. A more efficient protection against rusting is thus obtained. A. R. POWELL.

Production of agglomerates of ores, mineral, etc. P. JUNG (U.S.P. 1,739,839, 17.12.29. Appl., 5.7.23).—See B.P. 200,524; B., 1924, 985.

Machine for mixing two or more substances with each other. H. ERIKSSON (U.S.P. 1,740,166, 17.12.29. Appl., 9.11.27. Swed., 16.6.27).—See B.P. 287,016; B., 1928, 372.

Extraction of volatilisable metals from ores and metallurgical products. II. PAPE, ASSR. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,736,665, 19.11.29. Appl., 19.4.27. Ger., 26.4.26).—See B.P. 252,679, B., 1927, 583.

Magnetic [alloy] material and appliance. G. W. ELMEN, ASSR. to BELL TELEPHONE LABS., INC. (U.S.P. 1,739,752, 17.12.29. Appl., 28.12.27. U.K., 29.8.27).—See B.P. 297,938; B., 1928, 899.

Manufacture of tools of tungsten. O. DIENER (U.S.P. 1,740,009, 17.12.29. Appl., 20.3.28. Ger., 19.1.28).—See B.P. 294,086; B., 1928, 716.

Working up mixed shavings of white metal and red metal. A. SCHWARZ (U.S.P. 1,739,992, 17.12.29. Appl., 10.6.27. Austr., 5.5.26).—See B.P. 270,740; B., 1928, 610.

Forming an electrically insulating and anti-corrosive oxide coating on aluminium material.

S. SETOH and S. UEKI, ASSRS. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,735,509, 12.11.29. Appl., 22.10.26. Jap., 30.10.25).—See B.P. 226,536; B., 1925, 725.

Rotary drum machines [for foundry sand] (B.P. 322,405). **[Alloy steel for] pressure vessel** (B.P. 322,702).—See I. **Heating furnace** (U.S.P. 1,735,866). **Separation of ores** (B.P. 305,102). **Magnetic cores** (U.S.P. 1,733, 592). **Cathodes for electron-discharge devices** (B.P. 316,536). **Composite metallic body** (U.S.P. 1,733,813).—See XI.

XI.—ELECTROTECHNICS.

Cone-fusion [electric] muffle furnace for high-temperature work. H. N. BAUMANN, JUN. (J. Amer. Ceram. Soc., 1929, 12, 740–741).—A laboratory muffle furnace for temperatures up to at least 1600° consists of a small muffle made of "refrax" bricks (recrystallised silicon carbide), heated by two slotted graphite electric resistors. Powdered petroleum coke is filled into the furnace to protect the resistors from oxidation and to act as a heat insulator. A variable-voltage transformer is a necessary part of the equipment. F. SALT.

Alternating-current electrolysis of water. J. W. SHIPLEY (Canad. J. Res., 1929, 1, 305–358; cf. A., 1928, 245, 1337).—Electrolysis of water takes place in many types of commercial hot-water heaters and steam generators operated with alternating current. As the hydrogen-oxygen mixture thus produced is highly diluted with steam at the point of generation there is little danger of explosion until most of the steam has condensed; in electric steam generators feeding steam accumulators a separate chamber should therefore be provided so that condensation takes place before the steam enters the accumulator. Current density is the primary factor in the alternating-current electrolysis of water at low voltages, and there is a critical current density for metal electrodes below which no gas is evolved, but above which all the current is utilised in the decomposition of the water according to Faraday's law for alternating current. This critical density varies with the temperature of the water, with the metal of the electrode, and with the periodicity of the current. For silver it is 1.6, for platinum 0.7, copper 1.9, oxide-coated copper 10, steel 4.5, soft iron 3.8, nickel 4.6, and aluminium <0.01 amp./cm.² The electrolytic capacity of the electrode is constant for copper, but varies with the quarter-wave area for platinum and silver; with a constant area the capacity decreases to a minimum with increasing frequency. With high-voltage currents decomposition of water may also occur thermally by arcing effects which are quite independent of alternating-current electrolysis, the rate of decomposition depending on the rate of consumption of electrical energy. Arcing is prevented by increasing the hydrostatic pressure in the neighbourhood of the electrodes; the arcing current decreases hyperbolically with increasing voltage when the area of the electrodes is kept constant, and increases with the diameter of the electrodes when the voltage is kept constant. The practical application of the results of the investigation to commercial boilers and heaters is discussed and recommendations are made for their safe operation. A. R. POWELL.

Determination of chlorides in zinc dross. JOAS-SART and LECLERC. **Chromium plating.** ALTER and MATHERS.—See X. **Determination of moisture.** LAMPE.—See XIX.

See also A., Jan., 44, **Preparation of hydroxylamine** (STSCHERBAKOV and LIBINA). 45, **Electrolysis of metallic oxides** (ANDRIEUX). **Electrolytic oxidation of ether** (MARIE and LEJEUNE). **Reduction of arylarsinic acid** (NAKADA). 53, **Separation of lead and bismuth** (COLLIN). 56, **Photo-cells and their application** (MARTON and ROSTAS). 87, **Reduction of aromatic acids** (SOMLO).

PATENTS.

Electric furnace. F. W. BROOKE, ASSR. to W. SWINDELL & BROS. (U.S.P. 1,735,419, 12.11.29. Appl., 2.6.28).—The furnace, which has a hearth of greater length than breadth, is provided with two sets of movable electrodes supported on one side and arranged along its greater dimension, each set taking current from opposite ends of the furnace, which is capable of a tilting movement about its major axis.

F. R. ENNOS.

Non-oxidising [electric] heating furnace. G. KELLER, ASSR. to A.-G. BROWN, BOVERI & Co. (U.S.P. 1,735,866, 19.11.29. Appl., 13.5.26. Ger., 30.5.25).—The furnace comprises a cylindrical heating chamber heated internally by a wire resistance and provided with a tightly fitting lid sitting in a liquid seal. An outlet pipe from the lid dips into a sump at the lower end of a brickwork pit in which the furnace is built. The articles to be annealed are placed in the heating chamber, which is then closed and filled with carbon dioxide. During the heating operation the gas is partly expelled into the sump and on cooling is sucked back into the chamber, thus preventing oxidation of its contents.

A. R. POWELL.

[Thermal fuse for] electric furnaces. L. W. WILD, and WILD-BARFIELD ELECTRIC FURNACES, LTD. (B.P. 322,975, 20.3.29).—A two-pole switch controlling the fuse circuit is operated by opening and closing the furnace door, both poles of the fuse being "dead" when the door is opened.

J. S. G. THOMAS.

Gas-treating devices for enclosed electrical apparatus. A. G. ELLIS, H. DIGGLE, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 321,513, 27.9.28).—Water taken up from air or other gas admitted to the casing of the electrical apparatus is deflected by spouts, baffles, etc. into a collecting tray or funnel communicating with a U-tube forming a seal separate and distinct from the main "breather" trap or seal, so that the water is continuously drained off without coming into contact with the liquid in the "breather" seal.

J. S. G. THOMAS.

Deoxidising apparatus for use with oil-immersed electrical apparatus. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., ASSEES. of C. A. STYER (B.P. 309,138, 3.4.29. U.S., 5.4.28).—In apparatus in which an inert atmosphere is maintained in the gas space of a tank containing an electrical, oil-immersed apparatus, a coherent block of solid deoxidising material is held in operative relation with a heating element during consumption

of the block. Such material may be prepared by adding a binder, *e.g.*, partly carbonised sugar, and about 1% of cobalt chloride, manganese chloride, sodium chloride, sodium sulphate, or potassium iodide to granular charcoal from which ash-forming constituents and hydrogen have been removed.

J. S. G. THOMAS.

Electromagnetic separation of complex ores. BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of J. A. SEEDE (B.P. 305,102, 30.1.29 U.S., 30.1.28).—Crushed gangue and ore flows through a magnetic field generated by coreless coils energised by alternating current the frequency of which is considerably beyond that of commercial supplies.

J. S. G. THOMAS.

Manufacture of magnetic cores. F. J. GIVEN, ASSR. to BELL TELEPHONE LABS., INC. (U.S.P. 1,733,592, 29.10.29. Appl., 6.2.29).—In making dust cores of permalloy, dust of an alloy containing more nickel than is desired is mixed with the dust of an alloy containing too little nickel in such proportions that the mass has the desired composition. The mixed dust is then annealed, the mass again pulverised, and the dust formed into cores as usual.

A. R. POWELL.

X-Ray tubes. BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of W. D. COOLIDGE (B.P. 298,987, 17.10.28. U.S., 18.10.27).—X-Ray tubes fitted with a metal window which is protected against bombardment by reflected electrons are claimed. [Stat. ref.]

J. S. G. THOMAS.

Vacuum tube. K. OKABE (U.S.P. 1,735,294, 12.11.29. Appl., 11.5.26. Jap., 5.3.26).—The tube contains auxiliary aluminium anodes coated with nickel and tungsten oxides.

J. S. G. THOMAS.

Electric-discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 321,438, 9.7.28).—In a rectifying discharge tube filled with a gas or vapour, *e.g.*, argon, neon, or helium, and a small proportion of another gas or vapour, *e.g.*, 0.025% of mercury vapour, having an ionisation voltage lower than the excitation voltage of a metastable state of the principal gas, the electric field near the anode is stronger than that near the cathode.

J. S. G. THOMAS.

Cathodes for electron-discharge devices. A. JUST (B.P. 316,536, 26.11.28. Austr., 30.7.28).—Molten iron is alloyed with one or more alkali, alkaline-earth, or rare-earth metals, *e.g.*, with potassium or lanthanum; the alloy is mechanically worked with iron and annealed in an inert atmosphere to make it more ductile, and then finally drawn into wire.

J. S. G. THOMAS.

[Heating of electrodes of electric] discharge tubes. ELECTRO MATÉRIEL, P. HINSTIN, and A. LEHMANN (F.P. 629,551, 6.5.26).—Electrodes are heated by radioactive material contained in a capsule.

J. S. G. THOMAS.

Production of composite [metallic] body. [Filament for radio valves.] J. W. MARDEN and H. C. RENTSCHLER, ASSRS. to WESTINGHOUSE LAMP Co. (U.S.P. 1,733,813, 29.10.29. Appl., 1.8.21).—The filament comprises a molybdenum wire coated with a film containing finely-divided thorium, zirconium, uranium, titanium, or vanadium as electron-emitting material, and pow-

dered aluminium, magnesium, chromium, or a rare-earth metal as the getter. A. R. POWELL.

Accumulator. L. BAROSSO (F.P. 631,126, 18.3.27. Ital., 19.3. and 15.5.26).—The negative plates, made of material which is not attacked by caustic alkali solution and mercury, are coated with zinc amalgam; the positive plates are made of Ag_4O_3 , CuO , $\text{Ni}(\text{OH})_2$, etc. and an alkali solution is used as electrolyte. Reactions which occur during charging and discharging are: $3\text{ZnHg}_x + 6\text{NaOH} + \text{Ag}_4\text{O}_3 \rightleftharpoons 3x\text{Hg} + 3\text{Zn}(\text{ONa})_2 + 4\text{Ag} + 3\text{H}_2\text{O}$. The operative voltage of the accumulator is 1.83–1.53 volts. J. S. G. THOMAS.

Accumulator. A. PETIT (F.P. 629,921, 28.2.27).—Negative electrodes formed of metals, *e.g.*, iron, nickel, or more especially zinc, which form soluble chlorides (and, if desired, sulphates) and do not decompose water, and insoluble positive electrodes composed of, *e.g.*, lead chloride, lead sulphate, cuprous chloride, mercurous chloride, or silver chloride, are immersed in water. Thus the accumulator may be prepared by passing a current between a copper electrode and a copper-lined iron vessel containing a solution of zinc chloride.

J. S. G. THOMAS.

Manufacture of electric batteries. A. HELBRONNER and E. DURT (B.P. 295,589, 13.8.28. Fr., 11.8.27).—An insoluble ferricyanide, *e.g.*, of zinc, copper, or silver, is used as depolariser in a cell having an electrode of zinc or other metal occupying a place above carbon, platinum, copper, etc. in the electrochemical series, and having zinc chloride or a double cyanide of zinc and an alkali metal, caustic soda, calcium chloride, etc. as electrolyte.

J. S. G. THOMAS.

Secondary electric cells. L. A. LEVY, and ALMEIDA ACCUMULATORS, LTD. (B.P. 320,916 and 321,405, [A] 27.4.28, [B] 3.5.28).—(A) Cells comprising an acid electrolyte containing salts of two or more different halogens other than fluorine, a cathode at which zinc is deposited during charging, and a conducting anode, *e.g.*, of graphite, substantially unattacked by the electrolyte, embedded in carbonaceous (graphite) granules contained in a low-resistance porous envelope capable of withstanding the bursting stresses set up in working are claimed. The apparent volume of the granules is large (*e.g.*, 10:1) compared with that of the anode. (B) In a cell comprising a cathode, *e.g.*, of Ironac or Tantiron, preferably roughened and coated with mercury, in an electrolyte (zinc chloride and zinc bromide), during charging a halogen (or halogens) is (are) evolved at the anode, and zinc which is deposited at the cathode is completely removed during discharge, the anode being associated with excess of halogens.

J. S. G. THOMAS.

Graphite and depolariser plates for galvanic cells. SIEMENS & HALSKE A.-G., Asses. of K. SCHENKEL (B.P. 299,784, 30.10.28. Ger., 31.10.27).—A layer of perforated material, *e.g.*, linen or paper acting as carrier for electrolyte, is pressed upon a layer of depolarising material placed upon a layer of carbon.

J. S. G. THOMAS.

Production of printing plates by electrochemical processes. C. SCHINZEL (B.P. 291,063, 25.5.28. Czechoslov., 25.5.27).—Soluble layers of gelatin or similar

colloids are partially tanned either by the direct action of an electric current (5–100 volts) on substances incorporated therein, or indirectly by reaction with substances produced by electric or electrolytic action on other incorporated substances, *e.g.*, sodium or calcium chloride, sodium nitrate, chlorates, vanadates, etc. Alternatively, the process may be applied to rendering tanned layers of gelatin etc. soluble. J. S. G. THOMAS.

Electric-discharge tubes. C. J. R. H. VON WEDEL (B.P. 307,424, 5.3.29. U.S., 7.3.28).

Heated filament for thermionic valves. S. LOEWE (B.P. 295,637, 13.8.28. Ger., 15.8.27).

Anode battery [with interchangeable dry batteries]. M. SCHMIDT (M. SCHMIDT ELEKTROTECHNISCHE & METALLWARENFABR.) (B.P. 322,852, 26.10.28).

Thermal treatment of pulverulent material (B.P. 305,231). **Rotary drum machines** (B.P. 322,405).—See I. **Conductivity of charcoal** (G.P. 454,855).—See II. **Mixtures of nitrogen and hydrogen** (B.P. 322,701).—See VII. **Zinc ores** (F.P. 628,810–11). **Alloy for electrical contacts** (U.S.P. 1,732,839). **Chromium plating** (F.P. 629,423).—See X.

XII.—FATS; OILS; WAXES.

Solidification curves of cacao butter. J. D. VAN ROON (Chem. Weekblad, 1929, 26, 619–620; cf. Straub and Malotaux, B., 1930, 109).—The difference in form of the two types of cooling curve obtained for cacao butter is ascribed to supercooling; there is no reason to suppose that the same glyceride will begin to separate first in every case, but even if that were so, different heat effects may still be expected in different cases. A note by STRAUB is appended. S. I. LEVY.

Manufacture of ghee substitutes. W. NORMANN (Chem. Umschau, 1929, 36, 337–339).—The purification, colouring, etc. of ghee substitutes are discussed. The particular granular consistency desired is obtained by regulated cooling of the fat mixture; it is only necessary, however, to control the cooling rate until the minimum on the cooling curve (of usual type) is reached, although the fat at this point is still liquid, the speed of cooling after this point being immaterial. Crystal aggregates may be isolated for examination by spreading a thin layer of fat on a porous plate and keeping it for a day over light petroleum. E. LEWKOWITSCH.

Chemical engineering in a modern stearic acid plant. T. R. OLIVE (Chem. and Met. Eng., 1929, 36, 720–723).—Pressed fats and tallows, after blending, are cleaned by means of an acid wash and hydrolysed with Twitchell's reagent in dilute sulphuric acid, a mixture almost universally adopted. Boiling for 5 hrs. produces 85–90% hydrolysis, at which stage the reagent is renewed and hydrolysis attains 95–97% during a further period of 10 hrs. Dehydrated stearic acid for distillation is run into cast-iron stills of two-piece construction, which are usually coal-fired, and under the action of superheated steam the pure acids volatilise and are condensed in cooled copper drums, the whole process being maintained under vacuum. The acid is cooled in a cascade of aluminium or granite pans and the solidified cakes, each separately wrapped, are

subjected to a pressure of 3000 lb./in.² which forces out a large part of the oleic acid. A further process of hot-pressing and boiling with dilute sulphuric acid yields the highest quality of stearic acid. Glycerin is recovered from the liquors by known methods.

C. A. KING.

Drying oils. XI. Unimolecular films of blown and heat-bodied oils and their constituent fatty acids and esters on water surfaces. J. S. LONG, W. W. KITTELBERGER, L. K. SCOTT, and W. S. EGGE (Ind. Eng. Chem., 1929, 21, 950—955; cf. B., 1929, 442).—The characteristics of such films when floated on water surfaces and subjected to compression in a modified Langmuir surface-tension balance were observed. The results are tabulated, graphed, and fully discussed. Oxidised oils occupy a greater area per mol. on a water surface than do heat-bodied oils of the same mol. wt. They also require a larger compressive force to crumple the unimolecular film. A method for the direct synthesis of glycerides etc. of saturated and unsaturated acids is described in which a stream of inert gas, converted by means of a rapidly rotated whizzer into a very large number of minute bubbles, is introduced into a mixture of the theoretical amounts of acid and alcohol maintained at 100—225°, the water of reaction being effectively removed. S. S. WOOLF.

Oxidation processes of drying oils. III. S. MERZBACHER (Chem. Umschau, 1929, 36, 339—342; cf. B., 1928, 678).—Oxidised boiled linseed oil ("Hobum") films were saponified in the cold and then extracted successively with cold water and with light petroleum. In the first case the products were azelaic acid, water-soluble oxyn-acids II (cf. Eibner and others, B., 1925, 679, 930), glycerin, small amounts of formic, propionic, and hexoic acids, and an undefined residue (total 25%), and in the second an extract (20%) containing higher saturated acids from the original oil, unchanged unsaturated acids, together with pelargonic, azelaic, and hexoic acids. The residue on being boiled with water gave a further supply of azelaic acid and left 26.3% of resinous oxyn-acids I. The large proportion of azelaic acid among the products is notable, but only a small proportion can have existed in the free state in the linoxyn before saponification. Octoic acid was again absent (cf. Part II, *loc. cit.*). Probably considerable quantities of the lower volatile fatty acids had been lost during the drying of the oil. E. LEWKOWITSCH.

Vegetable oils of the Union of S.S.R. IV. Nature of oils of Palmæ in connexion with the climate of district of origin. S. IVANOV and Z. P. ALISSOVA (Chem. Umschau, 1929, 36, 401—403; cf. B., 1929, 987).—A liquid oil (64.08%) was obtained from the kernels of *Cocos Bonetti* grown in Suchum (northern limit of distribution of the coco-palm) which had acid value 0.7, saponif. value 259.65, iodine value 23.6, thiocyanogen value 19.78; it consisted therefore of the following glycerides: linoleic 4.41%, oleic 18.54%, saturated 77.9%. These figures illustrate the theory that the proportion of unsaturated acids in a vegetable oil and its degree of unsaturation increase with the severity of the climate of origin. Hydrogenation of

the Suchum oil yields a satisfactory substitute for coconut oil. E. LEWKOWITSCH.

Oil content of sunflower seeds. N. USPENSKI (Masloboino-Zhir. Delo, 1928, No. 6, 31—40).—The oil content decreases with the increase in nitrogen content of the soil above the limit required for the maximal growth of leaves. The humidity must not be below a similar limit. CHEMICAL ABSTRACTS.

Relation of bulk density, size, and colour of mustard seeds to their oil content. V. MILASHEVSKI (Masloboino-Zhir. Delo, 1928, No. 9, 10—12).—The bulk density of mustard seeds is not a measure of their oil content. Large seeds yield oil and cake of higher quality than that from small seeds. CHEMICAL ABSTRACTS.

Kepayang oils. C. D. V. GEORGI and G. L. TEIK (Malayan Agric. J., 1929, 17, 392—397).—The seeds of Pokok kepayang (*Pangium edule*, Reinw.; N. O. *Flacourtiaceae*) contained 8.7% of kernels which yielded 44.5% of moisture and 24.8% of a yellow oil of pleasant odour. Although the plant is closely related to the genus *Hydnocarpus*, the oil is optically inactive. The (extracted) oil had: d_{15}^{20} 0.9132, n_D^{20} 1.4660, saponif. value 196.5, iodine value (Wijs) 108.3, acid value 0.2, unsaponifiable matter 0.6%; the fatty acids had: titer value 20.4°, mean mol. wt. 280.3, iodine value (Wijs) 111.1. The kernels contained a tannin and a cyanogenetic glucoside. Akar kepayang (or Akar klaypayang; cf. Gimlette, "Malay Poisons and Cures," p. 140) (*Hodgsonia capmocarpa*, Ridley; N. O. *Cucurbitaceae*) seeds (20% of fresh fruit) yielded 9.6% of kernels containing a cyanogenetic glucoside and a light yellow non-drying oil of nutty odour. A small quantity of stearine was deposited after keeping a few days. The (pressed) oil had (cf. *ibid.*, 1913—14, 2, 67; 1923, 11, 39): d_{15}^{20} 0.907, n_D^{20} 1.4613, saponif. value 201.2, iodine value 67.1, acid value 3.6, unsaponifiable matter 0.4%; and the fatty acids: titer value 42.1°, mean mol. wt. 272.6, iodine value 68.2. The oil is entirely different from that obtained from *P. edule*. E. LEWKOWITSCH.

Allanblackia Stuhlmannii seeds from Tanganyika territory. ANON. (Bull. Imp. Inst., 1929, 27, 455—457).—The kernels of the seeds (av. wt. 10.6 g., 22.6% shell) of *Allanblackia Stuhlmannii* gave, on extraction, 66.5% of a white, almost odourless fat having: d_{15}^{100} 0.8549, n_D^{40} 1.457, m.p. 40.0°, solidif. pt. of fatty acids 60.0°, acid value 9.9, saponif. value 189.6, iodine value (Wijs) 39.6%, unsaponifiable matter 0.76%. The residual meal had: moisture 13.9%, crude proteins 14.9%, fat 1.4%, carbohydrates (by diff.) 58.6%, crude fibre 7.7%, ash 3.5%. It contained no alkaloids or cyanogenetic glucosides, but a small amount of tannin was present which imparted an astringent taste. Its nutrient ratio was 1:4.2 and it had food units 99.4. E. H. SHARPLES.

Catalyst for hydrogenating oils. A. MOSHKIN (Masloboino-Zhir. Delo, 1928, No. 10, 16—17).—Regenerated nickel catalyst must be cooled in hydrogen or carbon dioxide. Sulphur is less poisonous than arsenic. CHEMICAL ABSTRACTS.

Influence of air on activity of catalysts used in hydrogenating oils. A. BAG (Masloboino-Zhir. Delo,

1928, No. 5, 6).—Reactivated nickel does not lose its activity when kept in air for 1 month, provided it is treated with carbon dioxide directly after exposure to hydrogen.

CHEMICAL ABSTRACTS.

Hydrogenation of linseed oil. V. VASILEV (Masloboino-Zhir. Delo, 1928, No. 7, 9—11).—Owing to the reduction of hydroxyl groups formed during the oxidation process, slightly deteriorated linseed oil absorbs more hydrogen than can be accounted for by the decrease in the iodine value.

CHEMICAL ABSTRACTS.

Oil analysis. S. KETTLE (Chemist-Analyst, 1929, 18, No. 5, 7).—For the determination of saponif. value, the oil (5 g.) is dissolved in benzyl alcohol (15 c.c.), the free acid being titrated using phenolphthalein as indicator; benzyl-alcoholic potassium hydroxide is added, the mixture boiled under reflux for 5 min., and the excess titrated. Bromine values are determined in carbon tetrachloride solution using potassium bromide, a little potassium iodide, and a known quantity of potassium bromate, shaking, and titrating the excess of bromine with sodium hydrogen sulphite solution. For the chlorine value the sample is treated with a saturated solution of phenyl iodochloride in carbon tetrachloride, the excess of chlorine being titrated with silver nitrate. For the determination of acid values a mixture of benzene (90%) and alcohol (10%) is preferred to alcohol. Brief heating with concentrated nitric acid is employed as a colour test for mineral oils.

CHEMICAL ABSTRACTS.

Determination of iodine value by Margosches' rapid method. S. YUSHKEVICH (Masloboino-Zhir. Delo, 1928, No. 9, 22—28; No. 10, 31—35).—The results agree with those obtained by the methods of Hübl and Hanus. The excess of iodine must reach 65%.

CHEMICAL ABSTRACTS.

Distillation of nut shells. GEORGI and BUCKLEY.—See II.

See also A., Jan., 92, **Saponin from seed kernels of *Achras sapota*, L.** (VAN DER HAAR).

PATENTS.

Recovery of fats and oils. J. A. S. VAN DEURS (B.P. 303,413, 11.12.28. Denm., 2.1.28).—The p_H of the raw material is altered, by the addition of acid (and water as required) or of acid-forming micro-organisms, until it is below 5, whereby the fatty matter is caused to coalesce as a continuous phase and separates easily as the material is shaken, stirred, or treated by centrifugal action. The material may previously be ground or warmed to liquefy the fat.

E. LEWKOWITSCH.

Production of [pale, fatty] organic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,399, 6.7.28).—Crude fatty acids, obtained by the oxidation of solid paraffin hydrocarbons, wax, etc., are submitted to a pressing or sweating treatment at slightly elevated temperatures, which, however, are below the m.p. of the pure acids (e.g., at 12—13°). The solid, pale residue (m.p. 45—48°) will furnish pale soaps.

E. LEWKOWITSCH.

Manufacture of unsaturated fatty acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,437,

30.8.28).—The crude oxidation products of paraffin wax are distilled, preferably under reduced pressure, and/or in the presence of an inert gas, e.g., carbon dioxide, steam, whereby the hydroxycarboxylic acids are converted into long-chain unsaturated acids.

E. LEWKOWITSCH.

Production of sulphoaromatic fatty acids. G. PETROV (G.P. 456,353, 19.5.25).—Crude oleic acid or sunflower seed oil and naphthalene or benzene are treated as in B.P. 252,212 (B., 1927, 348). The sulphoaromatic fatty acids in the form of their neutral alkali salts are suitable as washing, wetting-out, or fat-splitting agents, and can be used for the esterification of acids and alcohols, acetylation of cellulose, condensation of phenols and aldehydes, and in the leather industry.

E. LEWKOWITSCH.

Saponifying free fatty acids contained in vegetable and animal oils and fats with alkali solutions *in vacuo*. W. GENSECKE (B.P. 321,080, 26.11.28).—The free fatty acids are saponified by alkaline solutions at approximately uniform low temperatures in a vacuum, the intensity of which is constantly increased (by means of a steam injector interposed between the neutralisation vessel and the steam condenser), until the soap produced is converted by dehydration into a state in which it may be separated from the fat by filtration.

E. LEWKOWITSCH.

Obtaining fatty acids wholly or almost wholly free from unsaponifiable matter. OEL- U. FETT-CHEMIE GES.M.B.H. (B.P. 296,079, 29.6.28. Ger., 26.8.27).—Such fatty acids (up to 99.6% pure) are obtained by distilling split (waste) fatty acids (e.g., bone, cotton oil soap-stock fatty acids), with the exception of tall oil, in a high vacuum with or without fractionating attachments.

E. LEWKOWITSCH.

Soap. H. E. CROCCO (U.S.P. 1,733,150, 29.10.29. Appl., 8.6.27).—The detergent comprises a base of 2—10 pts. of water-soluble soap with 1 pt. each of a non-volatile liquid, detergent hydrocarbon, a fatty sulphonated compound, and sodium sulphate.

E. LEWKOWITSCH.

Manufacture of detergents. RÖHM & HAAS A.-G. (F.P. 630,409, 7.3.27. Ger., 12.3.26).—Stable detergents containing pancreatic enzymes are prepared from comparatively dry materials, so that the products contain not more than 10% of water, by compressing into blocks mixtures of dry soap and dried pancreas; softening agents (sodium oxalate), salts of bile acids, and alkali salts may also be added.

L. A. COLES.

Cooking and agitating apparatus for extracting oil from palm fruit. DUCHSCHER & CIE. SOC. EN COMM. (B.P. 303,497, 27.10.28. Ger., 6.1.28).—The cooker is fitted with a hollow double bottom, with openings for the admission of steam and the withdrawal of condensed liquors, and with a rotating agitator; a secondary agitating unit is provided which may be brought into action if required.

E. LEWKOWITSCH.

Treatment of mustard oil-containing seeds prior to extraction of the oil by pressure. L. MELLERSH-JACKSON. From F. KRUPP GRUSONWERK

A.-G. (B.P. 322,681, 21.1.29).—The cleaned and ground material is moistened with a small quantity (2½%) of water, or water acidified with, *e.g.*, sulphuric (1/1000), hydrochloric, or citric acid, and thoroughly mixed for 20–45 min. until uniformly moist before pressing. A highly pungent oil is thus obtained. E. LEWKOWITSCH.

Manufacture of lanolin-like compositions. R. HAUSCHKA, Assr. to G. RICARDO (U.S.P. 1,728,205, 17.9.29. Appl., 24.9.25).—A natural ester, *e.g.*, castor oil, beeswax, or pine rosin, is heated with an aldehyde or ketone, *e.g.*, formaldehyde, acetone, and the resulting oil separated and cooled. A metal or a metallic oxide, *e.g.*, iron, or nickel oxide, may be added as a catalyst, and an oleaginous hydrocarbon as a diluent.

F. G. CLARKE.

Imparting (A) a black or (B) a stone colour to leather polish. S. H. DENNINGTON (B.P. 322,969 and 322,970, 7.3.29).—The usual mixture of oils and fats used in the polish is coloured with a mixture of (A) iron calcium phosphate, powdered ivory black, and pure carbon black, or (B) lithopone, barium sulphate, iron oxide, and ivory black.

H. ROYAL-DAWSON.

Manufacture of products resembling [natural] wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,854, 16.7. and 15.10.28).—Montan wax bleached by oxidation, or a conversion product thereof, is subjected to a short treatment with oxygen, alone or in conjunction with hydrocarbons (*e.g.*, paraffin wax), fatty materials (*e.g.*, castor oil), resins, or waxes, etc., preferably in the presence of oxidising catalysts; the free carboxyl groups produced may be esterified, *e.g.*, by glycol. (Cf. B.P. 296,145, 305,552, 308,996, and 315,283; B., 1928, 779; 1929, 273, 425, 804).

E. LEWKOWITSCH.

Production of esters of saccharides of higher unsaturated fatty acids. L. ROSENTHAL and W. LENHARD, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,739,863, 17.12.29. Appl., 28.10.24. Ger., 10.11.23).—See G.P. 411,900; B., 1925, 642.

Purifying oils (U.S.P. 1,732,371).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of degree of "chalking" of paints. R. KEMPF (Farben-Ztg., 1929, 35, 650–652).—The significance of "chalking" as a stage of failure of paints is discussed. The usual method of observing chalking, *i.e.*, rubbing the finger tip over the surface, is shown to be subject to various inaccuracies. The method of applying damp, black paper to white paints, using a definite pressure in a suitable apparatus (details of which are given), was developed. After trials of various black materials, *e.g.*, filter paper, various textiles, developed photographic paper, the last-named was found the most satisfactory. Chalking is rated by the amount of whitening of the paper, and five arbitrary grades are defined and illustrated by photographs, as are the results of typical tests according to this method. For paints other than white, the damp paper applied must be of a suitably contrasting colour.

S. S. WOOLF.

Detection of benzene in varnishes. E. H. JACKSON (Chemist-Analyst, 1929, 18, 15).—The fraction b.p. 65–100° is washed and nitrated; the nitrobenzene is reduced with zinc and hydrochloric acid, the aniline being liberated with sodium hydroxide and detected with bleaching powder. Toluene does not interfere.

CHEMICAL ABSTRACTS.

Can shellac be substituted by cellulose lacquer for wood polishing? R. FREUND (Farben-Ztg., 1929, 35, 653–654).—Cellulose wood-finish is dearer than French polish in initial cost, and its consumption per unit area of wood is greater. Nevertheless, owing to its rapidity and ease of working, it is the more economic proposition. Both are equally subject to shrinkage, a fault that is shown to be dependent on the wood and not on the finish.

S. S. WOOLF.

Waste lithopone. STROKOV.—See VII.

See also A., Jan., 45, **Oxidation products of ether** (MARIE and LEJEUNE). 57, **Fossil resin** (CORTELEZZI). 79, **Amorphous resins and lacs from s-triphenylbenzene** (VORLÄNDER and others). 81, **Chromotrope CR lakes** (ROSENHAUER and others).

PATENTS.

Preparation of pigments and the like. J. W. C. CRAWFORD, G. E. SCHARFF, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 318,937, 9.3.28 and 9.1.29).—Water is removed from water-wet pigment pastes by adding to the paste a non-reactive and substantially non-miscible liquid, *e.g.*, benzene, toluene, and subsequently heating the mixture, the water being distilled off in azeotropic mixture with the non-reactive liquid. The latter is removed from the condensate by suitable means, *e.g.*, in an automatic separator, and returned to the wet pigment, the process being repeated. In place of benzene etc. a mixture of this with ethyl alcohol may be used, in which case the ternary condensate is allowed to settle and the upper layer (mainly benzene) is separated and returned to the still. If higher alcohols be used, a water-benzene mixture distils over first and the alcohol is recovered subsequently by distillation. The speed of dehydration may be increased by agitating the mixture and by the addition of a small amount of a suitable wetting, dispersing, or emulsifying agent. The dehydrated suspension prepared as above may be finally dried below 50° at normal or under reduced pressure, and may be reduced to a very fine state of division with moderate grinding. High-boiling solvents, *e.g.*, tricresyl phosphate, and softening agents, *e.g.*, castor oil, may be introduced originally, or at any intermediate stage.

S. S. WOOLF.

Manufacture of white lead. E. C. WALKER, Assr. to NAT. LEAD Co. (1,732,490, 22.10.29. Appl., 1.8.21).—In the American modification of the Dutch process of making white lead the lead grids are laid on the inclined floor of a spiral stack in order to relieve the overburden on the lower grids and prevent collapse of the charge when corrosion is nearly complete. A. R. POWELL.

Producing pigmentary base products for lead paint. A. V. BLOM (U.S.P. 1,736,066, 19.11.29. Appl., 5.4.27).—An alloy of 85% Pb, 13% Sb, and 2% Sn

is melted in a flat pan-shaped hearth under a layer of calcium resinate equal to 0.5% of the weight of alloy, and a slow current of air is drawn through the metal from numerous small tuyères in the hearth of the furnace by applying suction to an opening in the cover.

A. R. POWELL.

Manufacture of oil lacquers and varnishes. I. G. FARBENIND. A.-G. (B.P. 296,679, 296,991—2, [A] 3.9.28, [B, C] 7.9.28. Ger., [A] 2.9.27, [B, C] 10.9.27).—Oil varnishes (paints) with high lustre, good covering power, and good brushing properties are obtained by incorporating in the customary raw materials a small quantity (about 3%) of (A) an aliphatic carboxylic acid unsaturated in the $\alpha\beta$ -position, *e.g.*, crotonic, acrylic, and sorbic acids, or their halogen, hydroxy-, or alkyl substituted derivatives; or (B) a monobasic hydroxy-carboxylic acid of the type $R'\cdot CH(OR)\cdot CO_2H$, where R and R' denote a hydrogen atom, or alkyl, aryl, or aralkyl groups, or esters or salts thereof, *e.g.*, tolyloxy-acetic acid, glycollic acid; or (C) an amide of a carboxylic acid (or carbonic acid) or *N*-substitution products, *e.g.*, methylolbutylurethane, methylolbenzamide. It is possible to incorporate in such varnishes considerably more pigment than is allowable by the customary manner of working.

E. LEWKOWITSCH.

Manufacture of lacquers, filling or priming compositions, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,466 and 322,537—322,543, [A—H] 2.6.28).—Such coating compositions, are based on mixtures of nitrocellulose (A—D) or lower fatty acid esters of cellulose (E—H) with synthetic resin condensation products of aliphatic dibasic acids with polyhydric alcohols (A, E), aromatic dibasic acids with polyhydric alcohols containing 4—6 hydroxyl groups in their molecule (B, F), aromatic dibasic acids with glycols (C, G), aromatic dibasic acids with hydroxyalkyl ethers of polyhydric alcohols containing 3 hydroxyl groups in their molecule (D, H).

S. S. WOOLF.

Preventing the deposition of driers in linseed oil varnishes. I. G. FARBENIND. A.-G. (B.P. 299,361, 13.9.28. Ger., 24.10.27).—The linseed oil to be used for the varnish is prepared (before or after the addition of the driers) by admixing the oxides (or hydroxides, carbonates, or metals) of calcium, magnesium, zinc, or cadmium in amount at least equivalent to the free fatty acids of the oil (*e.g.*, 0.1—0.5% as a rule) and heating to about 100°; the soaps formed are removed by filtration.

E. LEWKOWITSCH.

Protective coating. J. E. THOMAS, ASSR. to CHICAGO WHITE LEAD & OIL Co. (U.S.P. 1,733,564, 29.10.29. Appl., 20.1.26).—An article coloured with a red aniline-dye stain has a protective coating, comprising a varnish vehicle and a sufficient quantity of an organic pigment, *e.g.*, paranitraniline-red, litho-red, toluidine-red, or madder-lake, to give the same colour as the stain and to prevent fading.

F. G. CLARKE.

Liquid coating composition [varnish for paper shells]. W. H. WOODFORD, ASSR. to REMINGTON ARMS Co., Inc. (U.S.P. 1,733,177, 29.10.29. Appl., 10.9.27).—Paraffin-coated cartridge cases are dipped in a 25% solution of an ester gum (glyceride of colophony or

copal) in acetone. A perfectly waterproof coating is obtained in a few seconds after exposure to the air.

A. R. POWELL.

Purifying and decolorising rosin with resorcinol. NEWPORT Co. (B.P. 294,526, 15.6.28. U.S., 25.7.27).—Resorcinol is added to a heated solution of rosin in a solvent relatively immiscible with resorcinol, *e.g.*, petroleum naphtha. On agitation and cooling, the resorcinol mass containing the impurities and colouring matter originally present settles out, and the purified rosin solution is decanted off and subjected to distillation. The resorcinol is recovered by water-extraction.

S. S. WOOLF.

Phenolic resin and its manufacture. G. W. MILLER, ASSR. to BAKELITE CORP. (U.S.P. 1,717,614, 18.6.29. Appl., 1.3.27).—Cresol is condensed at 110° with furfuraldehyde in presence of lime or other basic condensing agent; the fusible resin produced is then incorporated with a non-reactive fusible resin, preferably a phenolic resin, and a further addition of lime at 150° is made, together with stearic acid or other lubricating agents.

R. BRIGHTMAN.

Production of phenolic condensation products. F. KURATH, ASSR. to ECONOMY PUSE & MANUF. Co. (U.S.P. 1,726,671, 3.9.29. Appl., 19.2.26).—Phenol-aldehyde "resoles" in the fusible stage, containing enough aldehyde (etc.) to yield a hard resin on heating, are treated with sufficient furfuramide to fix this aldehyde (etc.) within the mass in substantially non-volatile and reactive form.

C. HOLLINS.

Mouldable resin composition containing a fabric filler. BAKELITE CORP., ASSEES. of C. L. CHAMBERLIN (B.P. 316,275, 31.8.28. U.S., 27.7.28).—The material comprises a woven fabric, *e.g.*, coutil, impregnated with a fusible resinous binder (which may be a reactive phenolic resin) and divided into dry flakes of sizes (*e.g.*, $\frac{3}{8}$ —1½ in. \times $\frac{1}{8}$ —1½ in.) small enough to allow flow in the mould and large enough to prevent the flakes sticking in irregular masses; the cut pieces of fabric are impregnated in a kneading machine with a solution of the resin and dried. The laminated moulded products have a high shock-resistance.

E. LEWKOWITSCH.

Manufacture of plastic masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,496, 6.9.28).—The liquid acid portion, insoluble in light petroleum, of an oxidation product of paraffin hydrocarbons, waxes, etc. is esterified at 150—220° with organic compounds containing at least two hydroxyl groups in the molecule, *e.g.*, glycols, glycerol, dextrose, mixed, if desired, with organic unsaturated or hydroxy-acids or glycerides thereof, *e.g.*, linoleic acid, ricinoleic acid, plastic masses being thereby obtained. The acids may be treated with siccatives, *e.g.*, lead oxides, or with sulphur before or during the heat treatment.

S. S. WOOLF.

Manufacture of plastic masses. SINIT A.-G. (G.P. 455,551, 5.12.23).—Residues from the distillation of natural resins, or oils with b.p. above 350° obtained in tar distillation, are heated with sulphur, an inert material, *e.g.*, asbestos, and an excess of the condensation products of acetylene. The resulting plastic masses

are suitable for purposes of electrical or heat insulation.

A. R. POWELL.

Synthetic plastic material. C. A. NASH, J. P. TRICKEY, and C. S. MINER, ASSRS. to QUAKER OATS CO. (U.S.P. 1,736,047, 19.11.29. Appl., 13.1.23).—A mixture of 94 pts. of phenol and 104 pts. of furfuraldehyde with 1% of sodium carbonate as an activator is heated just below the b.p. until a stiff, plastic mass is produced. This is cooled, thinned by the addition of more furfuraldehyde, mixed with asbestos, moulded, and heated at 80—230° until it sets to a hard mass. A. R. POWELL.

Softening and rendering impervious materials composed of casein treated with formaldehyde or of cellulose derivatives. G. JAKOVA-MERTURI and J. A. POGGIOLI (B.P. 291,765, 7.6.28. Fr., 8.6.27).—Materials composed of casein treated with formaldehyde, or cellulose derivatives, e.g., cellulose acetate, celluloid, are softened and rendered impervious by treatment with a hot solution comprising one or more hypochlorites of the alkali metals and an alkali hydroxide, and subsequently with a hot solution of colophony and sulphur (preferably 10% of each) in an oil or fat. While soft and hot the materials may be moulded etc.; on being cooled in cold water the products harden immediately without incidence of brittleness. S. S. WOOLF.

Moulding mixture. L. V. REDMAN, ASSR. to BAKELITE CORP. (U.S.P. 1,732,533, 22.10.29. Appl., 9.11.26).—The material is made by mixing casein 1 pt., carbamide 1 pt., paraformaldehyde 1 pt., ethylene glycol 0.4 pt., water 0.5 pt., and concentrated ammonia solution 0.13 pt., in a ball mill and passing the product through differential sheeting rolls whereby a tough, plastic mass is obtained. A. R. POWELL.

Carbon pigment (U.S.P. 1,730,389).—See II. Artificial cork (B.P. 322,752).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Interfacial energy between rubber and filling material. F. HARTNER (Kolloidchem. Beih., 1929, 30, 83—108).—A calorimeter for the measurement of the heats of swelling and of wetting of rubber in benzene is described. The physical properties of rubber are improved by the addition of a filling material such as lamp-black, zinc oxide, basic magnesium carbonate, or kaolin, and the effect of the filling material depends on the interfacial energy between the rubber and the filler. Using the apparatus described, a method is devised for calculating this value from measurements of (a) the heat of swelling of raw rubber in benzene, (b) the heat of wetting of the filling material by benzene, and (c) the heat of swelling of the mixture in benzene. The mechanical properties of rubber containing different amounts of filling material have also been examined. The heat of swelling of rubber increases with the age of the preparation.

E. S. HEDGES.

See also A., Jan., 53, Determination of minute amounts of copper (CALLAN and HENDERSON). 91, Quebrachitol from serum of *Hevea* latex (LEVI).

PATENTS.

Manufacture of rubber compositions. J. H. REEL and H. E. CUDE, ASSRS. to NAUGATUCK CHEM. CO. (U.S.P. 1,735,547, 12.11.29. Appl., 23.5.27).—An emulsion of 100 pts. of water in 15 pts. of spindle oil and 5 pts. of stearic acid is mixed at 65—90° with 100 pts. of masticated crêpe rubber in an emulsifying machine to obtain a viscous dough, which is treated with sufficient alkali to combine with the stearic acid, whereby the emulsion reverses to one of the oil-in-water type and changes from a sticky mass to a smooth, velvety product resembling concentrated latex. A. R. POWELL.

Manufacture of accelerators for rubber vulcanisation. GOODYEAR TIRE & RUBBER CO., ASSEES. of J. TEPPEMA (B.P. 303,827, 31.10.28. U.S., 10.1.28).—2-Thiolbenzthiazole ("mercaptobenzthiazole") is combined with nitrosodimethylaniline or nitroso-β-naphthol by melting together the ingredients. The product, (R·S)₂NR', is a vulcanisation accelerator.

C. HOLLINS.

Preservation of rubber. GOODYEAR TIRE & RUBBER CO., ASSEES. of A. M. CLIFFORD (B.P. 309,161, 29.1.29. U.S., 7.4.28).—The substantially non-accelerating reaction product of sulphur and an amine, particularly a secondary arylamine, is incorporated in rubber as a preservative agent. This type of product contains the grouping $S < \begin{smallmatrix} R \\ R' \end{smallmatrix} > NH$, where R and R' are hydrocarbon radicals. Thiodiphenylamine, thiophenyl-β-naphthylamine, and thio-ββ-dinaphthylamine are mentioned. D. F. TWISS.

Manufacture, from latex, of an artificial gutta-percha and a non-hygroscopic rubber. E. S. ALICOHEN (U.S.P. 1,739,566, 17.12.29. Appl., 10.9.28. Holl., 20.1.27).—See B.P. 313,373; B., 1929, 652.

XV.—LEATHER; GLUE.

Chrome tanning. IX. Effect of rendering chrome-tanning liquors basic with sodium carbonate. E. STIASNY, E. OLSCHANSKY, and S. WEIDMANN (Collegium, 1929, 565—574).—Normal chromium salts were rendered basic by calculated amounts of sodium hydroxide and carbonate and the basicity of the product was determined by the usual method and also by oxidising and precipitating with barium chloride. In the usual method the boiling during the acid titration removes some of the carbonate groups from the carbonato-complexes, thus giving a higher basicity figure. By the precipitation method the basicity figures were 25% (calc. 33%) and 47.1% (66.6%). The oxidation method is applicable to basic chromium chloride and nitrate solutions only. For basic carbonato-chromium sulphate liquors, a portion of the liquor is boiled with a measured quantity of N-ammonia solution and N-ammonium chloride and about 6.8 g. of barium chloride, filtered into 30 c.c. of N-hydrochloric acid, diluted to 1 litre, and 100 c.c. of the filtrate are mixed with formaldehyde and titrated with 0.2N-sodium hydroxide. From the titration figure thus obtained the total acid radicals in the chrome liquor can be calculated and the

actual basicity determined. Close agreement has been shown between this formaldehyde titration method and indirect methods of determining the carbonate and sulphate ions in the complex salts. The more basic the carbonato-chromium sulphate compound, the greater is the difference between the basicity figure as given by the usual method of determination and the actual basicity as obtained by the formaldehyde titration method. These differences diminish slowly on ageing such liquors, but rapidly by boiling. The carbonato-chromium complexes change into hydroxo-complexes, hence the differences between rendering the chrome liquor basic with sodium hydroxide and carbonate, respectively, disappear. The carbonato-complexes are not formed to the same extent if the chrome liquor is vigorously stirred during the addition of the sodium carbonate solution, or if the chrome liquor is boiled before adding the sodium carbonate. Less carbonato-complex is formed with chromium sulphate than with chromium chloride solutions. Less carbonato-complex is formed in a chromium chloride solution to which sodium sulphate has been added. D. WOODROFFE.

The Luckhaus quick-tanning process. L. JABLONSKI (Collegium, 1929, 574—578).—The pelts are suspended from the upper cover of a cylindrical vessel which may revolve on an axis passing through the cylinder to the bottom end. The upper cover is provided with pipes leading to a pump; the lower end is fitted with inlet and exit tan-liquor pipes and with a heating device. The upper cover carrying the pelts is fitted into the cylinder, which is then evacuated, whereby liquor is drawn in from the bottom. The tan liquor is maintained clear by filtration, and a quick tannage ensues. The efficacy of this and other quick-tanning processes is attributed to the opening up of the pelt fibres rather than to evacuation of the pelt itself. Alcohol-dehydrated pelt tans quickly until the pores are filled; tannage then ceases. Synthetic tannins tan more quickly than natural tannins, but they do not give weight. D. WOODROFFE.

Rapid method of determining the chromium in used chrome [tanning] liquors. F. FEIGL, K. KLANFER, and L. WEIDENFELD (Collegium, 1929, 589—594).—A quantity of the filtered chrome liquor sufficient to contain about 0.02—0.04 g. Cr is mixed with 10 c.c. of 20% sodium hydroxide solution and 25—30 c.c. of saturated bromine water, boiled for 5 min. until pure yellow in colour, 10 c.c. of a 2% solution of potassium thiocyanate are added, and the mixture is boiled for a further $\frac{1}{2}$ —1 min. The chromium in the product is then determined iodometrically. In presence of iron the solution is acidified with phosphoric acid, otherwise sulphuric acid is employed. Acidification should follow addition of the potassium iodide solution, otherwise momentary reduction will occur. The method is applicable to the determination of chromium in chrome-tanned leathers. The excess of hydrogen peroxide in these oxidation processes can also be removed by boiling the oxidised chrome liquor with 5 c.c. of a 5% solution of nickel nitrate for 3 min., but this method is not as good as the other for liquors containing a large amount of dissolved protein matter. D. WOODROFFE.

Determination of insoluble matter in tannin extracts. E. STIASNY (Collegium, 1929, 578—583).—Very wide variations in the results are obtained in the determination of insoluble matter by the filter-paper and filter-candle methods owing to the influence of the method of dissolving the extract for analysis. It is extremely difficult to ensure that different analysts shall dissolve the extract in exactly the same way. Much better concordance has been obtained by the sedimentation method of determining the insoluble matter. The vessel in which the tannin solutions are cooled should contain 10 litres of water for each flask placed therein, not more than six of which should be present. Water kept at 18°, with as little variation as possible, should be allowed to flow through the container at such a rate that it would fill the container in 10—12 min. at most. The flasks should be 10 cm. apart, be immersed in water up to the neck, and be moved about during the first 15 min. D. WOODROFFE.

Tanning value of *Anogeissus latifolia* leaves. ANON. (Bull. Imp. Inst., 1929, 27, 452—455).—Examination of three samples of leaves from *Anogeissus latifolia* ("Dhawa"), grown in India, gave: moisture 12.3—14.0%; insoluble matter 33.7—44.5%; extractive matter (non-tans) 10.0—13.8%; tannin 32.2—38.5%; ash 3.9—4.4%; tintometer readings (solution containing 0.5% of tannin in 1-c.c. cell), red 1.5—1.9, yellow 4.9—6.5. Leather produced by the three samples was similar and of light colour and good quality. E. H. SHARPLES.

Determination of proteolytic bating enzymes. M. BERGMANN and O. DIETSCH (Collegium, 1929, 583—589).—The bating material (10 g.), after digestion with 500 c.c. of water for 30 min. at 18° with frequent shaking, is filtered and 25 c.c. of the clear filtrate are mixed with 75 c.c. of a 15% solution of ammonium sulphate, the mixture is heated for 15 min. in a water-bath at 35°, a specially prepared, grey gelatin strip is immersed in the preparation for 20 min., then washed in running water, and dried with alcohol, and the bleaching of the colour is compared with that of standard strips treated with enzyme preparations of known concentration, from which that of the particular bating material can be deduced. D. WOODROFFE.

Utility of the Zeiss step-photometer in tannery laboratories. A. KÜNTZEL (Collegium, 1929, 549—560).—The relative proportions of normal colour, white, and black can be obtained by examining coloured black or white leathers with the Zeiss step-photometer. It can also be used for colorimetrically determining the concentration and purity of dye solutions, and the colour value of vegetable tan liquors. Its suggested use for measuring the brightness of the glaze on a leather is not good. It can be used to show the relationship between constitution and colour of different chrome and vegetable tanning solutions, and with the addition of a nephelometer it can be used to determine the precipitation point of chrome tanning liquors. D. WOODROFFE.

Viscosity measurements as a method of testing glues. E. SAUER and O. BOCK (Z. angew. Chem., 1929,

42, 1176—1178).—The viscosities of mixtures of solutions of two grades of glue in various proportions are less than those of its constituents, supposing a linear relation to exist. The viscosity of glue solutions decreases rapidly when they are kept at 100°, the final values for various samples being from 40 to 72% of their initial viscosities.
J. A. V. BUTLER.

KePAYANG oils. GEORGI and TEIK. *Allanblackia Stuhlmannii* seeds. ANON.—See XII.

See also A., Jan., 33, **Hydrophile properties of collagen** (MEUNIER and LE VIET).

PATENTS.

Printing plates (B.P. 291,063).—See XI. **Leather polish** (B.P. 322,969—70).—See XII.

XVI.—AGRICULTURE.

Soil profile studies. II. Methods used in the profile survey of New Jersey soils. J. S. JOFFE and L. L. LEE (Soil Sci., 1929, 28, 469—479; cf. Joffe, B., 1929, 730).—Factors of importance in the examination of soil profiles are discussed. Methods of sampling for volume-weight determinations and for the physical and chemical examination of individual horizons are presented.
A. G. POLLARD.

Biochemical and biophysical factors in forest soils. D. FEHÉR and S. VAGI (Bot. u. Forstl.-Chem. Inst. kön. ung. Hochschule Berg- u. Forstingen. Forstl. Vers., Sopron, 1926, 28; Proc. Internat. Soc. Soil Sci., 1929, 4, 259—261).—The carbon dioxide content of forest air is greater than that of the free atmosphere, but decreases with the height above ground to about 8—10 m., where it approximates to that of the free air. Soil acidity is less important in forest soils than in arable areas, and only becomes injurious when acid humus and peat begin to be formed. At this stage the p_H value of the soil is sufficiently low to cause injury to the trees. Bacterial numbers decrease with soil p_H values, there being an accelerated depression at about p_H 4.0. The proportion of carbon dioxide in the air layer immediately in contact with the soil surface is directly related to bacterial activity.
A. G. POLLARD.

Microflora of forest soils. R. BOKOR (Bot. Inst. kön. ung. Hochschule Berg- u. Forstingen. in Sopron, Forstl. Vers., 1926, 28; Proc. Internat. Soc. Soil Sci., 1929, 4, 257—258).—Bacteria in forest soils are less numerous than in similar soils under cultivation, although the number of species represented is not markedly different. Soil reaction largely controls the number of organisms present. In soils of similar p_H value, bacterial numbers increase with the humus content and air capacity of soils, and these two factors are sufficient to explain differences in bacterial flora of soils bearing different species of trees. The absolute water capacity of a soil increases directly with the proportion of soil particles less than 0.5 mm. in diam. The absolute air capacity increases with the proportion of particles greater than 2.0 mm. in diam. and decreases as the number of particles of 0.01 mm. in diam. increases. Under similar physiological conditions bacterial numbers vary in-

versely as the number of soil particles of 0.01 mm. in diam. An increase in the latter leads to a predominance of anaerobic bacteria and the occurrence of soil acidity. Free nitrogen-fixing organisms tolerate a greater degree of acidity in forest soils than in cultivated soils. The number of pectin- and cellulose-destroying organisms varies inversely with the absolute air capacity of the soil. Among anaerobic organisms butyric acid-producing types exist in considerable numbers. Nitrifying organisms are restricted by the acid reaction of the soil and are dominated numerically by denitrifying species. Active bacterial activity occurs to a depth of 50—60 cm. below the soil surface.
A. G. POLLARD.

Microflora of Hungarian alkali soils. R. BOKOR (Mikrobiol. Lab. Bot. Inst. kön. ung. Hochschule Berg- u. Forstingen. Forstl. Vers., Sopron, 1928, 30, 206—209; Proc. Internat. Soc. Soil Sci., 1929, 4, 251).—Methods for the examination of alkali soil microflora are described. Solonetz soils have a characteristic flora (with a predominance of actinomycetes) which is not altered by the usual ameliorative processes applied to these soils. Highly organic manures are valuable on such soils.
A. G. POLLARD.

Physical and chemical examination of Danish heath soils, with special reference to the colloid and nitrogen contents. F. WEIS (Kgl. Danske Vidensk. Selskab. Biol. Medd., 1929, 7, 9; Proc. Internat. Soc. Soil Sci., 1929, 4, 254—257).—Among the many soils examined fertility was generally associated with a high proportion of fine particles. Light dressings of lime, insufficient to increase the p_H value of the soils, caused a rapid increase in ammonification and nitrification for a short period. The nitrogen content of the humus decreased with increasing depth from the surface. During the podsolisation of heath soils, sols of alumina, ferric hydroxide, and silica are formed and carried downward, being subsequently precipitated as gels to form a hardpan. Ferric hydroxide sol is precipitated first, followed in order by alumina and silica. Fertility decreases with the extent of podsolisation. The amount and distribution of inorganic colloids is a measure of the value of these soils for cultivation.
A. G. POLLARD.

Nature of acidity of mineral soils. S. OSUGI (Proc. III Pan-Pacific Sci. Cong., 1926 [1928], II, 1960—1971).—The inversion of sucrose by mineral acid soils is attributed to the surface action of soil particles; the p_H of the aqueous extract is not sufficiently low. The clay extract has a higher p_H than that of the coarser parts, and a higher p_H exists in the adsorbed water film of the soil.
CHEMICAL ABSTRACTS.

I. Microbiological analysis of soil fertility.
II. Rôle of phosphorus in nitrogen fixation.
III. Microbiological determination of the soluble phosphate requirement of soils. J. ZIEMIECKA (Rocz. Nauk Roln. i Leśnych, 1929, 21; Proc. Internat. Soc. Soil Sci., 1929, 4, 239—240).—I. The "direct" method of Winogradsky for examining microbiological processes in soils allows of precise measurements of the phosphate and lime requirements of soils by means of the activity of *Azotobacter*.

II. The amount of easily soluble phosphate utilised by *Azotobacter* per unit of nitrogen fixed decreases with

the amount of phosphate available. The minimum amount of phosphate necessary per 20 mg. of nitrogen fixed and per 2 g. of mannitol decomposed is 9.39 mg. P_2O_5 in the form of potassium dihydrogen phosphate. The phosphorus of tricalcium phosphate and of basic slag is utilisable by the organisms to a limited extent.

III. On phosphate-deficient soils the activity of *Azotobacter* is increased by phosphatic fertilisers. This forms a basis for the measurement of the phosphate requirement of non-acid soils. A. G. POLLARD.

Permeability of soils. GANOSSIS (Compt. rend. Acad. d'Athènes, 23.11.28; Proc. Internat. Soc. Soil Sci., 1929, 4, 236—237).—Treatment of soils with sodium chloride, nitrate, or carbonate rendered them impermeable at varying periods after treatment. This effect was reversed by subsequent treatment with chloride, nitrate, or sulphate of calcium, chloride or nitrate of potassium, or with hydrochloric or nitric acid. Repeated dressings of organic manures tend slowly to diminish soil permeability. Clay soils treated with potassium carbonate acquire properties more resembling those of sandy soils. A. G. POLLARD.

Freezing, thawing, and soil humidity in woods and fields. N. A. KATSHINSKY (Proc. Sci. Res. Inst. Physico-math. Fac., 1st Moscow State Univ., 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 232—236).—The nature of the thawing process in soil together with the soil-water movements accompanying it are discussed. A. G. POLLARD.

Development of the phenomenon of soil drought. L. S. LITVINOV (Agric. Exp. Sta. Perm, No. 2, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 246—250).—Plant injury during drought is not a characteristic of any particular soil-moisture content, but arises when the water-holding power of the soil just exceeds the suction force of the plant roots as demonstrated by the cessation of "bleeding" from the cut stem of the plant. The decrease of moisture content of soils under constant climatic conditions is paralleled by the amount of plant "bleeding," and by examining the latter the period of drought injury to the plant may be forecast. The moisture content at which plant injury begins varies with the soil type and the species of plant. A. G. POLLARD.

Measurement of carbon dioxide production in soils. D. FEHÉR (Bot. Inst. Roy. Hung. High Sch. Eng. Mines and Forests in Sopron, 1927, 29, 237—243; Proc. Internat. Soc. Soil Sci., 1929, 4, 228—229).—Lundegårdh's bell apparatus is adapted for measuring the carbon dioxide respiration of soils. A. G. POLLARD.

New apparatus for the mechanical analysis of soils by displacement. J. H. GOLLAN (Bull. Soc. Chim. biol., 1929, 11, 940—942).—An apparatus is described whereby analyses of soils are effected to comply with the standard of the International Soil Conference (Washington). C. C. N. VASS.

Influence of increasing amounts of calcium sulphite and sulphate on the germination and young development of grain plants. K. SCHARER and W. SCHROFF (Landw. Versuchs-Stat., 1929, 108,

217—251).—Experiments on the yield of wheat, rye, barley, and oats are recorded.

CHEMICAL ABSTRACTS.

Stimulating effect of external applications of copper and manganese on certain chlorotic plants of the Florida Everglades soils. O. C. BRYAN (J. Amer. Soc. Agron., 1929, 21, 923—933).—Copper or manganese sulphate was applied to the leaves of cow-peas or sorghum or to the soil with equal success. Plants grown on the copper-treated soil contained appreciable quantities of copper. CHEMICAL ABSTRACTS.

Increasing protein content of pasture grasses by frequent light applications of nitrogen. C. R. ENLOW and J. M. COLEMAN (J. Amer. Soc. Agron., 1929, 21, 845—853).—With frequent mowing the protein content was higher. The protein content of pasture grass can be maintained at a high level by frequent light applications of a nitrogenous fertiliser. Rainfall, rather than available soil nitrogen, is a limiting factor in forage production. CHEMICAL ABSTRACTS.

Sunn hemp. S. D. TIMSON (Rhodesia Agric. J., 1929, 26, 668—682).—*Crotalaria juncea* contains 53% H_2O ; the dry matter contains N 2.31, P_2O_5 0.26, K_2O 1.26%. The seed contains H_2O 8.6, protein 31.2, other nitrogenous compounds 3.4, fat 4.3, starch 41.1, fibre 8.1, ash 3.3%. The seed may contain an alkaloid, but does not contain cyanogenetic glucosides. CHEMICAL ABSTRACTS.

Nitrogen fixation by the root-nodule bacteria of the Leguminosæ. E. W. HOPKINS (Soil Sci., 1929, 28, 433—447).—The nitrogen-fixing power of various cultures of *Rhizobia* in the absence of a host plant was examined. No positive results were obtained. A. G. POLLARD.

Sugar beet culture on sandy soils. S. L. LOUWES, J. HUDIG, C. MEIJER, O. J. CLEVERINGA, and F. DECHERING (Z. Pflanz. Düng., 1929, 8B, 608—614).—On sandy soils sugar beet yields are largely influenced by the "lime condition" of the soil, being adversely affected by both deficiency and excess of lime. The examination of the "lime condition" and the selection of suitable fertilisers for these soils are discussed. A. G. POLLARD.

Sugar beet trials with various nitrogen fertilisers in 1928. H. RÖSSLER (Z. Pflanz. Düng., 1929, 8B, 599—605).—In the field trials recorded the efficiencies of Chili saltpetre, sodium nitrate, calcium nitrate, and a mixture of Leuna saltpetre and potassium nitrate were, in general, of the same order, but in each case the percentages of dry matter and sugar were rather higher in the case of sodium nitrate. A. G. POLLARD.

Influence of nitrogen manuring on the yields from mineral soil pastures. MÖLLER and PÖCKER (Z. Pflanz. Düng., 1929, 8B, 582—591).—Trials of meat and milk production indicate the value of the intensive pasturing system. The nature of the nitrogen fertilisers used largely affected the results obtained. Urea was rapid in action, and calcium nitrate still more rapid, but its action was short-lived. Best results were obtained with Nitrophoska. Part of the nitrogen

dressing is preferably applied in the summer. Nitrogen fertilisers ensure good pasture in dry seasons.

A. G. POLLARD.

Nitrogen manuring of fish-ponds. E. WALTER and O. NOLTE (Z. Pflanz. Düng., 1989, 8B, 605—608).—Polemical against Kuhnert (B., 1929, 448).

A. G. POLLARD.

Nutritive requirements of milch cows in relation to the composition of the milk produced. N. HANSSON (Proc. VIII World's Dairy Cong., 1928, 190—197).—The results of tests carried out over a period of 20 years are summarised.

CHEMICAL ABSTRACTS.

Potassium nitrate [as a fertiliser]. F. MÜNTER (Z. Pflanz. Düng., 1929, 8B, 591—599).—In certain cases Nitrophoska (a ternary mixture of ammonium chloride, ammonium phosphate, and potassium nitrate) showed unduly low efficiency when used with sugar beet. The physiologically acid nature of ammonium phosphate is probably responsible for this. With sugar beet on neutral or acid soils ammonium phosphate is much less effective than either superphosphate or basic slag, but on chalked, neutral soils it produced higher yields than either of these fertilisers. In pot cultures and field trials potassium nitrate proved as efficient a source of nitrogen as sodium nitrate for potatoes, tobacco, sugar beet, and barley, but slightly inferior with wheat.

A. G. POLLARD.

Nitrate changes in a fertile soil as influenced by sodium nitrate and ammonium sulphate. N. PORGES (Soil Sci., 1929, 28, 449—455).—Variations in the nitrate content of a soil rich in nitrogen following treatment with sodium nitrate and ammonium sulphate are examined. The increased nitrate contents of the soil after incubation are irregular, and may correspond either to more or less than the amount of nitrogen added. A portion of the nitrate added to soil is rapidly converted into an insoluble condition, but becomes available at later periods. The fate of this nitrogen is discussed. The sum of the nitrogen removed by crops and that removed by leaching is approximately 70—77% of that originally available. Treatment of soil with sodium nitrate or ammonium sulphate leads to the mineralisation of part of the original soil-nitrogen.

A. G. POLLARD.

Available phosphorus of soil resulting from moisture and temperature variations, Big Horn Mts., Wyoming. T. J. DUNNEWALD (J. Amer. Soc. Agron., 1929, 21, 934—936).—The available phosphorus decreases with increase in rainfall, and in the lower soil zones of all the profiles as compared with the surface zones where most of the organic matter has accumulated.

CHEMICAL ABSTRACTS.

Value of raw sewage sludge as a fertiliser. J. F. MÜLLER (Soil Sci., 1929, 28, 423—432).—Chemical analyses and pot experiments show dried, raw sewage sludge to have notable value as a fertiliser. The addition of soluble nitrogen to reduce the C : N ratio of the mixture below 8 largely increases its crop-producing power. Supplementing with phosphate and small amounts of potash appears desirable. The water-

holding capacity of sandy soils is markedly improved by treatment with sludge.

A. G. POLLARD.

Values of sewage sludge as fertiliser. KLEBERGER and T. SCHRADER (Z. Pflanz. Düng., 1929, 8B, 577—581).—Examination of the clarification sludge from the process described, both by analysis and by Neubauer's method, showed it to be a valuable general fertiliser. The contained phosphorus exists mainly in colloidal organic combination. Soils repeatedly fertilised with the sludge would probably need rather more frequent liming than normally.

A. G. POLLARD.

Chemical and microbiological principles underlying the transformation of organic matter in stable manure in the soil. S. A. WAKSMAN and R. A. DIEHM (J. Amer. Soc. Agron., 1929, 21, 795—809).—In the decomposition of stable manure the pentosans and celluloses are diminished much more rapidly, and the lignins less rapidly, than is the total organic matter. Only the soluble nitrogen undergoes rapid nitrification in the soil. The organic nitrogen present originally in the manure, and that synthesised by the micro-organisms in the compost, become available only slowly.

CHEMICAL ABSTRACTS.

Extraction of adsorbed cations from soil by electrodialysis. B. D. WILSON (Soil Sci., 1929, 28, 411—421).—The adsorption of calcium, potassium, magnesium, and aluminium from solutions of their acetates by electrodialysed soil was unaltered by previously rendering the dialysed soil alkaline with calcium or magnesium acetate. Equivalent amounts of calcium, magnesium, and potassium were adsorbed from individual solutions, and in subsequent electrodialysis calcium and potassium were removed in equivalent amounts, but in much greater quantity than was magnesium. Soil adsorbed aluminium when the clay complex was saturated with hydrogen or with calcium, but was only recovered in minute amounts by electrodialysis. Electrodialysed soil removed quantities of calcium, potassium, and magnesium from mixed solutions, equivalent to those removed from individual solutions. The degree of adsorption of the cations and the rapidity of their removal by electrodialysis were in the order $K > Ca > Mg$.

A. G. POLLARD.

Superphosphate. HALE.—See VII. **Determination of moisture.** LAMPE.—See XI. **Sunflower seeds.** USPENSKI.—See XII.

See also A., Jan., 107, **Sugar-beet pulp etc. as nutriment for swine** (WOODMAN and others). 120, **Permeability of plant-cell membranes to sugar** (IRAMDAR and VARADPANDE). **Absorption of potash and phosphoric acid by cereals in early growth** (SCHRADER). **Physiology of apples** (BROWN). **Determination of sulphur and phosphorus in plants** (BERTRAND and SILBERSTEIN). **Determination of manganese in plant ash** (GARNIER).

PATENTS.

Manures or fertilisers. E. J. BURBAN (B.P. 291,461, 2.6.28).—Treatment of rock phosphate with nitric acid (d 1.20—1.27) gives a product which, when mixed with calcium phosphate, and limestone or quick-

lime to neutralise excess acidity, is claimed to be useful as a weed-killer and fertiliser. [Stat. ref.]

E. HOLMES.

Manufacture of granular mixed fertilisers containing ammonium nitrate. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,424, 21.9.28. Cf. B.P. 320,708; B., 1929, 1026).—Melts containing ammonium nitrate and, e.g., ammonium phosphate, potassium salts, but no calcium carbonate, are granulated by centrifugal spraying at a temperature 15–35° above their solidifying point. Suitable forms of apparatus are described.

L. A. COLES.

Production of fertiliser compound. A. MARTIN and J. JOHNSTON, JUN. (U.S.P. 1,733,745, 29.10.29. Appl., 28.10.26).—Humus fertiliser is made by finely grinding vegetable waste, fermenting the mass for 10–14 days in an open vat, agitating every 24 hrs. and keeping the mass warm, and mixing the product with an equal amount of sawdust.

W. G. CAREY.

Preservation of flowers, foliage, and the like. H. J. VALENTINE (B.P. 322,806, 12.9.28).—The materials are first treated with a preservative (e.g., alcoholic solution of boric acid and resin) having an acid reaction for colours red to pink or an alkaline reaction for colours blue to white. Coatings are then applied, in turn, of a waxy material, a resinous binding material, and a suitable colloid (gelatin, albumin, etc. subsequently hardened with formaldehyde). Preservatives and a filler (e.g., zinc oxide) may be incorporated in one or both of the coatings.

A. G. POLLARD.

Herbicide solution of hygroscopic chlorate. R. N. CHIPMAN (B.P. 322,438, 30.8.28. Cf. B.P. 273,268; B., 1929, 448).—Mixed aqueous solutions of sodium chlorate and rather more than the equivalent amount of calcium or magnesium chloride are claimed as useful non-drying herbicides. For use against plants of marine ancestry insufficient water to hold in solution all the sodium chloride formed is used in the manufacture.

E. HOLMES.

Treatment of phosphate rock (U.S.P. 1,736,553).—See VII. **Pest-destroying agents** (B.P. 322,179).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Sugar beet culture. LOUWES and others. **Sugar beet fertilisers.** RÖSSLER; also MÜNTER.—See XVI.

See also A., Jan., 36, **System strontium oxide-sucrose-water** (REINDERS and KLINKERBERG). 69, **Composition of cholla gum** (SANDS and KLASS). 72, **Starches and their amyloses** (TAYLOR and WALTON). **Structure of starch** (KARRER and VON KRAUSS). 115, **Fermentation of dextrose, lævulose, and arabinose** (PEDERSON).

PATENT.

Starch conversion product. D. J. BLOCK, Assr. to W. D. STEIN (U.S.P. 1,733,574, 29.10.29. Appl., 21.1.27).—See B.P. 282,178; B., 1928, 170.

Cellulose from sugar cane (B.P. 322,763). **Adhesive** (G.P. 455,014).—See V.

XVIII.—FERMENTATION INDUSTRIES.

High-temperature yeasts. STAIGER and GLAUBITZ (Z. Spiritusind., 1930, 53, 2–3).—Samples of sterile malt wort were seeded at 30°, 35°, 40°, 45°, and 50° with corresponding quantities of *Saccharomyces Pombe*, *S. Thermitonum*, and a distillery yeast, termed Race M, and the yields of alcohol and acid and the biological strengths of the yeasts compared after a fermentation of 72 hrs. The results differed little at 30° and 35°, and an optimal amount of alcohol was obtained at 35°. At 40° the yields of alcohol were lower and *S. Pombe* and *S. Thermitonum* were slightly more resistant than was the distillery yeast. At 45°, although less active, *S. Thermitonum* showed a marked superiority over the other two yeasts, whilst at 50° the fermentative activity of all three was destroyed. By seeding the yeasts at 25° and raising the liquid slowly to the same five temperatures as in the first experiment, the yield of alcohol and biological stability were greater, and decreased but little until 45° was reached. As before, the greater yields of alcohol at the higher temperatures were obtained with *S. Thermitonum*. At 50° the fermentative activities of all three yeasts were again reduced almost to zero.

C. RANKEN.

Influence of black mould on the production of amylase in green malt. J. DEHNICKE (Z. Spiritusind., 1930, 53, 1–2).—The occurrence of black mould may be due to weather conditions during the harvest or to damp storage of the barley. The mould generally appears on the green malt about the ninth day of growth, and by checking the production of amylase and destroying the amount already secreted, the diastatic power of the malt on the ninth day may be halved after 16 days of growth. The increase in acidity of the malt due to the mould is so small that the destruction of the amylase cannot be attributed to that cause. To minimise the loss due to the lowered saccharifying power, the malt may either be used in its short grown condition before the attack of the mould, or, if longer grown, washed with water at 25–30° before crushing. With either method the proportion of malt to potatoes in the mash will require to be raised above normal. It is preferable to treat the suspected barley by adding disinfectants such as formalin, montanin, or sulphuric acid to the steep water.

C. RANKEN.

Separation of protein-tannin during the boiling of wort. B. D. HARTONG (Woch. Brau., 1929, 46, 543–545).—The separation of hop tannin from the boiled wort was greater than that of malt tannin, and the precipitation of protein was greater with hop tannin than with malt tannin. The higher the concentration of hop tannin in the wort, the less was the content of total tannin after boiling owing to the coagulated hop tannin-protein compounds adsorbing and removing part of the malt tannin. Owing to oxidation being relatively restricted during boiling in the brewery copper the precipitation of tannin from the hopped wort was less than when the wort was boiled in laboratory experiments. A shorter duration of boiling also diminished the amount of tannin separated. The introduction into the copper of special apparatus to increase the turbulency of the boiling produced increases in the precipitation of

the protein-tannins. Under such conditions the hop tannins could be completely separated, leaving only malt tannins in solution. Such a beer is less sensitive to the influence of chilling. C. RANKEN.

Separation of fusel oil during rectification.

B. LAMPE (Z. Spiritusind., 1929, 52, 389).—Effective separation of fusel oil from the alcoholic distillate by means of added water or sodium chloride solution takes place when the content of alcohol is about 42% by wt. Under normal conditions the alcohol content of the distillate in the lowest three compartments in the rectifier varies between 40 and 45% by vol., and the spirit tapped from these compartments gives a high yield of fusel oil in the separator. Under certain conditions, depending on the composition of the mash and the working methods, the spirit from these compartments may yield little or no fusel oil owing to the concentration of the alcohol being very much less than 42% by wt., and the optimum zone for fusel oil may be reached only in the fourth and fifth compartments from the foot of the rectifier. Accordingly, to get optimum results the compartments from which to draw the alcoholic distillate during rectification should be determined in each case by practical experiment. C. RANKEN.

Development of distillation processes for producing ethyl alcohol. G. T. REICH (Chem. and Met. Eng., 1929, 36, 716—719).—Consequent on the increased price of molasses and the production of synthetic methyl alcohol, improved methods of distillation of ethyl alcohol have been necessary. The tendency has been in the direction of lower fuel consumption in the stills combined with a better recovery of by-products. Amongst the systems described are those of Barbet, Chute, and Reich. C. A. KING.

Detection and determination of formic acid and formaldehyde in vinegar. H. C. S. SNETILAGE (Chem. Weekblad, 1929, 26, 611—612).—Reduction with magnesium ribbon under specified conditions, and application of the Schiff-Elvove test permit the detection of 0.01% of formic acid. Formaldehyde, if present, is removed by distillation after neutralisation.

S. I. LEVY.

See also A., Jan., 113, **Proteolytic enzymes of green malt** (LINDERSTRÖM-LANG and SATO). 114, **Fermentation of maize by *Clostridium acetobutylicum*** (STILES and others). **Fermentation of dihydroxyacetone** (VIRTANEN and others). 115, **Fermentation of dextrose, lævulose, and arabinose by organisms from spoiled tomato products** (PEDERSON). 121, **Amylase from *Zea mais*** (PATWARDHAN). **Enzymes from seeds of *Cæsalpinia bonducella*** (PATWARDHAN).

PATENTS.

Production of products from organised substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,465, 2.6., 2.11., and 8.11.28).—The cellular structure of organised substances such as yeast is decomposed by autolysis or by protracted enzymic treatment, the enzymes required being added, if necessary, in the form of bacterial cultures. After filtration of the water-soluble from the water-insoluble products, sterols and

other products are extracted from the latter by organic solvents, preferably after treatment with alkali.

C. RANKEN.

Pasteurising apparatus for beer, wine, etc.

GEBR. SCHÄFFLER MASCHINENFABR. (G.P. 456,091, 21.2.25).—The apparatus comprises a series of travelling vats which pass in succession through a pasteurising chamber. This chamber is provided with circulating vessels from which heating or cooling liquids are passed through the jackets of the vats on the countercurrent principle so as to bring the contents gradually to the correct pasteurising temperature and just as gradually to cool them again. A. R. POWELL.

Denaturant [for ethyl alcohol].

S. ISERMANN, J. W. ORELUP, and E. OHLSSON (U.S.P. 1,730,850, 8.10.29. Appl., 24.5.27).—Use of a chlorophenol, or preferably of a mixture of *o*- and *p*-chlorophenols, is claimed. F. G. CLARKE.

Butyl [alcohol]-acetic fermentation process.

W. J. EDMONDS, ASSR. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,740,162, 17.12.29. Appl., 3.4.26. Renewed 24.5.29).—See B.P. 268,749; B., 1928, 345.

Separation of liquids (B.P. 304,756).—See I. **Detergents** (F.P. 630,409).—See XII.

XIX.—FOODS.

Activated milk. HOFFMAN (Proc. VIII World's Dairy Cong., 1928, 460—462).—An apparatus for the irradiation of milk with ultra-violet light without causing change of flavour is described. With raw milk, a protective gas (e.g., nitrogen) may be used, but this is superfluous with pasteurised milk in which vitamins-A and -C have already been destroyed. Experiments illustrating the prophylaxy and cure of rickets in rats by feeding activated milk are described.

CHEMICAL ABSTRACTS.

Ash and non-fatty solids content, sp. gr., and determination of refractive index of the calcium chloride serum in individual samples of milk.

L. BÉM (Proc. VIII World's Dairy Cong., 1928, 767—768).—No strict relation between the refractive index and the other values was observed. After 12—18 hrs. the average increase in the refractive index was 0.5°. In general, morning milk has a higher ash content and a smaller solids-not-fat content, sp. gr., and refractive index than has midday or evening milk.

CHEMICAL ABSTRACTS.

Reductase time and bacterial count of milk.

A. VAN RAALTE and M. M. LERNER (Chem. Weekblad, 1929, 26, 613).—The reductase time and number of bacteria show no absolute parallelism, but examination of 186 samples showed that a milk with a shorter time than 3½ hrs. has a count of more than 1 million per c.c.

S. I. LEVY.

Determination of nitrates and nitrites in whey.

E. OHLSSON and H. FREDHOLM (J. Assoc. Off. Agric. Chem., 1929, 12, 429—434).—In the absence of nitrites, nitrates in whey may be determined by extraction with ether for 60—70 hrs. in Widmark's rocking apparatus (cf. A., 1928, 96, 1064), and, after removal of residual

ether, reducing the nitric acid with a zinc-copper couple in the presence of magnesia, distilling the ammonia into boric acid, and titrating with hydrochloric acid against bromophenol-blue (cf. Scales and Harrison, A., 1917, ii, 41; 1920, ii, 386; 1921, ii, 345). If nitrite be present in addition to nitrate, nitric acid is determined by extraction as before, firstly, after removal of the nitrite by precipitation with 2:4-diamino-6-hydroxypyrimidine sulphate (cf. Hahn, A., 1917, ii, 382), and, secondly, after oxidation of the nitrite with potassium permanganate, the difference in the determinations being the nitrate originally present as nitrite. The errors in check analyses did not exceed 1.4%.

H. J. DOWDEN.

Fermentation taste of butter. A. I. VIRTANEN (Valion Lab. Julkaisuja, 1929, 12 pp.; Chem. Zentr., 1929, ii, 361).—The bacterial causes of fruity and putrefactive taste of unsalted or slightly salted butter were investigated.

A. A. ELDRIDGE.

Digestibility trials on Indian feeding stuffs. IV. Punjab hays (II). V. American and Indian cotton seeds. P. E. LANDER and L. C. DHARMANI (Mem. Dept. Agric. India, 1929, 10, 169—180, 181—192).—IV. Examination of daily nitrogen balances in feeding experiments with four hays collected from Rawalpindi, Murree, Lahore, and Amballa showed that only the last hay was of a maintenance standard for Montgomery heifers. This hay was mostly made from "Anjan grass," *Pennisetum Cenchroides*, Rich, and was relatively rich in phosphates, potash, total ash, and protein. (Cf. B., 1928, 835.)

V. American cotton seed, var. 285 F., 289 F., and 4 F., of *Gossypium hirsutum*, is richer in fat and protein than *Desi* cotton seeds (*G. indicum*). Digestibility coefficients of the various seeds do not vary very much, but the digestible starch equivalent and digestible protein per 100 lb. of cotton seed, and the protein ratio, reveal the superior value of var. 285 F., and to a smaller extent that of the other American varieties. The adhering lint on the American varieties of seed had no ill effects on the cattle used in the tests. E. HOLMES.

Detection and determination of nitrates and nitrites in meat foods. H. C. S. SNETHLAGE (Chem. Weekblad, 1929, 26, 612).—Modifications of the diphenylamine method are described for the detection. For determination of the nitrate, the colorimetric brucine method is most suitable, and of the nitrite, the Grierson-Romijn reagent is suitable for a colorimetric method.

S. I. LEVY.

Simplification of analytical methods. H. C. S. SNETHLAGE (Chem. Weekblad, 1929, 26, 603—604).—It is suggested that time and expense might be saved, in the examination of foods and drugs, by the adoption of short methods known to give results having a constant relation to the results obtained by the methods legally prescribed, the latter being employed only in cases of doubt or special importance.

S. I. LEVY.

Rapid determination of moisture. B. LAMPE (Z. Spiritusind., 1929, 52, 387—388).—The moisture content of the substance is deduced from its dielectric constant, which is determined in a special form of apparatus described by the author. Advantage is

taken of the fact that the dielectric constants of starch, sugar, proteins, and fats are all less than 10, whereas that of water is relatively high, at 81. At present the method is confined to the determination of moisture in cereals, fruits, and vegetables, and the results differ by less than 0.5% from those obtained by the use of the steam drying-oven. The grain need not be ground and potatoes require only to be mashed. In the determination, which occupies only a few minutes, the substance is placed between the metal plates of a condenser, and owing to a greater quantity of the substance being used for the measurement than is possible with other methods, a better average sample with its increased accuracy is permitted.

C. RANKEN.

Composition of some Philippine fruits, vegetables, and forage plants. A. VALENZUELA and P. J. WESTER (Philippine J. Sci., 1930, 41, 85—102).

Ghee substitutes. NORMANN. *Erucastrum elongatum* seed oil. BELYAEV. *Allanblackia Stuhlmannii* seeds. ANON.—See XII. Combined sugar in egg-white. KITAMURA. Detection of saponins in food. KOFER and others.—See XX.

See also A., Jan., 118, Vitamin-A of head lettuce leaves (KRAMER and others). 119, Vitamin-C from lemon juice (GRETTE and KING). 121, Girasole and chicory (TRAUB and others). Theosterols of cacao (LABBÉ and others).

PATENTS.

Manufacture of butter. E. PERKS (B.P. 322,416, 25.4.29).—Cream in a thin layer is subjected to continuous vibration of suitable amplitude and frequency, churning being thereby effected. E. B. HUGHES.

Treatment of grain, cereals, flour, and the like. N. V. ELECTROCHEM. IND. (B.P. 307,426, 6.3.29. Holl., 7.3.28).—The product (grain, flour, etc.) is treated with a current of air which has been passed through a solution containing persulphuric acid and ammonium chloride.

E. B. HUGHES.

Material for improving baking processes. AKTIEBOLAGET PRACTIC CO., LTD. (G.P. 455,114, 24.8.24).—Milk containing lactic bacteria, nutrients such as ammonium sulphate, and lactose is maintained at 30—40° for 24 hrs., diastatic ferments are added, and the mixture is kept at 30—35° for a time such that the acidity does not exceed 1%. The product stimulates fermentation when used in the preparation of dough.

L. A. COLES.

Preservation or conservation of meat. H. WATKINS-PITCHFORD (B.P. 322,254, 7.9.28. Addn. to B.P. 291,818; B., 1928, 622).—The method of injection described in the prior patent is modified.

F. R. ENNOS.

Centrifugal separators [for cream]. J. and A. PERSONS (B.P. 307,075, 1.3.29. Belg., 2.3.28).

[Portable roller] mills for fine grinding [of grain]. K. BERBEKA (B.P. 322,383, 19.1.29).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Combined sugar in serum-albumin, egg-white, and peptone. K. KITAMURA (J. Kyoto Prefect. Med. Coll., 1927, 3, 20—24).—Serum-albumin (Kahlbaum)

contained 1.2—2.6% of combined, but no free, sugar. Egg-white contained 0.43—0.54% of free, and 0.20—0.36% of combined, sugar. Teruuchi or Witte peptone contained 0.6—1.0% of free, but no combined, sugar.

CHEMICAL ABSTRACTS.

Santonin from Scottish-grown *Artemisia*. J. Courts (Pharm. J., 1929, 123, 603—604).—The air-dried leaves and flower-heads of *Artemisia maritima*, L. (?), grown in Scotland yielded 0.81% of santonin.

E. H. SHARPLES.

Loss of alkaloids during desiccation of plants.

A. GUILLAUME (Compt. rend., 1929, 189, 706—708).—For the desiccation of *Lupinus mutabilis*, the optimum conditions which avoid loss of volatile alkaloids are stabilisation with alcohol vapour and subsequent drying at 70°. Without previous stabilisation, the highest yield of alkaloids is obtained by drying the plant at 70—80°.

A. A. GOLDBERG.

Aldehyde-oxidation reactions for phenols, particularly the opium alkaloids.

C. C. FULTON (J. Assoc. Off. Agric. Chem., 1929, 12, 434—441).—An extensive study has been made of phenol-aldehyde colour reactions in the presence of strong acid, and of the modifications produced by subsequent oxidation, especially in the case of the opium alkaloids. The oxidising reagents are prepared by mixing with 3 c.c. of concentrated sulphuric acid (a) 0.5 c.c. of bromine water, (b) 1 c.c. of 10% ferric sulphate solution, (c) 1 c.c. of dilute nitric acid (5 drops of concentrated acid in 50 c.c. of water), or (d) 1 c.c. of dilute nitric acid (1:30). By mixing the aldehyde and the oxidant, a "combination" reaction occurs which reproduces the effects of the reagents when used separately. As a development of earlier work (cf. A., 1928, 1386), the "combination" reagents recommended are (i) 0.8 c.c. of 10% ferric sulphate solution added, while cooling, to 6 c.c. of Marquis' reagent (2 drops of 37% formaldehyde in 3 c.c. of concentrated sulphuric acid), and (ii) 0.8 c.c. of 10% ferric sulphate solution added to 6 c.c. of Marquis' reagent diluted about 800 times with sulphuric acid; the characteristic colours given by morphine, codeine, ψ -morphine, and papaverine are described. Alkaloids such as cotarnine or hydrastinine, which contain an aldehyde group, or those, such as narcotine, narceine, or hydrastine, which yield an aldehyde on hydrolysis, can be made to give colour reactions with phenols, particularly with ψ -morphine.

H. J. DOWDEN.

Detection of saponins in medicine and food.

L. KOFLER, R. FISCHER, and H. NEWESLY (Arch. Pharm., 1929, 267, 685—699).—Strips of filter paper are covered with an alcoholic solution of cholesterol up to 3 cm. from the lower end and the alcohol is allowed to evaporate; the strip is then immersed in the solution and capillarisation allowed to take place. Any saponin present combines with the cholesterol, whilst other substances remain unaffected. The lower portion of the filter paper is washed with water, dried, and boiled for 2 hrs. with xylene, when the saponin-cholesterol compound is decomposed and the cholesterol dissolved away. The strip is then washed with ether, dried, and placed in blood-gelatin, when a hæmolytic area replaces that in which the cholesterol was present, and

no hæmolysis is produced in the remainder of the strip. The cholesterol-saponin compound is insoluble in water. This test is very sensitive and has been applied to a number of articles of food and to medicaments.

A. I. VOGEL.

Valuation of Ceylon citronella oil. A. W. R. JOACHIM (Perf. Ess. Oil Rec., 1929, 20, 475—477).—

A comparison of the analyses of 15 different market grades and of six standard grades of Ceylon and Java citronella oils showed that (a) there is no relationship between the total geraniol content of Ceylon oils and their density, refractive index, and optical rotation; (b) citronella oils with very great differences in geraniol content have very different physical properties; (c) the geraniol content of Ceylon oils varies from 55% to 64%; (d) the quality of Ceylon oils judged by their geraniol contents bears little relationship to their behaviour in Schimmel's adulteration test, and that the test is unreliable since pure, unadulterated oils containing, at times, high percentages of geraniol do not pass the test. Of the 21 oils, only one passed the "raised" Schimmel's test.

E. H. SHARPLES.

Cymbopogon oils from India. ANON. (Bull. Imp.

Inst., 1929, 27, 458—460).—Samples of oils distilled from *Cymbopogon clandestinus* and from a new, hitherto undescribed, species of *Cymbopogon* had the following respective constants: d_{20}^{25} 0.9319, 0.9734; $\alpha_D +45.96^\circ$, -48.67° ; n_D^{20} 1.495, 1.497; acid value 3.0, 4.9; ester value before acetylation 11.3, 10.7; ester value after acetylation 167.1, 178.1; aldehydes and/or ketones 18%, 11%; solubility in 70% alcohol at 15° 1:2.1 vols., 1:1.8 vols.

E. H. SHARPLES.

Petrolatums. TSCHERNOZHUKOV; TARASOV.—See II. **Activated milk.** HOFFMAN. Analytical methods.—SNETHLAGE.—See XIX.

See also A., Jan., 45, **Electrolytic reduction of arylarsinic acid** (NAKADA). 61, **Differentiation of α - and β -glycerophosphates** (FLEURY and MARQUE). 67, **Determination of citronellal** (WATERMAN and ELSBACH). 85, **Homologues of ephedrine** (FOURNEAU and SÁENZ DE BURUAGA). 87, **Homologous salols** (KRAUZ and REMENEC). 98, **Microchemical reactions for strychnine** (WAGENAAR). **Sinomenine, disinomenine, acutumine, and sinactine** (GOTO and SUDZUKI). **Microchemical test for alkaloids** (ROSENTHALER). 111, **Pharmacology of some p-hydroxybenzoic acid esters** (SCHÜBEL and MANGER). 116, **Tetraiodophenolphthalein as antiseptic and germicide** (NICKEL). **Quinoline derivative as antiseptic** (ARMITAGE and GORDON). 117, **Substitutes for insulin** (KAUFMANN). 118, "**Progynon**" (BUTENANDT). 121, **Liquorice root and extract** (HOUSEMAN and SWIFT).

PATENTS.

Manufacture of α -amino- β -arylpropionic acids and their substitution products. F. HOFFMANN—LA ROCHE & Co. A.-G. (B.P. 318,582, 4.3.29. Ger., 6.9.28).—The Erlenmeyer synthesis (A., 1893, i, 580) is shortened by the use of hydriodic acid (preferably with acetic anhydride and red phosphorus), by means of which the azlactone may be hydrolysed, the resulting benzamido-

cinnamic acid reduced, and the benzoyl group removed in one operation. The azlactone from hippuric acid and 3:5-di-iodo-4-anisoxymethylbenzaldehyde is converted by boiling with hydriodic acid (*d* 1.7), acetic anhydride, and red phosphorus into α -amino- β -(3:5-di-iodo-4-anisoxyphenyl)propionic acid, from which thyroxine is obtained by iodination. C. HOLLINS.

Manufacture of basic derivatives of substituted quinolinecarboxylic acids. SOC. CHEM. IND. IN BASLE (B.P. 294,118, 16.7.28. Switz., 15.7.27).—4-Carboxy-derivatives of 2-hydroxy- or 2-halogeno-quinolines or 2-quinolones, or their *C*-, *O*-, or *N*-substitution products, are esterified with aminoalcohols; the halogeno-esters may subsequently be treated with a sodium alkoxide, and *O*-ether-esters may be converted into hydroxy-esters by partial hydrolysis with dilute acid. Examples are: β -diethylaminoethyl 2-hydroxyquinoline-4-carboxylate, m.p. 125–126°, and its ethyl ether, b.p. 136°/0.02 mm. [hydrochloride, m.p. 186° (decomp.); hydriodide, m.p. 157°]; β -diethylaminoethyl *N*-methyl-2-quinolone-4-carboxylate (hydrochloride, m.p. 205°); β -diethylaminoethyl 2-chloroquinoline-4-carboxylate, b.p. 140–145°/0.01 mm.; β -diethylaminoethyl 2- β -diethylaminoethoxyquinoline-4-carboxylate, b.p. 170°/0.01 mm. (hydrochloride, m.p. 217°); ethyl 2- β -diethylaminoquinoline-4-carboxylate, m.p. 36°, b.p. 135–140°/0.015 mm. (hydrochloride, m.p. 162°; hydriodide, m.p. 143°); β -diethylaminoethyl 2-alkoxyquinoline-4-carboxylates in which the alkyl group is methyl, m.p. 23° (hydriodide, m.p. 133°), *n*-propyl, b.p. 154°/0.025 mm. (hydrochloride, m.p. 138°), allyl (hydriodide, m.p. 135°), isoamyl, b.p. 165–170°/0.05 mm. (hydriodide, m.p. 124°), *n*-heptyl, b.p. 172–175°/0.01 mm., *n*-octyl, m.p. 35°, b.p. 180–182°/0.03 mm., cyclohexyl, m.p. 41° (hydriodide, m.p. 146°), and benzyl, b.p. 210–215°/0.001 mm. (hydriodide, m.p. 105°). The carboxyl group may be in other than the 4-position. C. HOLLINS.

Cigar and tobacco flavouring and moistening substances. (SIR) G. C. MARKS. From RUMDOR CORP. (B.P. 322,340, 1.12.28).—Tobacco is enclosed in a container with a flavouring agent consisting of solidified rum (e.g., 95% of rum and 5% of sodium stearate).

E. B. HUGHES.

Barbituric acid derivative. F. BOEDECKER (U.S.P. 1,739,662, 17.12.29. Appl., 21.11.25. Ger., 6.12.24).—See B.P. 244,122; B., 1927, 237.

Manufacture of 4-hydroxy-3-acetamidoaryl-1-arsonic acid. L. BENDA and O. SIEVERS, ASSYS. to I. G. FARBENIND. A.-G. (U.S.P. 1,739,820, 17.12.29. Appl., 16.7.26. Ger., 28.5.25).—See B.P. 278,789; B., 1928, 37.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Desensitising action of sodium sulphite; addition of desensitisers to developers. F. M. HAMER (Phot. J., 1929, 69, 479–480).—Solutions of sodium sulphite varying in concentration from 3.3% to 15% showed definite desensitising properties, sufficient to

account for the increased action of pinakryptol-green and safranin in sodium sulphite solution. When working with Ilford Special Rapid and Special Rapid Panchromatic plates in conjunction with metol-quinol developer, no confirmation could be obtained of the statements that safranin and pinakryptol-green act more powerfully when added to the developer (cf. Hübl, B., 1926, 612; Crabtree and Dundon, *ibid.*, 853). The preliminary bath was in fact to be preferred.

J. W. GLASSETT.

Antifogging and antisensitising effects. S. E. SHEPPARD (Phot. J., 1929, 69, 482–483).—A new representation of the combination of antifogging and antisensitising agents, such as phenazine, thioanilides, and iminazoles, with silver bromide is put forward in which it is suggested that, according to the extent of the disturbance of the electrostatic equilibrium at the interface of the silver halide with the sensitising or development nuclei, complexes of varying stability are produced by the co-ordination of the silver and halide ions at the C:N linking to form a heterocyclic ring. If the disturbance is extreme a covalent combination of higher stability may take place.

J. W. GLASSETT.

Recent advances in our knowledge of the latent photographic image. S. O. RAWLING (Phot. J., 1929, 69, 471–478).—A critical survey, together with bibliography, of the recent work on the chemical identity of the sensitivity nuclei, and the various hypotheses concerning the mechanism of the aggregation of silver at these nuclei during exposure, the nature of the latent image, the primary action of light in the formation of the latent image, and the effect of adsorbed ions on the photosensitivity of the silver halides.

J. W. GLASSETT.

Employment of by-products of saccharin manufacture in photography and photometry. W. HERZOG (Z. wiss. Phot., 1929, 27, 177–179).—The application of *p*-toluenesulphonyl chloride as a photographic developer, of esters of *p*-toluenesulphonic acid in the synthesis of sensitisers of the cyanine type, and of Chloramine as a “hypo-remover” are described.

H. F. GILLBE.

See also A., Jan., 46, Photographic action of slow electrons (ROSSI and BERNARDINI). 51, Determination of iodides and bromides in chlorides (TOMÍČEK and JÁNSKÝ). 96, Cyanine dyes (HAMER).

PATENTS.

Production of photographic prints. I. G. FARBENIND. A.-G. (B.P. 307,512, 9.3.29. Ger., 10.3.28).—A direct print-out material yielding full-tone negatives of the original in brown or brown-black tints is prepared by treating a suitable support, which may be coated with a gelatin layer, with a solution of the chromate of a diazo compound (monodiazotised *p*-phenylenediamine). The salt may be prepared *in situ* by successive treatments with the chromate and diazo compound solutions. The salt unchanged by the light action is readily removed without decomposition by washing in water.

J. W. GLASSETT.

Photographic printing process. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 322,430, 30.7.28).—A non-fugitive organic substance (other than a ferric salt) which is partially reduced by light action, *e.g.*, diketocompounds of anthraquinone, aminonaphthol derivatives, certain dyes, is admixed with a colloid and coated upon a suitable support. After exposure the image is physically developed by successive treatments with a solution of a silver or mercury salt and an organic reducing agent in acid solution. *E.g.*, a sensitive coating composed of sodium anthraquinone-2 : 7-disulphonate 2 g., gelatin 10 g., and water 100 c.c., may be developed with a 1% silver nitrate solution, followed by a solution containing *p*-methylaminophenol sulphate 0.25 g., acetic acid 3 c.c., and water 1 litre; or a coating containing a saturated solution of flavindulin-O 10 c.c., 8% gelatin solution 50 c.c., 8% chrome alum solution 1 c.c., may be used in conjunction with a 1% silver nitrate solution followed by 1% *p*-phenylenediamine solution 4 c.c., 10% acetic acid 2 c.c., water 90 c.c. Both are fixed in faintly acid 5% sodium thiosulphate solution. J. W. GLASSETT.

Kinematograph films. I. G. FARBENIND. A.-G. (B.P. 300,991, 22.11.28. Ger., 22.11.27).—For film-identification purposes, one or each margin is treated with a dye solution, *e.g.*, ultramarine (5 g.), glycol acetate (100 c.c.), cellulose acetate (2 g.). Alternatively, the margins are given a thin coating of a 1–5% solution of collodion to prevent penetration of the developing and fixing solutions, and thus provide a thin edge of unchanged silver salt. J. W. GLASSETT.

[Preparation of multi-colour screens for] colour photography. H. WADE. From COMP. D'EXPLOIT. DES PROC. DE PHOTOGRAPHIE EN COULEURS L. DUFAY ("VERSICOLOR DUFAY") (B.P. 322,432–3, 3.8.28).

Printing plates (B.P. 291,063).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Microscopical examination of colloidal powders in polarised light. A. MICHEL-LÉVY and H. MURAOUR (Compt. rend., 1929, 189, 1192–1194).—Examination in the polarising microscope of numerous guncottons containing 11.7% N, and of mixtures of guncottons containing 11% and 13% N, has shown that highly nitrated cottons may be detected in mixtures from the furrowed yellow fibres parallel to the direction of stretch, as distinct from the uniform field normally obtained. In such mixtures the rate of combustion is raised (though the total nitrogen, and temperature and composition of the gases evolved, may be unchanged) owing to the formation of surface cavities by the rapid combustion of the highly nitrated portions and the consequent increase in emission surface. J. GRANT.

See also A., Jan., 74, **Salts of fulminic acid** (WÖHLER and WEBER; WÖHLER and BERTHMANN). 99, **Mercury compounds of thiophen and selenophen** (BRISCOE and others).

PATENT.

Coating composition (U.S.P. 1,733,177).—See XIII.

XXIII.—SANITATION; WATER PURIFICATION.

Reduction of biochemical oxygen demand of sewage by chlorination. H. G. BAITY and F. M. BELL (Sewage Works J., 1929, 1, 279–285).—Application of 5–7.5 pts. per million of chlorine reduced the demand by 7.4%; 8–9 pts. left 0–0.1 pt. of residual chlorine, whilst 10–15 pts. left 0.2–0.5 pt. of residual chlorine, the reduction in biochemical oxygen demand being 15.5 and 42.7%, respectively. The advantageous application of chlorine is discussed. CHEMICAL ABSTRACTS.

Zeolite water-treating system of the Beacon Street [Detroit] heating plant. J. H. WALKER and L. F. COLLINS (Ind. Eng. Chem., 1929, 21, 1020–1024).—Governing factors in the plant described were a congested site, demanding high rates of heat transfer, and nearly 100% make-up water as against 5% only in a modern power plant. Consideration of perfect elimination of scale therefore outweighed operating cost, and zeolite softeners were installed. The water leaving the softeners was dosed with 5% sulphuric acid by an automatic proportioning device. The de-aerating heaters and continuous blow-down arrangement are described. Some of the alkaline boiler-water from the blow-down was recirculated to the de-aerator to eliminate bicarbonate which was found to cause corrosion. The acid supplied contained 40 p.p.m. of sulphuric acid and 4.5 p.p.m. of phosphoric acid. After three years of work no serious difficulty has arisen, but corrosion of the zeolite tanks (probably due to carbon dioxide) has been noticed. The efficiency of utilisation of sodium chloride in regeneration is not more than 30%. Detailed analyses with *p_H* values and operating costs are given. (Cf. White and co-workers, B., 1927, 798.) C. IRWIN.

Air conditioning. BULKELEY.—See I. **Tar emulsions.** EADIE.—See II. **Sewage sludge as fertiliser.** MÜLLER; also KLEBERGER and SCHRADER.—See XVI.

See also A., Jan., 71, **Glucosides of simple and chlorinated *p*-hydroxybenzoic acids [as bactericides]** (SABALITSCHKA and SCHWEITZER).

PATENTS.

Composition for destroying noxious creatures [fumigant]. O. LIEBKNECHT (U.S.P. 1,736,448, 19.11.29. Appl., 8.1.27. Ger., 19.1.25).—A solution of chromyl chloride in carbon tetrachloride, with or without the addition of a chloronaphthalene, is claimed.

A. R. POWELL.

Manufacture of agents for combating and destroying pests. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,179, 27.8.28).—The agents comprise cyanides readily decomposed by moisture, *e.g.*, alkaline-earth or magnesium cyanides, together with non-reacting organic liquids (petroleum fractions, halogenated hydrocarbons, etc.), and, if desired, wetting agents and/or hygroscopic salts. L. A. COLES.

Heat exchanger for waste water (B.P. 322,366). **Air conditioning** (U.S.P. 1,735,611).—See I. **Absorptive carbon** (U.S.P. 1,736,051).—See II.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEB. 28 and MAR. 7, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Rapid determination of dust in air. L. C. McNAIR and J. F. HIRST (J. Ind. Hygiene, 1929, 11, 336—337).—See B., 1929, 701.

PATENTS.

Drying apparatus. B. MÜLLER (G.P. 394,572, 31.12.21, and Addn. G.P. 456,033, 27.10.26).—(A) The hot air and steam from the drying chambers pass through injector outlets into a flue, and thereby fresh air is drawn along the flue into the stack so that it exerts suction on the drying chambers and thus produces more rapid removal therefrom of the moist air. (B) Each chamber is provided with a number of these flues.

A. R. POWELL.

Drying, finishing, and improving mass-production articles. J. TRAUTMANN (G.P. 456,816, 28.11.23).—The articles traverse a series of chambers through which a current of gas or vapour is passed in the same direction or countercurrent. The temperature and pressure of the gas are increased or diminished as it passes from one chamber to the next, and devices are interposed between consecutive chambers for cleaning, drying, heating, or compressing the gas stream as required.

A. R. POWELL.

Protective heat- and sound-insulating coverings. RHEINHOLD & Co. VEREIN. KIESELGUHR- U. KORKSTEINGES. (B.P. 305,048, 28.1.29, and Addn. B.P. 310,950, 26.2.29. Ger., [A] 30.1.28, [B] 4.5.28).—The covering consists of thin sheet-metal or other material bent into angular or trough form and completely or nearly severed into links. In (A) the sheets are used alone, though they may be coated with something further to reduce the radiation; in (B) the sheets are used to support other insulating materials, *e.g.*, powder or "air-layer" foils.

B. M. VENABLES.

Evaporator with two separate heating systems. E. POKORNÝ (G.P. 456,193, 8.5.26).—The open ends of the tubes of the upper system dip below the surface of the liquid in the lower system so that the steam produced in the latter serves to force the liquid through the upper system. A ring-shaped vertical wall between the two systems serves to prevent ingress of steam from the lower into the upper.

A. R. POWELL.

Rendering gas-tight vessels for use at high temperatures or with increased or diminished pressure. C. A. SCHAEERER (Swiss P. 123,986, 20.12.26. Ger., 24.12.25).—Between the joints is interposed a hollow rubber washer filled with a liquid having a b.p. above the temperature at which the vessel is to be used.

A. R. POWELL.

Rendering vacuum vessels gas-tight. ROTA KESSEL- U. MASCHINENBAU-GES.M.B.H. (G.P. 456,719,

11.2.27).—The outside of the vessel is plated with a suitable metal in an electrolytic bath while a vacuum is maintained inside. In this way any pores are rapidly filled up until the walls become quite gas-tight.

A. R. POWELL.

Production of a fire extinguisher. E. BÉCHARD (F.P. 633,400, 14.8.26).—Carbon tetrachloride or other chlorinated hydrocarbon and a mineral, vegetable, or animal oil are stored in a fire extinguisher either mixed together or separated by a thin layer of water. The oil protects metal from attack by the halogenated hydrocarbon, and also raises the b.p. of the latter so that the fire-extinguishing material is less volatile and consequently more effective.

J. S. G. THOMAS.

Foam process of fire-extinguishing. F. NEUVILLE (F.P. 631,626, 23.6.26).—An anhydrous, finely-powdered mixture of basic products, *e.g.*, sodium bicarbonate, with aluminium sulphate, a foam-producing material, *e.g.*, extract of liquorice, glucose, saponin, and a hygroscopic material, *e.g.*, an alkali salt, is directed upon the fire by a stream of water.

J. S. G. THOMAS.

Substantially suppressing phosgene-formation when extinguishing fires with carbon tetrachloride.

I. G. FARBENIND. A.-G. (B.P. 317,843, 13.6.29. Ger., 25.8.28).—A liquid hydrocarbon, *e.g.*, 5% of solvent naphtha, decalin, or petroleum, and ammonia (2—3%) are added to the carbon tetrachloride. L. A. COLES.

Removal of the solvent from evaporators attached to absorption refrigerators working periodically.

E. KINDERMANN (G.P. 435,994, 11.3.25, and 457,311, 29.5.27).—(A) The residual liquid in the evaporator after the evaporation period is returned to the absorption vessel after the following refrigerating period. (B) The heating current is not switched on in the evaporator until the temperature of the liquid therein has reached a predetermined value.

A. R. POWELL.

Lubricating agent for the compressors of refrigerators. GEBR. BAYER (G.P. 456,102, 26.8.26).—

Substances such as ferric chloride which increase its density and decrease its surface tension are added to the glycerol used as a lubricant in the compressors of refrigerating plant.

A. R. POWELL.

Pulverisers of the impact type. R. HADDAN. FROM FUEL EFFICIENCY ENG. CORP. (B.P. 322,971, 8.3.29).—The rotor of a disintegrator comprises a number of paddles mounted on a disc; a method of detachably securing the paddles is described. B. M. VENABLES.

Grinding face for pulverising, grinding, or mixing machinery. G. CLARK (B.P. 322,974, 15.3.29).

—The stationary grinding surface is composed of a number of blades secured in the casing; they are spaced

* The remainder of this set of Abstracts will appear in next week's issue.

and inclined so that air, water, or other fluid may be drawn in by the motion of the rotor.

B. M. VENABLES.

Production of uniform, pulverulent mixtures. H. BOLLMANN (B.P. 314,941, 17.4.29. Ger., 5.7.28).—A viscous or wax-like material (*e.g.*, vegetable phosphatide) is mixed roughly with a powdered material (*e.g.*, meal) by known means, and the mixture is compressed, with or without heat, and then reduced to powder by a machine such as a disintegrator. The consumption of power is less than by kneading throughout.

B. M. VENABLES.

Pasteurisation of liquids. BRASSERIES NANTAISES, and L. PIERRE (F.P. 630,293, 4.3.27).—The liquid is passed through a system of spiral tubes provided with double walls in which the heating or cooling fluids are circulated countercurrent to the flow of the liquid. Between the heating and cooling systems an expansion vessel of large cross-section is interposed so that the liquid remains therein for sufficient time to complete pasteurisation.

A. B. MANNING.

Prevention of foaming of heated liquids. H. BACH (G.P. 455,871, 12.2.26).—Two horizontal sieves rotating in opposite directions about a common axis are arranged above the surface of the foaming liquid. A device to reverse the direction of rotation of the sieves periodically and brushes to clean the meshes are provided.

A. R. POWELL.

Purification and decolorising of aqueous solutions [raw sugar juice etc.]. G. E. VAN NES (B.P. 297,430, 19.9.28. Holl., 21.9.27).—The solutions are treated with a lyophilic colloid, *e.g.*, albumin, starch, silica, or material forming the same, and then with an acid and a base yielding an insoluble salt, *e.g.*, carbon dioxide and lime, at a temperature and p_H value such that the colloid is deflocculated, after which the precipitated material is removed.

L. A. COLES.

Centrifugal machines. N. FORSTER, and SUPER-CENTRIFUGAL ENGINEERS, LTD. (B.P. 322,942, 30.1.29).—The machine is of the decanting type with imperforate bowl. The decanting scoop is mechanically traversed in an adjustable definite ratio to the revolutions of the bowl, so that it may always be kept close to the layer of the heavier solid (in the case of two or more solids). It is stated that when a gluten liquor contains fibre and starch, the fibre may be decanted with the liquor, leaving a cake of starch granules only in the bowl.

B. M. VENABLES.

Centrifugal separating apparatus [for cleaning gas]. B. POCHOBRADSKY (B.P. 322,810, 14.9.28).—The gas passes axially through a centrifugal rotor of which the peripheral speed is much higher than the axial speed of the gas. The rotor is provided with blades shaped to accelerate the gas without shock, and is surrounded by a wall rotating with it, thus collecting the dust in a layer of concentrated dirty air. The latter is separated from the main axial stream of clean air by means of a stationary, annular wall closely adjacent the outlet side of the rotor, and is despatched to a cyclone or bag-chamber, whence the air, which may still contain some dust, is returned to the rotor.

B. M. VENABLES.

Continuous filtration. PATENTAKTIEBOLAGET GRÖNDAL-RAMÉN (G.P. 419,284, 29.5.23, and Addn. G.P. 455,718, 21.6.23. Swed., [A] 28.5.22).—(A) In order to classify the material during filtration the suspension is kept stirred during the filling of the filter chamber by blowing air through the filter cloth. (B) In operating this process with a rotary drum-filter, compressed air is passed into the filtrate container through a tube in the axis.

A. R. POWELL.

Apparatus for increasing the vacuum in the drying zone of vacuum drum-filters. R. WOLF A.-G. (G.P. 456,337, 25.4.25).—An airtight band revolving in the opposite direction to the drum at a variable speed is arranged above the outer surface of the filter cake so that this builds up between the periphery of the drum and the band free from drying cracks.

A. R. POWELL.

Air filters. H. WITTEMEIER (B.P. 322,967, 6.3.29).—The air is caused to flow through a series of compartments so arranged that their capacities at first increase and then decrease, whereas the communicating passages alter in the reverse manner. One form of construction comprises a pile of alternate corrugated imperforate and perforated flat, annular plates arranged round a suction pipe.

B. M. VENABLES.

Filters for air or other gases. E. L. JOSEPH (B.P. 322,986, 24.4.29).—The air is passed through narrow, sinuous spaces between vertical plates which are kept irrigated by a viscous liquid; the upper ends of the corrugated plates are spaced apart by bars, and the assembly forms the bottom of a tank for the liquid which can flow slowly down the plates through a number of grooves or holes formed for that purpose in the bars.

B. M. VENABLES.

Gas and air filter with glass-wool as filter medium. K. & T. MÖLLER GES.M.B.H. (G.P. 456,676, 29.8.25).—The filter consists of a series of pads of glass-wool sewn together so that all the fibres in each pad are parallel to one another, and every pad is at right angles to those above and below. The edge of the pads are cemented together to form a gastight joint.

A. R. POWELL.

Porous material for filters etc. G. DURRSCHMIDT (F.P. 633,018, 21.4.27).—An acid-resisting material for filters comprises a sintered mass of a granular, natural or artificial abrasive bonded with glass ground to an impalpable powder and granular or powdered fuel.

A. R. POWELL.

Apparatus for the wet purification or absorption of gases. A. MÜSER (G.P. 456,416, 22.4.26).—The apparatus comprises a vertical, rotating cylinder containing a series of parallel, horizontal discs evenly spaced along the axis and held together by a number of vertical rods so as to form a series of disintegrator-like chambers into each of which a purifying liquid is sprayed while the gas is passed upwards through the cylinder. The rotation of the drum causes the formation of a mist which collects the solid particles in the gas and absorbs any soluble constituents.

A. R. POWELL.

Complete removal of dust from air and gases. DEUTS. LUFTFILTER-BAUGES.M.B.H. (G.P. 456,612, 7.4.23).

—Gases, vapours, or different kinds of dust are mixed with the gas to be purified and the mixture is passed over oil-coated surface filters. Cement-laden air is treated with an oil spray and air containing copper oxide dust with sulphur dioxide before filtration.

A. R. POWELL.

Instrument for measuring humidity. MORLAND & IMPEY, LTD., and A. G. RENDALL (B.P. 322,878, 24.11.28).—The bulbs of a differential, wet- and dry-bulb thermometer are filled with the liquid and vapour of a substance of which the vapour pressure changes rapidly with temperature. The out-of-balance height of the indicator liquid in the bend of the U-tube will then be a measure of the drying potential of the atmosphere, and the pressure difference will be so great that a heavy indicator such as mercury may be used. The change of level may be confined substantially to one limb by forming a bulb in the other at the junction of the two liquids.

B. M. VENABLES.

Filling body for absorption towers etc. E. KÖHNEN (G.P. 455,809, 13.10.26).—The filling bodies comprise hollow spheres or polyhedra provided with several screw-shaped radial channels extending inwards and meeting in the central hollow portion.

A. R. POWELL.

Drying kiln. R. DE REYTERE (U.S.P. 1,742,586, 7.1.30. Appl., 5.7.28. Belg., 11.7.27).—See B.P. 293,765; B., 1929, 542.

Drying oven. K. J. R. ROBERTSON and A. FOWLER, ASSRS. to CARRIER ENG. CO., LTD. (U.S.P. 1,742,099, 31.12.29. Appl., 20.6.29. U.K., 18.6.28).—See B.P. 320,644; B., 1930, 1.

Centrifugal separators. D. PERRIER, ASSR. to SOC. ANON. FRANÇ. COMP. IND. DES MOTEURS À EXPLOSIONS (C.I.M.E.) (U.S.P. 1,742,095—6, 31.12.29. Appl., [A] 23.1.28, [B] 15.11.28. Ger., [A, B] 22.1.27).—See B.P. 283,963; B., 1928, 430.

Separation or recovery of gases and vapours by solid absorbents. A. GODEL, ASSR. to SOC. DE RECHERCHES ET D'EXPLOIT. PETROLIFÈRES (U.S.P. 1,742,247, 7.1.30. Appl., 23.6.26. U.K., 28.5.26).—See B.P. 270,099; B., 1927, 464.

Refrigerating machines. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of C. STEENSTRUP (B.P. 301,746, 3.12.28. U.S., 3.12.27). C. E. COOMBE (B.P. 323,464, 21.8.28).

Absorption refrigeration apparatus. PERFECTION STOVE CO. (B.P. 303,069, 27.12.28. U.S., 27.12.27).

Brine tanks and coolers [for refrigeration apparatus]. YORK SHIPLEY, LTD. FROM T. SHIPLEY (B.P. 323,261, 26.9.26).

Generator for absorption refrigerating apparatus. H. D. FITZPATRICK. FROM N.V. KODOWA REFRIGERATOR CO. (B.P. 323,666, 1.2.29).

Preventing moisture deposition on glass or other transparent surfaces in refrigerated cabinets. A. T. HAWKINS (B.P. 323,088, 15.11.28).

Atomisers. E. GAGNAN (B.P. 308,255, 8.3.29. Fr., 20.3.28).

Atomisers for very fluid liquids. E. BOUCHER (B.P. 322,927, 14.1.29).

Shaft furnaces (B.P. 322,819).—See X. **Precipitation of dust from gases** (B.P. 323,186). **Gas-purification plant** (G.P. 455,872). **Protection of metal surfaces** (U.S.P. 1,736,986—7).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Changes produced by oxidation in the pitted tracheids of certain New Zealand forest trees, and their significance in the study of coals. W. P. EVANS (Trans. N.Z. Inst., 1929, 60, 481—482).—Thin strips of wood were mounted on glass, charred by slowly passing them through the flame of a Méker burner, and examined microscopically; preliminary oxidation was alternatively accomplished by mixtures of chromic and sulphuric acids. Oxidation was found to split the secondary walls of the tracheids into narrow bands arranged spirally, which are similar to and easily confused with tertiary thickenings. The material composing the bordered pits is more resistant to oxidation, which appears to begin near the edges of the compressed pit-funnels. These points are illustrated by numerous photomicrographs.

R. H. GRIFFITH.

Adsorption as a general characteristic of coal and peat. Genesis, occurrence, gas content, weathering, spontaneous ignition, coke production, and hydrogenation. B. PENTEGOV (Pub. Univ. Extr. Orient [Vladivostok], 1929, [vii], No. 12, 28 pp.; Chem. Zentr., 1929, ii, 816—817).—The adsorptive powers of coal (a) and coke (b) increase with the age of the material. For lignite $a:b$ for oxalic acid is 1—2, for bituminous coal 0.3—1, for peat <1 , for anthracite about 1. On weathering, b falls less rapidly than a ; for otherwise equivalent conditions the values are greater for upper than for lower layers, $a:b$ being greatest where there is least metamorphosis. With increasing gas content $a:b$ falls. The relationship between the tendency towards spontaneous ignition, the coking properties, and the adsorptive power is discussed. The hydrogenation products soluble in ether are the larger in amount the greater is the adsorption value of the coal.

A. A. ELDRIDGE.

Formula for calculating the calorific value of Japanese coals. K. NAKAMURA (J. Fuel Soc. Japan, 1929, 8, 119—120).—The formulæ at present in use cannot always be satisfactorily applied to Japanese coals on account of their richness in volatile matter; the formula $K = bF + a(V - cA)$, where K is the calorific value (kg.-cal.), F is fixed carbon (%), V is volatile matter (%), A is ash (%), and a, b, c are constants, has been found by the author to be applicable to almost all such coals. Values for a vary with the class of coal, $b = 7800$, and $c = 10\%$ of the ash content.

D. G. MURDOCH.

Relative inflammability of coal dusts: a laboratory study. A. L. GOLBERT and R. V. WHEELER (Safety in Mines Res. Bd., 1929, Paper No. 56, 25 pp.).—A routine method has been developed for measuring the inflammability of coal dust by determining the amount of finely-powdered inert material required to be added to suppress the ignition of the dust. A small sample of the dust is blown by a stream of oxygen through a short, narrow tube of transparent quartz,

heated by a gas flame from a Bunsen burner, and tests are made with the addition of increasing quantities of inert material until inflammation is suppressed. It is shown that the results of this laboratory test are in good agreement with those of large-scale gallery tests of the same coal dusts. A laboratory test, capable of greater refinement than the routine test allows, was also devised to study the effects on the inflammability of coal dusts of such factors as their chemical composition and degree of fineness; only a rough relationship between the volatile matter and carbon contents and the inflammability of coal dusts has been traced (cf. B., 1929, 41), but the inflammability of coal dusts and the "reactivity," i.e., the amount of oxygen absorbed under standard conditions, of their ulmin compounds are closely related. Although the inflammability of coal dusts depends primarily on the reactivity of their ulmin compounds, the presence of abnormal quantities of non-ulmin constituents may confer an abnormal degree of inflammability because the last-named compounds are the principal oil-yielding constituents and yield their oils at a low temperature; occluded gases in coal dusts do not materially affect their inflammability, but "weathered" dusts, containing oxygen, loosely combined with the coal substance are rather more inflammable than fresh dusts.

C. B. MARSON.

Carbonisation of bituminous and brown coals in an atmosphere of gas under pressure. F. FISCHER, T. BAHR, and H. SUSTMANN (Brennstoff-Chem., 1930, 11, 1—9).—Strong, coherent, and dense semi-cokes have been produced by the carbonisation of non-caking or highly swelling coals at 600° in an autoclave wherein the pressure developed by the gas evolved was permitted to rise to a predetermined value and was maintained thereat until carbonisation was complete. Brassert coal, e.g., which gave a feebly coherent coke of apparent sp. gr. 0.67 under normal carbonising conditions, yielded an exceptionally strong coke of apparent sp. gr. 0.88 when carbonised under a gas pressure of 50 atm. The type of coke produced by this process depends on the gas pressure reached while the material is in the plastic state. To obtain the coke of maximum strength and density from some coals it was necessary to supplement the pressure due to the gas evolved by the introduction of compressed nitrogen. The required increased pressure could in some cases also be produced by the addition of water to the coal. The process was accompanied by a considerable decrease in the tar yield, e.g., to $\frac{1}{3}$ — $\frac{1}{2}$ of that obtained at atmospheric pressure, and a corresponding increase in the yields of coke and gas. Attempts to improve the yield of tar by carrying out the distillation under pressure, but in a current of nitrogen, resulted in a deterioration in the quality of the coke.

A. B. MANNING.

Brown coal: [determination of] volatile constituents. M. DOLCH and K. GIESELER (Braunkohle, 1988, 27, 608—613; Chem. Zentr., 1929, ii, 514).—The advantages of the authors' V2A-steel apparatus are discussed.

A. A. ELDRIDGE.

Determination of the volatile matter in coke. A. P. SCHACHNO and M. D. SHUKOVSKAJA (Izvestia Teploech. Inst., Moscow, 1929, No. 7, 38—50).—The

results obtained in the determination of volatile matter in coke are influenced by the age of the platinum crucible employed, and are always higher in new than in used crucibles. The greatest divergence observed was 3.54% and the mean about 1.5%. The ageing of the crucible proceeds rapidly during the first 200—300 determinations, and after 700—800 determinations the results are only about 0.5% different from those obtained with old crucibles.

T. H. POPE.

[Determination of] water content of lignite coke.

M. DOLCH, E. PÖCHMÜLLER, and H. DAVID (Braunkohle, 1929, 28, 429—434; Chem. Zentr., 1929, ii, 517).—Heating at 108° or distillation with xylene does not completely remove the water. The material is therefore boiled for a few minutes with absolute alcohol, a definite quantity of the liquid is mixed with an equal quantity of petroleum, and the separation temperature is accurately determined. The proportion of water corresponding with the value observed is determined by means of control curves.

A. A. ELDRIDGE.

The Burkheiser [gas-]purification process.

P. G. G. MOON (Gas J., 1929, 188, Inst. Gas Eng. Suppl., 45—52).—In the original process the gas from the retorts or coke ovens was cooled and the tar removed in the usual way; the virgin-liquor from the hydraulic main was worked up by customary methods and the vapours were returned to the main gas stream, so that the whole of the ammonia, hydrogen sulphide, and cyanogen were present in the gas when it entered the purifiers. The purifiers, which consisted of iron boxes, contained a specially prepared, hydrated iron oxide and removed hydrogen sulphide and cyanogen; after continued use the material was revived *in situ* by blowing a current of air through the box, the ferric sulphide being thereby oxidised and the rise in temperature sufficient to cause the liberated sulphur to burn. After leaving the purifiers the coal gas was passed through a scrubbing tower containing ammonium bisulphate and bisulphite solution, which rapidly absorbed the ammonia, forming a neutral solution of ammonium sulphate and sulphite; this solution was passed to another tower, where it came into contact with the gases from the purifiers undergoing revivification, e.g., oxides of sulphur, nitrogen, and excess air. It was here acidified again and a mixture of ammonium bisulphate and bisulphite formed. By pumping the liquor alternately through these scrubbers, ammonia and oxides of sulphur were removed until the solution became saturated, when solid ammonium sulphate and sulphite crystallised out. The chief difficulties of the process were in the revivification of the spent oxide; in the final modification both purification and revivification are carried out by wet processes. Hydrogen sulphide and cyanogen are extracted in a rotary scrubber by a wash consisting of ferric oxide and free sulphur suspended in ammonia liquor; the sludge, containing ammonium polysulphide and thiocyanate in solution and ferric sulphide and sulphur in suspension, is treated with concentrated gas liquor (ammonium sulphide), whereby the free sulphur is converted into polysulphide. The mixture is allowed to settle in a tank and the solution of polysulphide and thiocyanate drawn off and heated

to 95°, when the polysulphide is dissociated into ammonium sulphide and free sulphur. The ammonium sulphide is used again to dissolve more sulphur, the unchanged thiocyanate is treated with lime, and the resulting ammonia returned with the ammonium sulphide; the sulphur is burnt in a separate kiln to provide the necessary oxides of sulphur for the recovery of ammonia, which is carried out by the original method. The remainder of the sludge, consisting chiefly of ferric sulphide, is revived by treatment with a current of air. It is claimed that purification by this method is complete and has no deleterious effects on the quality of the gas, the plant occupies a much smaller ground space than that of the present purifiers and ammonia plant, the cost of sulphuric acid normally used for sulphate manufacture is saved, greater yields of ammonium sulphate are obtained, and difficulties associated with the disposal of sulphate plant effluents practically disappear.

C. B. MARSON.

Zinc process for extraction of ammonia and sulphuretted hydrogen from [coal] gas. J. W. COBB (Gas J., 1929, 188, Inst. Gas Eng. Suppl., 45).—The hot gas is passed through a solution of zinc sulphate maintained at 80–90°, zinc sulphide and ammonium sulphate being thereby obtained: $(\text{NH}_4)_2\text{S} + \text{ZnSO}_4 \rightarrow \text{ZnS} + (\text{NH}_4)_2\text{SO}_4$; the solution is then filtered and the ammonium sulphate recovered by evaporation. The zinc sulphide is roasted with an excess of air, the resulting gases being passed through water in which the oxidised solid product from a previous roasting is suspended; the regenerated zinc sulphate is used again. By this method it is possible to make ammonium sulphate from the sulphur in the coal without providing sulphuric acid from outside, but the slowness of the oxidation process in regenerating the zinc sulphate makes economic success doubtful. It is concluded that the succession of operations necessary, for any size of plant, would make the labour costs too heavy unless the plant were being worked on a considerable scale.

C. B. MARSON.

Production of acetylene from methane. I. Cracking under vacuum. P. K. FROLICH, A. WHITE, and H. P. DAYTON. **II. Cracking in the electric arc.** P. K. FROLICH, A. WHITE, R. R. UHRMACHER, and L. T. TURTS (Ind. Eng. Chem., 1930, 22, 20–22, 23–25).—I. The yield of acetylene obtained by cracking methane, containing 1.7–2.0% of ethane and 0.9–1% of propane, in an electrically heated quartz tube increases with temperature, and reaches a 4% conversion on the basis of methane reacting at 1150°. At high temperatures acetylene is a main primary product of cracking, but it has a marked tendency to polymerise into benzene etc.; at temperatures above 1150° decomposition into carbon and hydrogen is rapid. Reduction of pressure below atmospheric does not appreciably affect the conversion, but as the total amount of cracking was reduced the yield of acetylene (calculated on the basis of methane converted) was slightly increased. At the least time of contact employed (0.06 sec.) about 11% of the entering methane appeared in the exit gases as acetylene with only a small amount of other products. At a contact time of 0.6 sec. there was considerable

carbon deposition and polymerisation with an acetylene yield below 5%.

II. Acetylene is formed by cracking methane at the temperature of the electric arc with simultaneous formation of carbon. The acetylene yield is considerably increased and the carbon deposition decreased by diluting the methane with hydrogen. With methane and hydrogen in the ratio 1:2, 51% of the methane is converted into acetylene in passing once through the arc, corresponding to a yield of 68% of acetylene on the basis of methane cracked. Ethane behaves similarly, forming acetylene and ethylene in nearly equal quantities.

H. S. GARLICK.

Oils from English hardwood tar. M. SCHOFIELD (Ind. Eng. Chem., 1930, 22, 63–64).—A yield of 52–59% of total oils was obtained by distilling to coking point samples of English hardwood tars. The variation in the proportion of phenolic constituents (31.8–42.8%) was due to the mode of preparation of the oils or of the parent tar. The oils distilling between 175° and 290° contained the greatest amount of phenolic constituents. The disagreeable odour of the oils from a mixed hardwood tar was noticeable in varying degrees in all fractions collected. The fraction boiling between 140° and 175° contained acrylic acid and other unsaturated compounds.

H. S. GARLICK.

Analysis of cold asphalts. J. MARCUSON (Mitt. Deut. Materialprüf., 1929, Spec. No. 6, 87–88; Chem. Zentr., 1929, ii, 517).—Alcohol (96%, 25 c.c.) is gradually added to the sample (10 g.), and the separated bitumen is washed with alcohol (10 c.c.); small quantities of bitumen remaining in the alcoholic solution are precipitated with water and shaken with benzene. The bitumen and benzene solution are evaporated together and weighed, the residue being examined for tar products with hydrochloric acid and diazobenzene. From the residue on evaporation of the alcoholic extract the soaps are extracted with water, the acids are precipitated and characterised as resin or naphthenic acids; any ammonia soap is detected on addition of sodium hydroxide. Clay remains insoluble in benzene; sulphite liquor is extracted by 70% alcohol, the mass left on evaporation yielding ligninsulphonic acid (which evolves sulphur dioxide when heated) with hydrochloric acid, containing calcium, reducing Fehling's solution, and giving the carbohydrate reaction with α -naphthol and sulphuric acid.

A. A. ELDRIDGE.

Coking of brown-coal tar pitch. J. P. KOETNITZ (Brennstoff-Chem., 1930, 11, 9–10; cf. B., 1929, 1037).—In view of the increased production of pitch due to the development of continuous methods of distilling brown-coal tar in place of the older methods in which the tar is distilled to coke, the possibility of coking the pitch is examined from the economical viewpoint. The products are coke, fuel oil, motor oil, paraffin wax, and gas. Pitches of high wax content can be coked with profit, but others yield a profit only when the coke is suitable for the manufacture of electrodes or when the price of the pitch is very low.

A. B. MANNING.

Conversion of "oil tar" from low-temperature carbonisation of coal into light oils. M. J. STEPHAN

(Colliery Guardian, 1929, Dec. 6; Gas J., 1930, 189, 92—93).—An account is given of the "Semo" (Prudhomme) catalytic process for obtaining from coal etc., treated by any low-temperature carbonisation process, the maximum yield of light oils suitable for motor or aviation spirit. The hot gases from the retorts are passed at 350—400° through purifiers containing tubes charged with finely-divided metals, which effect complete removal of sulphur. Regeneration of the purifiers may be effected by passing an air current. The catalyst vessels contain tubes charged with specially treated, finely-divided nickel and cobalt, and through these the purified gas, to which has been added "mixed" gas, water-gas, and residual gas from the catalyst vessels, is rapidly passed; the reaction temperature must not be allowed to exceed 230°. The emerging gases are successively air- and water-cooled and then passed over activated carbon, to obtain the heaviest, middle-light, and very light oils, respectively, the residual gas passing to a holder. A small-scale trial carried out in France, using Durham coking coal, is described, and the products obtained by ordinary low-temperature carbonisation at 550° are compared with those obtained after catalytic treatment of the "oil-tar" vapours from the same plant operated under identical conditions. Approximately equal amounts of oil were obtained by each process, but by fractional distillation the "oil tar" yielded only 10% (by vol.) of light oil, whereas the catalysed oil gave 27.9% of aviation spirit (d_{40}^{20} 0.727) and 71% of light oil, the residue of soft pitch being soluble in benzol and suitable for varnish work. The small proportion of the light oil boiling above 300° has a high viscosity and possible lubricating properties. It is claimed that the capital and operating costs of a large-scale catalyst plant will not be excessive, and that the process will economically solve the difficulty of disposal of low-temperature carbonisation oil product. D. G. MURDOCH.

Cracking of high-boiling coal-tar acids. C. E. SENSEMAN (Ind. Eng. Chem., 1930, 22, 81—83).—High-boiling coal-tar acids were fractionated to remove material boiling below 207° and the residue was run through a cracking apparatus similar in construction to that of Dean and Jacobs (B., 1922, 534A). After each run the condensate was fractionated to remove material boiling at 207° or below. Water, benzene, xylenes, cresols, and smaller quantities of phenol and coumarone derivatives were identified as products formed in the process. Phenols of lower b.p. can be produced to the extent of 25% in a single cracking by operating at 650° with a charging rate of 3.33 c.c. per min., or at 700° with a charging rate of 6.66 c.c. per min. Recycling increases the yield to 36%. Above 700° the yield of condensable material was small, the quantity of gas and free carbon formed increasing rapidly. Pressures greater than atmospheric do not favourably affect the production of lower-boiling phenols. Condensation tests on the cracked material for the production of resins show the need of some polymerising agent, such as hexamethylenetetramine. H. S. GARLICK.

‡ **Mechanical dehydration of tar.** T. WATANABE (J. Fuel Soc. Japan, 1929, 8, 117—119).—Dehydration of low-temperature tar has been successfully effected

by means of the Sharples super-centrifuge. The water content and velocity of its separation depend to a certain extent on the free carbon content, and the centrifuge is designed so as to manipulate the separation of free carbon and dust and to effect subsequent dehydration of the tar in one stage. The cost of reducing the water content to 2% or less is sufficiently low to enable a better return to be realised from the utilisation of the tar as a wood preservative than as fuel oil. The dehydrated tar is superior to ordinary creosote oil as a wood preservative in respect of its sterilising power, waterproofing qualities, and impregnating velocity.

D. G. MURDOCH.

Clamecy process for the complete treatment of pyroligneous acid. E. CHARLES (Chim. et Ind., 1929, 22, 1059—1066).—This is a continuous process using only low-pressure steam and operating without separating tar and pyroligneous acid. The mixture is pumped into an overhead tank and all further movement is by gravity. Precautions to avoid stoppages caused by tar are described. After removal of methyl alcohol the crude liquor passes in succession through three heaters, the final residue being tar freed from acetic acid. The vapours contain traces of heavy oils which may be separated by washing with oil of low b.p., but this method adds to the steam consumption. It is, however, essential to remove the traces of alcohol and methyl acetate. This is effected in a column, the trays of which are filled to a certain level with wood-tar distillate. The vapours leaving this form a mixture of constant b.p., which is condensed, the oil being returned to the column, and the water, which contains 6—10% of oil, together with the methyl alcohol and acetate, distilled in a column with exhaust steam. The liquid from the preliminary column is passed to a second column where it is stripped of light oil, the residue consisting of 65—75% of acetic acid, 10—20% of tar oil, and the remainder water. This crude acid is redistilled continuously, giving an 80% product. This is either sold as such or further purified. Potassium permanganate is used for the removal of formic acid and empyreumatic matter. The theory of the use of the entrainer (tar distillate) in the preliminary extraction of acetic acid is discussed. The steam consumption up to the production of 80% acid is 1.43 kg./kg. of crude pyroligneous acid. The final purification requires a further 0.25 kg. The whole process can be controlled by one operator.

C. IRWIN.

Processes for the removal of phenol from ammoniacal liquor and effluent spent liquors. Report IV of Liquor Effluents Research Committee. A. PARKER (Gas J., 1929, 188, Inst. Gas Eng. Suppl., 55—60).—Typical methods for the recovery of phenols from ammoniacal or spent liquor by means of solvents such as benzol, trichloroethylene, dephenolised light tar oil, etc., by solid adsorbents such as activated carbon, by volatilisation, and by their extraction from crude coal gas at temperatures above the dew point of the gas, before liquor condenses, are described.

C. B. MARSON.

Report on visit to Germany to inspect methods adopted for recovery of phenols from ammonia liquors and kindred subjects. A. C. MONKHOUSE

(Gas J., 1929, 188, Inst. Gas Eng. Suppl., 60—64).—A detailed description of plants in operation or in course of construction, with the object of recovering phenol by solvents such as benzene is given. Methods embodying bacterial purification and electrostatic precipitation are also described. C. B. MARSON.

Decomposition of phenols with phosphoric acid. B. MÓRY (Szénkis. Közl., 1928 [1927], 2, 90—95; Chem. Zentr., 1929, ii, 817).—When coal tar is heated with phosphoric acid under pressure and with stirring, polymerisation and then decomposition of phenols into lower-boiling products take place. A. A. ELDRIDGE.

Manufacture of asphalt or coke from crude oil. L. ZUCKERMANN (Petroleum, 1929, 25, 1691—1693).—High-vacuum distillation of Boryslav crude oil gave asphalt of which 94—98% was soluble in benzene, a 2—6% yield of insoluble constituents (coke), and 5—7% of paraffin. The asphalt so produced was slightly contaminated by coke. Ordinary distillation yielded asphalt, of which 20% was insoluble in benzene (*i.e.*, coke contamination), and 12% of paraffin. The residues at high temperatures appeared to be decomposed in this form of distillation, part of the asphalt being converted into coke. It is therefore recommended to use high-vacuum distillation when asphalt is desired and ordinary crude-oil distillation or a cracking distillation of a paraffin-containing crude oil, for coke manufacture. W. S. E. CLARKE.

Employment of benzol as motor fuel. R. CHIROL (Bull. Soc. d'Encour., 1929, 128, 770—774).—Curves showing the distillation ranges of the light and heavy products of the Raschig process are given. The light product has an end-point of 180° and a distillation curve very similar to that of ordinary motor benzol, which is about mid-way between that of aviation spirit and ordinary touring petrol. Road tests show the acceleration to be more sensitive than with ordinary spirit. Similar results have been obtained by a process of steam-distillation in conjunction with the use of a dephlegmator and special reflux; 60—65% of a light product distilling between 60° and 180° was obtained. The heavy product contained very little light fractions, consisting chiefly of solvents of the benzene class. Attempts are being made to utilise other hydrocarbons, particularly naphthalene, in conjunction with benzol, either in the form of vapour or in solution therein. H. S. GARLICK.

Knocking in internal-combustion engines and the use of anti-knock fuels. A. E. DAWKINS (Proc. Soc. Chem. Ind. Victoria, Australia, 1929; Dept. Defence, Munitions Supply Bd., June, 1929, 35 pp.).—The incidence and effects of knocking, the relationship between efficiency, compression ratio, and knocking, the compression ratios of modern engines, the determination of knocking properties of fuels and the anti-knock value of dopes, their manufacture, commercial use, and mode of action are reviewed. C. B. MARSON.

Distillate yields in cracking. S. A. KISS (Ind. Eng. Chem., 1930, 22, 10—12).—The law of unimolecular reaction velocity has been applied to the calculation of distillate yields in cracking. Several formulæ have been developed covering three types of cracking, *e.g.*, cracking

with or without by-products and cracking with by-products and secondary decomposition. Other formulæ give the cracking rate as a function of temperature.

H. S. GARLICK.

Increase of colour stability of cracked benzenes. ANON. (Petroleum, 1930, 26, 56—57).—Liquid-phase cracked benzenes show a tendency to deteriorate in colour especially in the presence of certain metals, *e.g.*, copper. The colour is restored by sunlight. This discoloration, which occurs only in the "doctor-treated" product and not in the "sour" product, is due to the "doctor" solution. The addition of an acid (sulphuric, hydrochloric, or acetic) changes the reaction of benzene by the "doctor" test from basic to acidic; *e.g.*, 1% of 30% sulphuric acid causes this change, and the yellow colour is not produced on keeping even in the presence of copper. On long keeping, small quantities of carbon disulphide, acetone, etc. are formed in benzene. The effect of weak acids on benzene stored in the dark has been studied, and the colour measured in the Saybolt chromometer and compared with that of the original. Untreated benzene shows a strong tendency to assume a lemon colour, and in other cases where a longer period is necessary to produce a deeper colour acid treatment prolongs this period. W. S. E. CLARKE.

Influence of various hydrocarbons, especially of the unsaturated (olefines) and aromatic type, on the illuminating capacities of lamp oils. N. DANAILA, V. STOENESCU, and S. DINESCU (Petroleum, 1930, 26, 47—52).—Experiments are described in which the intensities and illuminating capacities of the extract obtained from Arbanasi crude oil from the fraction distilling between 160° and 272° are compared with those of certain blends of the extract with benzene hydrocarbons, naphthalene, olefines, and finally petroleum asphalt. A small Louchaire type of lamp was used for this work. Blends with benzene hydrocarbons and naphthalene showed that the presence of aromatic constituents could increase the intensity, and only in cases when this aromatic content exceeded a certain limit could an eventual decrease of illuminating power occur. The maximum aromatic content necessary to produce the best intensity is found to vary from oil to oil and from lamp to lamp, and a correct type of lamp is required for each individual case. Deficiencies in the illuminating capacities of Rumanian burning oils are not attributed to a high aromatic content, but are due rather to the actual burning and to the type of lamp used. The effect of refining Arbanasi lamp oil distillate with liquid sulphur dioxide (Edeleanu) gave an oil of less intensity in consequence of a decrease in its aromatic content. Refining with floridin and sulphuric acid, respectively, produced an oil of greater intensity than that of Arbanasi extract, but no marked difference in its illuminating capacity was noticeable. Thus acid refining removes many asphaltic and oxygen-, nitrogen-, and sulphur-containing constituents which are detrimental to burning, but the removal of which affects the content of aromatic hydrocarbons. Addition of olefinic hydrocarbons (*e.g.*, hexadecylene) even in small quantities is found to decrease the illuminating power of the oil. W. S. E. CLARKE.

Action of refining agents on pentamethylene sulphide in naphtha solution. P. BORGSTROM, R. W. BOST, and J. C. MCINTIRE (Ind. Eng. Chem., 1930, 22, 87—88).—Sulphuric acid, mercuric salts, refluxing with permanganate, and washing with sodium hypochlorite solution almost completely removes pentamethylene sulphide from naphtha solutions of varying sulphur content. It is partially removed by silica gel, treatment with aluminium trichloride, and extraction with liquid sulphur dioxide. Metallic sodium does not appreciably affect it. In behaviour it resembles the alkyl sulphides more than thiophen.

H. S. GARLICK.

Determination of the tar number in transformer oils. H. BURSTIN (Petroleum, 1929, 25, 1704).—In determining this value by the V.D.E. (German Electrotech. Assoc.) method, a maximum value of 0.1 is allowed. As the maximum amount of tar obtainable is 0.04 g., concordant results are almost impossible to obtain. Errors are introduced owing to temperature deviations during the prolonged tarring time and to the method making no allowance for thermometer corrections. Further, in the water-washing of the benzol solution, a sharp separation of the two layers is often impossible owing to the formation of an emulsion. Also the ratio of the weight of the flask used to that of the tar produced is unreasonably large. Even with extreme care duplicate analyses seldom come within the limits allowed. The author considers that further work is necessary to establish the minimum possible experimental error.

W. S. E. CLARKE.

Formolite analyses of crude oils. A. M. NASTUKOV (Petroleum, 1929, 25, 1551—1552).—Werner's results on the formolite analysis of seven Galician crude oils are discussed. The present author, in giving corrected values for the various characteristics tabulated by Werner, attributes the abnormalities in the values of i and a for Harklova oil to the higher yields of distillates in the high-vacuum distillation.

W. S. E. CLARKE.

Road materials. CLARK.—See IX. **Metallising of combustible material.** SCHOOP.—See X. **Wood-tar creosote.** SICKMAN and FISCHER.—See XX. **Treatment of spent gas liquor.** O'SHAUGHNESSY.—See XXIII.

PATENTS.

Manufacture of agglomerated fuel. M. H. HUE (B.P. 322,713, 8.4.29).—Anthracite dust or similar fuel waste, after a preliminary coking at 1200° and pulverising, is mixed with a binding substance consisting of petroleum pitch, fuel oil, and vegetable oil, and the mixture is compressed and coked at 300—400°. A. B. MANNING.

Distillation apparatus for fine-grained materials [rice husks]. G. SAUERBREY MASCHINENFABR. A.-G. (G.P. 456,891, 16.12.22).—The apparatus comprises a revolving drum internally heated by hot gases from a grate which impinge on a cylindrical core terminating in a cone with its apex pointing against the gas stream in such a way as to direct it evenly against the inside walls of the drum. The outside of the drum is provided with small, scoop-shaped, longitudinal ridges which take up the charge from the lowest part of the drum

and distribute it in a thin layer over the surface as the drum revolves. The charred product drops off the far side of the drum into an air-tight discharge hopper and the volatile products of the distillation are drawn through a hood above the drum into collecting chambers.

A. R. POWELL.

Distillation of finely-divided coal or similar material. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 307,889, 15.3.29. Ger., 15.3.28. Cf. B.P. 289,137; B., 1928, 436).—The material is spread in a layer less than 1 cm. thick over the heating surface, and is displaced relatively to the surface, e.g., by giving the latter a jerking movement, during the treatment.

A. B. MANNING.

Dry distillation of solid carbonaceous materials. W. E. TRENT (B.P. 322,348, 8.12.28).—The heat is imparted to the material by means of an endless cable which passes alternately through a heating device and through the material. The cable is trained back and forth over a number of pulleys disposed in the distillation chambers situated on opposite sides of the heating chamber. The material is introduced continuously into the distillation chambers and travels through them by gravity; it is stirred as well as heated by the moving cable. The heating chamber comprises a furnace containing a number of hollow tubes through which the different sections of the cable pass.

A. B. MANNING.

Distillation of solid carbonaceous material. L. C. KARRICK (B.P. 322,502, 7.7.28).—The material is fed continuously through a vertical retort wherein it is brought into contact with an upward current of superheated steam under high pressure. The steam and the volatile products of distillation issuing from the retort are condensed under the same pressure in a heat exchanger, in which the heat evolved is used to generate steam at a lower pressure. The retort is provided with a charging bin at the top for the material and a discharge bin at the bottom for the coke, with a feed-regulating mechanism in the lower part of the retort. The steam supply to the retort is divided before it reaches the superheater; part passes through the latter and enters the retort at about the centre, while the remainder passes directly to the retort at a lower level and becomes superheated therein as it passes through the hot coke, which at the same time is cooled. A series of such retorts may be used, working at different pressures in such a manner that the steam required for the distillation of the material in the lower-pressure retorts is derived from the heat exchanger in which the steam and volatile oils from the higher-pressure retorts are condensed.

A. B. MANNING.

Retort with rotating base for the low-temperature distillation of coal, sawdust, etc. SOC. DE FOURS À COKE ET D'ENTREPRISES INDUSTRIELLES (F.P. 632,098, 13.7.26).—A thin layer of the material in the retort is heated to a uniform temperature. If a liquid is to be distilled a constant layer thereof is maintained in the horizontal part of the retort.

A. B. MANNING.

Low-temperature distillation of fuels. A. J. A. HËRENG (B.P. 322,332, 26.11.28; cf. Roszak, B., 1928, 344).—The dried and preheated fuel is fed on to a

travelling chain grate on which it is carried through the distillation chamber. It is heated therein to the optimum temperature for low-temperature carbonisation, not above 500°, the sources of heat being: (a) the sensible heat of a current of hot inert gas which is passed downwards through the fuel, (b) the sensible heat of the chain grate itself, and/or (c) radiation from the walls of a combustion chamber. The products of distillation pass to a condensing system; the heavy, dust-laden tar which first separates is returned to the distillation chamber. The semi-coke issuing from the distillation chamber may be (i) burnt directly under a boiler, together with all or part of the gas, if desired; (ii) pulverised to give powdered fuel; (iii) separated in a compact form suitable for use as a domestic fuel; or (iv) fed directly to a generator for the production of water-gas. To aid in the agglomeration of the fuel when a compact product is desired, a second chain grate may be mounted within the chamber in such a way as to rest on and compress the fuel during carbonisation.

A. B. MANNING.

Distillation and condensation of solid and liquid colloidal mixtures. J. MÜLLER (G.P. 457,220, 1.4.25).—The preheated material, *e.g.*, powdered coal, oil shale, coal tar, is blown into the top of a tower by means of a stream of hot gas, whereby the more volatile products are vaporised and the less volatile disintegrated into a mist. The gases pass down through a series of filters, each filter consisting of a number of inclined steps and each succeeding filter being kept at a lower temperature than that above it by suitable cooling means. In this way fractional condensation of the products of distillation is effected. Means are provided for continuous draining of the filters and for heating the waste gases for utilisation again in the spraying device.

A. R. POWELL.

Recovery of valuable products from varieties of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,489, 3.9.28).—Coals, tars, etc., which are to be subjected to destructive hydrogenation under pressure, are preheated to not above 350°, and are mixed with part of the hydrogenating gas, also preheated to the same temperature. The mixture is then further heated to bring it to the reaction temperature (about 430°) prior to introducing it into the reaction chamber, wherein it is brought into contact with the remainder of the preheated hydrogenating gas. The reaction being exothermic, no separate supply of heat to the reaction chamber is necessary. If desired, the product may be recirculated through the apparatus. The process may be carried out in the presence of catalysts, preferably those immune from poisoning by sulphur. Those parts of the apparatus which come into contact with gaseous or vaporised substances at high temperatures are constructed of, or coated with, highly alloyed steels, aluminium, or other chemically resistant materials.

A. B. MANNING.

Metallurgical charcoal. G. DE KORFF and G. JAKOVA-MERTURI (F.P. 632,623, 11.4.27).—Dry, hard wood is carbonised in closed retorts at a temperature not exceeding 250° and the charcoal obtained is saturated with a hot solution of sodium acetate or borate,

or is ground with the solid salts. Lignite may be treated in the same way. The product is especially suitable for carburising iron in the manufacture of steel.

A. R. POWELL.

Activating and revivifying spent activated carbon. A. E. WHITE. From J. J. NAUGLE (B.P. 322,185, 25.7.28).—A relatively shallow layer of the material contained in an elongated, horizontal trough is heated at about 450° in the presence of air or other activating gas, and at the same time is subjected to a to-and-fro agitating action of such a character as to propel the charge along the trough and expose all the particles of the material to the action of the oxidising gases. The heating may be effected by passing an electric current through the material. The process is particularly applicable to the calcined lignin residues from the manufacture of wood pulp by the soda process.

A. B. MANNING.

Apparatus for generating water-gas. J. PINTSCH A.-G. (B.P. 299,421, 15.10.28. Ger., 26.10.27).—Pulverulent fuel is injected down a vertical reaction pipe which is traversed also by a current of steam and water-gas preheated to a sufficiently high temperature to bring about rapid interaction of the fuel and steam. Regeneration is effected in two chambers arranged on either side of the reaction pipe; the chequer work of one chamber supplies heat to the water-gas and steam entering at the bottom, while the other chamber is being heated by the combustion of part of the water-gas produced. After traversing one or more ash-separating chambers the hot current of gas is brought into contact with the raw powdered fuel in a drying and/or distilling apparatus, in which the fuel is pre-treated before being charged into the generator itself. Part of the water-gas is recirculated through the apparatus. When brown coal is used the steam evolved in the drying process is utilised in the generation of the water-gas, the circulatory current then being passed through a cooling apparatus in order to regulate its steam content.

A. B. MANNING.

Manufacture of gases and grates therefor. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,553, 14.9.28. Addn. to B.P. 214,544; B., 1924, 549).—In order to prevent the powdered coal falling through the grate of the apparatus described in the prior patent, the grate bars are so shaped that the direction of flow of the gasifying agent is altered at least once through an angle of about 180° on passing through the grate. To minimise distortion of the grate bars the parts thereof which are more directly exposed to the high temperature are inlaid with a refractory material or a sheet of iron-nickel-chromium alloy with a substratum of asbestos.

A. B. MANNING.

Manufacture of gases [from the products of destructive hydrogenation of coal]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 322,734, 3.9.28).—The constituents of the condensate obtained by subjecting the gases and vapours leaving the reaction chamber to condensation under pressure are separated by fractionally releasing the pressure. The gas first evolved under these conditions consists principally of hydrogen and can be used again in the hydrogenation process, if necessary after removal of the hydrocarbons

by thermal decomposition. The gases subsequently released contain paraffin hydrocarbons, *e.g.*, propane, butane, and pentane. By repeated compression and fractional release of pressure a more complete separation of these gases is effected. A. B. MANNING.

Regulation of gas-producer plants. MOTORENFABR. DEUTZ A.-G. (B.P. 302,607, 17.12.28. Ger., 16.12.27).—Gas producers having two combustion zones and variable loading are provided with automatic means for regulating the air supplies to the upper and lower fires according to the temperatures and/or pressures at certain places in the producer. Thus a thermo-electric element built into the combustion zone may operate a relay which, in turn, controls an electromagnet acting on a throttling damper in one of the air supply pipes. Two such elements similarly controlling the two air supply pipes may be used to maintain a constant difference of temperature between the corresponding positions in the producer; or the two air supply pipes may be connected to a pressure regulator which actuates throttling valves in the pipes when a given pressure difference or pressure ratio is exceeded in such a way as to restore the required pressure difference or ratio. Where a number of gas producers are used to feed the same gas line, similar methods of control may be used to maintain the required distribution of air among the producers. A. B. MANNING.

Production of producer gas with a low sulphur content for use in open-hearth furnaces. CONCORDIA BERGBAU A.-G., and J. I. BRONN (G.P. 455,595, 11.7.24).—The producer is charged with a mixture of uncoked fuel and a small proportion of lime and/or an alkali hydroxide or carbonate, ferric oxide, or finely-divided iron. A. R. POWELL.

Manufacture of gas of the nature of producer gas from liquid fuel. W. W. TRIGGS. From WILLIAMS OIL-O-MATIC HEATING CORP. (B.P. 322,733, 10.8.28).—Oil and water are introduced in the form of a fine spray, together with a limited supply of air, into a chamber containing refractory material which is maintained at incandescence during normal working. The excess oil and the water vapour interact in passing over or through the incandescent material to form a fixed gas, which may be used for any desired heating purpose. The air supply to the apparatus is adjusted automatically by a thermostatic device, *e.g.*, a bimetallic strip, controlled by the temperature of the chamber. The atomising pump is driven by an electric motor which also is automatically controlled by a thermostat. A. B. MANNING.

Extraction of gases from and gasification of fuels. T. LICHTENBERGER, L. KAISER, and F. MEYER (B.P. 322,959, 20.2.29).—Fuels are distilled and gasified by introducing them, in a more or less coarsely-divided form, together with the steam necessary for the production of water-gas, into a bath of a molten salt, *e.g.*, sodium chloride. The apparatus consists of two chambers, communicating only below the level of the molten salt. One chamber forms a reverberatory furnace in which the salt is heated; the other forms the gasification chamber. A. B. MANNING.

Removal of benzol from coal gas. SOC. DES ÉTABL. BARBET (F.P. 632,061, 7.7.26).—The gas is scrubbed under a definite pressure in two cast-iron columns provided with plates presenting a large area over which the scrubbing liquid can trickle. A. B. MANNING.

Extraction of bituminous materials by means of solvents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,749, 6.9.28).—Oils and bitumens are extracted from bituminous materials, *e.g.*, the residues obtained in the destructive hydrogenation of coal, by using as a solvent the mixture of hydrocarbons boiling between 80° and 150°, and preferably having a high content of aromatic compounds, obtained by the destructive hydrogenation or cracking of coal, tar, mineral oil, etc. A. B. MANNING.

Bituminous emulsions and their use in the manufacture of road-making and building materials. Methods of applying bitumen to a concrete foundation. COLAS PRODUCTS, LTD., L. G. GABRIEL, and J. F. BLOTT (B.P. 322,792—3, 13.9.28).—(A) An aqueous bituminous emulsion suitable for admixture with concrete etc. is produced by intimately mixing, preferably in a colloid mill, molten or liquid bituminous material with an aqueous or acid solution of gelatin or glue. The proportion of gelatin used does not exceed 5% by wt. of the bitumen. A bitumen-impregnated concrete suitable for road-making is made by mixing the emulsion with about 9 times its weight of concrete or other aggregate. (B) To a layer of concrete, still in the plastic condition, a layer of bitumen-impregnated concrete and then a coating of the bitumen emulsion are applied. A. B. MANNING.

Preparation of emulsions [of tar etc.]. H. BASSET and V. SZIDON (F.P. 630,168, 4.3.27).—A mixture of asphalt, tar, or tar oil, etc. with tall oil is intimately mixed, *e.g.*, by means of a colloid mill, with a solution containing sufficient caustic alkali to saponify the tall oil, and a vegetable mucilage. A. B. MANNING.

Bituminous compositions. T. F. KING (B.P. 322,431, 3.8. and 17.10.28).—A mixture containing, approx., chalk 67%, alum 1%, pitch 16%, and coal tar or similar bituminous material 16% is used (a) for spraying roads, or (b) as a binding material, when admixed with a suitable aggregate, for the formation of roads or the manufacture of blocks for street paving etc. A. B. MANNING.

Working-up pitches and asphalts. H. SUIDA (Austr. P. 108,697, 14.3.24).—The material is treated continuously in a combined carbonising and gas-generating apparatus, in one part of which it is coked while in the other part the coke is transformed into producer gas. The hot producer gas provides the heat necessary for the carbonising process. A. B. MANNING.

Treatment of [mineral] oil. Oil stills [for petroleum etc.]. FOSTER WHEELER, LTD. From FOSTER WHEELER CORP. (B.P. 318,700—1, 3.7.28).—(A) Lubricating oil fractions with small overlapping viscosities are produced by passing a stock oil through a tubular oil heater, where it is rapidly heated to the maximum temperature with the minimum of cracking, and then fractionally distilling and condensing it in a tower in

which the condensates formed in the upper portion from vapours liberated in the lower portion are subjected to intermittent contact with steam and with vapours passing up through the tower, the different fractions being separately withdrawn from the tower at suitable levels. (b) The heating apparatus comprises a furnace chamber separated by a bridge wall, which has an opening over the top extending across the entire apparatus to avoid excessive turbulence of the hot gases, from a heat-absorbing chamber with a heating surface therein receiving heat by convection through hot gases from the furnace chamber, and a second heat-absorbing surface adjacent to the roof of the furnace chamber, consisting of superimposed, staggered, bare tubes, subjected to unrestricted and direct radiation from the fire. Oil passes through the tubes in the heat-absorbing chamber and thence through tubes in the furnace chamber, the heating surface of which is such that sufficient heat is abstracted to prevent overheating of the oil while it passes through the heat-absorbing chamber, but is so designed that the oil is brought to cracking temperature and kept there.

H. S. GARLICK.

Distilling oils by means of a metal bath. S. SEELIG (B.P. 295,935, 18.8.28. Ger., 20.8.27).—Oil is supplied to a molten metal bath through a coil the convolutions of which are in contact within a tube used for separating the oil from the wall of the reaction vessel. The tube is spaced apart from the interior wall of the reaction vessel and extends from the upper part thereof to nearly the bottom so as to form an air space above the surface of the molten metal between the tube and the inner wall, the oil rising within the tube.

H. S. GARLICK.

Distillation process [for liquid hydrocarbons]. J. C. RYDER, Assr. to PETROLEUM DERIVATIVES, INC. (U.S.P. 1,727,380, 10.9.29. Appl., 5.1.24).—Liquid hydrocarbons are made to flow continuously in a thin stream over a heated surface while under high vacuum.

H. S. GARLICK.

Production of low b.p. oils etc. by destructive hydrogenation of coals, oils, etc. I. G. FARBENIND. A.-G. (B.P. 287,855, 26.3.28. Ger., 26.3.27).—Surrounding the contact chamber and isolated from the shell of the reaction apparatus is a chamber into which hydrogenating gas, preheated to 400°, is admitted under pressure and led in spirals from below upwards, subsequently passing into the reaction chamber through an electric heating device before coming into contact with the materials under treatment which have already been raised to reaction temperature by other means. The reaction products enter a second annular chamber provided with spiral guides and arranged concentrically with the reaction chamber, or may be led downwards through pipes situated inside the reaction chamber in order to effect heat exchange.

H. S. GARLICK.

Purification, hydrogenation, and desulphurisation of liquid or gaseous hydrocarbons as, e.g., oils or other liquid or gaseous products of carbonisation etc. H. ROSTIN (B.P. 286,255, 1.3.28. Ger., 1.3.27).—Hydrogen or other reducing gases at 200–300° are conducted through a vertical tube filled with iron oxide in the form of rust or iron ore, preferably granu-

lated. When reduction is complete the products to be treated are led together with hydrogen sulphide into the bottom of the tube, the reduced iron decomposing the hydrogen sulphide and the nascent hydrogen reacting with the materials under treatment. When the iron is sulphurised, the flow of material under treatment and of hydrogen sulphide is stopped and desulphurisation accomplished by introducing hydrogen or gases containing it at an elevated temperature into the top of the tube.

H. S. GARLICK.

Manufacture of liquid oxidation products from hydrocarbon materials. C. ARNOLD. From STANDARD OIL DEVELOPMENT Co. (B.P. 321,494, 7.9.28).—Aliphatic alcohols, aldehydes, and acids are produced from normally gaseous or low b.p. hydrocarbons such as occur in natural or refinery gas, by adding to the hydrocarbon gas an oxidising gas containing free oxygen in concentration not exceeding 15% and which may be less than 5%. The gaseous mixture is passed through a reaction chamber under a substantially uniform pressure exceeding 500 lb./in.² (preferably 1500–3500 lb./in.²), and at an elevated temperature predetermined in relation to the selected catalyst contained therein. The mixture discharged from the reaction chamber is cooled and the oxidation products are separated from the unoxidised hydrocarbons, by scrubbing with a solvent consisting of water and methyl alcohol, which are returned with fresh oxidising gas to the reaction chamber. By such means high yields of oxidation products, chiefly methyl alcohol, can be obtained from methane; similarly, propane and butane give high yields of oxidation products consisting essentially of methyl, ethyl, and propyl alcohols.

H. S. GARLICK.

Treatment of hydrocarbon material. T. W. S. ROBINSON and C. DE GANAHL (B.P. 321,473, 15.8.28).—The production of coke, tar, or solid end-products from the material issuing in the hot state from oil-cracking devices is prevented by bringing such hydrocarbon material into direct contact with cooling liquids under conditions adapted substantially to preclude contact of the hot material with the walls of the cooling chamber until its temperature has been reduced below that at which the separation of coke or tarry matter will be promoted by such contact, which is normally about 430°.

H. S. GARLICK.

Cracking of oils. R. H. CROZIER (B.P. 319,224, 18.5.28).—Coal, shale, etc. are distilled in a retort and the vapours drawn off at different levels through mains arranged so that incoming vapours bubble through the residual fractions contained therein, the fractions being carried from main to main and the heaviest fractions being passed back into the retort to be cracked in contact with the residual coke therein. Pressure-equalising means are provided to maintain the liquid level in the mains, and at the lower end of the retort one or more flues are arranged to act as bracing members and to provide for the introduction of one or more gas burners operated from the gas produced by the cracking operation.

H. S. GARLICK.

Conversion of hydrocarbons of high b.p. into those of low b.p. I. G. FARBENIND. A.-G. (B.P. 296,700, 27.7.28. Ger., 5.9.27).—Hydrocarbons of high

b.p. are subjected to a continuous cracking process under pressure, with or without hydrogen, and for a short time so that carbonaceous deposits are formed with the minimum production of gas. The pressure on the cracked products is then reduced, in order to recover the products of low b.p. which may have been produced, prior to treating the residues in a second stage to destructive hydrogenation under pressure with added hydrogen in the presence of catalysts.

H. S. GARLICK.

Manufacture of hydrocarbons of high b.p. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 320,846, 18.5.28).—Mixtures of hydrocarbons of medium mol. wt. and containing appreciable amounts of aromatic constituents are converted into products of high b.p. and increased viscosity by subjecting them to the action of a halogen or a halogen-exchanging halogen compound with simultaneous or subsequent addition of an element of groups III—VIII capable of existence in the metallic form, or a compound thereof, or mixtures of these substances, as catalysts, at below 150° (preferably below 100°). In some cases the addition of small quantities of elements of groups I and II to the above catalysts is advantageous. The resultant products are subjected to further condensation at the same temperature in the presence of the catalysts, preferably after the removal of any halogen or halogen compound. H. S. GARLICK.

Cracking of [hydrocarbon] oil. E. W. ISOM, E. C. HERTHEL, and H. L. PELZER, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,727,707, 10.9.29. Appl., 17.11.24).—Heavy oil is reduced to light hydrocarbons and coke by heating to cracking temperature under pressure and removing the cracked hydrocarbons as formed, by injecting into the oil hydrocarbon vapours at a temperature above the cracking point, then reducing the pressure while still continuing the feed of vapours, and finally injecting a gaseous medium at a temperature above the vaporising temperature of the heaviest hydrocarbon remaining in the residue. H. S. GARLICK.

Cracking of [hydrocarbon] oil. STANDARD OIL DEVELOPMENT Co., ASSECS. of A. C. SPENCER and E. W. LUSTER (B.P. 295,399, 11.8.28. U.S., 11.8.27).—Oil is heated to cracking temperature in a coil and passed to a drum where conversion is allowed to take place. The hot, cracked oil product is passed to a tar separator from which the vapours enter the first fractionating tower. A portion of the condensate is removed, reheated in a separate heater without decomposition, and returned to the first fractionating tower, the vapours from which pass to a second fractionating tower for recovery of gasoline. H. S. GARLICK.

Distillation and cracking of [hydrocarbon] oil. C. J. PRATT (B.P. 293,829, 2.7.28. U.S., 13.7.27).—Condensate from a fractionating tower is heated to cracking temperature in a treating furnace while a stream of raw hydrocarbons is heated to a relatively low temperature in a pretreating furnace. The hot vapours are then blended in a mixing vessel or preconditioner, into which the two streams of hot oil are admitted separately through opposed inlets separated by baffle plates to ensure intimate contact. The uncondensed vapours and the heavy hydrocarbon end-products pass

out from the preconditioner separately, the former going to a fractionating tower. The whole process is effected at substantially atmospheric pressure.

H. S. GARLICK.

Cracking or distillation of hydrocarbon oils. H. MAGNUS (B.P. 307,511, 9.3.29. Ger., 10.3.28).—Oil is treated in cracking zones in which overstepping of a determined temperature is prevented by a device comprising a thermostat acting through relays and influenced by the temperature within the heating zone. This device also controls the fresh oil supplied to the heating zone or to a coil arranged in the reaction vessel in such manner that at a predetermined temperature the quantity of oil flowing through is increased or the temperature of the inflowing medium is lowered.

H. S. GARLICK.

Conversion of hydrocarbon oils. G. EGLOFF and J. C. MORRELL, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,733,656, 29.10.29. Appl., 24.7.22. Renewed 18.3.29).—Oil is vaporised under pressure in the presence of a metallic oxide, and the heavier fractions are condensed, injected with an oxidising gas, and returned to the oil undergoing treatment. H. S. GARLICK.

Regeneration of alkaline purifying beds [used in treatment of petroleum vapours]. R. C. WHEELER and P. W. PRUTZMAN (U.S.P. 1,728,156, 10.9.29. Appl., 24.6.27).—Steam is mixed with the oil vapours passing through packs of solid caustic soda used for their treatment, in sufficient quantity only to disengage the reaction products formed thereon and permit their removal by continuous drainage. H. S. GARLICK.

Treatment of casing-head gasoline. E. C. HERTHEL, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,733,800, 29.10.29. Appl., 13.6.27).—The gas and vapour mixture, substantially water-free, is treated with solid cupric chloride before liquefaction. H. S. GARLICK.

Manufacture of insulating oils. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 321,187, 1.8.28).—The fraction, b.p. above 160°/12–25 mm., from the condensation products of olefines with naphthalene or an oxygen-free naphthalene derivative, consisting of a mixture of polyalkylated naphthalenes, is subjected to a customary mineral oil refining process.

H. S. GARLICK.

Manufacture of lubricating and insulating oils. I. G. FARBERIND. A.-G. (B.P. 297,798, 8.9.28. Ger., 28.9.27).—Hydrocarbon oils of any kind are subjected to the action of high-frequency electric currents in the presence of added, powdered, brown or mineral coal or finely-divided wood, charcoal, or colloidal or pulverulent metals or metal oxides, or coal ash, etc. dispersed in the oils under treatment. H. S. GARLICK.

Purification of transformer and switch oils. A.-G. A. HERING (Austr. P. 108,438, 9.8.26).—The oils are circulated in a closed apparatus in which they are heated *in vacuo* in order to bring about separation of the sludge and evaporation and removal of any precipitated moisture. The apparatus may be heated electrically, but to avoid overheating of the oil a vapour-jacket is interposed between the source of heat and the container. The heated oil is further purified by centri-

fuging and is then sprayed into the transformer by means of a pump, in such a way as to dislodge any sludge deposited therein.

A. B. MANNING.

Treatment of transformer, switch, and turbine oils prepared from petroleum. A.E.G.-UNION ELEKTRIZITÄTS-GES., Assecs. of H. VON DER HEYDEN and K. TYPKE (Austr. P. 108,434, 8.4.26. Ger., 14.4.25).—Shortly before use the oils are treated with reducing agents in order to remove any oxygen dissolved therein and to render inactive any oxidation products which might act as oxygen carriers.

A. B. MANNING.

Preparation or conversion of oils. R. FEIGE (F.P. 632,293, 6.4.27. Ger., 19.5.26).—Solid, liquid, or gaseous fuels are submitted to the action of a high-tension electric discharge. The solid fuels are finely ground and treated in admixture with gases or vapours; oils are vaporised and preferably mixed with catalytically acting materials in the form of dust, cloud, or gas before their introduction into the reaction chamber. The electrodes are wholly or partly constructed of, or coated with, catalytically acting materials, *e.g.*, tin. The reacting materials are preferably preheated and the reaction is carried out under pressure.

A. B. MANNING.

Refining of oil. T. C. HEISIG, Assr. to GALENA-SIGNAL OIL Co. (U.S.P. 1,728,059, 10.9.29. Appl., 19.5.26).—Heavy, viscous asphalt- or naphthene-base oils are diluted with naphtha, and 1% of 93% sulphuric acid is added to absorb water. The acid water is removed and the relatively dry, diluted oil is agitated with about 4% of 98% sulphuric acid. The sludge is drawn off, the diluted stock with its contained acid and acid compounds mixed with an absorbent, the diluent distilled off while subjecting the mixture to violent agitation in the presence of heat and steam, and the hot oil is filtered.

H. S. GARLICK.

Recovery of oils from industrial residues. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,891, 23.7.28 and 15.3.29).—The residues (acid resins and acid tars) from the treatment of hydrocarbons with metal halides or their refining with sulphuric acid are dry-distilled at above 360° in the presence of substances having a neutralising or refining action, with the simultaneous introduction of inert gases or vapours. It is preferable to employ residues of different kinds which contain at least one residue containing metal halides, or one acid residue, or both.

H. S. GARLICK.

Manufacture of a ceresin-like wax. G. L. WENDT and C. BANTA, Assrs. to STANDARD OIL Co. (U.S.P. 1,735,555, 12.11.29. Appl., 2.3.23).—Uncracked crude oil residuum is diluted with a mixture of a wax solvent and a miscible non-solvent liquid and cooled; the precipitated wax is separated.

H. S. GARLICK.

Motor fuel. RÜTGERSWERKE-A.-G., and L. KAHL (F.P. 632,193, 5.4.27. Ger., 28.5.26).—The fuel consists of a liquid eutectic mixture of fuels, *e.g.*, naphthalene, phenanthrene, acenaphthene, diphenyl oxide, and fluorene, which are themselves solid at the ordinary temperature, if desired, in solution in a liquid hydrocarbon.

A. B. MANNING.

Motor fuel. SOC. ANON. PROGIL (F.P. 631,560, 14.6.26).—A light or heavy fuel is mixed with acetaldehyde, paraldehyde, or metaldehyde.

A. B. MANNING.

Fuel for internal-combustion engines etc. J. G. AARTS (Austr. P. 108,678, 28.12.25. Ger., 13.5.25).—Activated carbon produced by the cracking of carbonaceous substances is incorporated with combustible fuels, *e.g.*, hydrogen, hydrocarbons, or mixtures thereof.

A. B. MANNING.

Homogeneous product from mineral oil and alcohol. W. HUESSY (F.P. 630,589, 9.3.27. Ger., 10.3. and 8.9.26).—In addition to the compound referred to in B.P. 267,530 (B., 1928, 843), oils (especially kerosene) are mixed with 10–20% of methyl or ethyl alcohol, a little water, and 1–5% (calc. on wt. of alcohol) of oxalic, formic, or hydrochloric acid. The vaporised mixture is passed at 400–500° over metal, *e.g.*, zinc, lead, or lead-nickel alloy, to which calcium oxide or hydroxide, wood charcoal, or silicates have been added, and then condensed. The product is sulphur-free and when mixed with benzene, gasoline, etc. forms a motor spirit or emulsifying agent.

W. J. BOYD.

Bleaching of paraffins etc. CHEM. WERKE KIRCHHOFF & NEIRATH GES.M.B.H. (G.P. 455,524, 10.2.25).—For bleaching of mineral waxes and oils, persulphate and sulphuric acid are employed at high temperatures.

W. J. BOYD.

Emulsifying agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,239, 7.8.28).—The products of oxidation of waxy materials such as hydrocarbons of high mol. wt. or natural waxes of animal, vegetable, or mineral origin, especially those of an acidic nature, are treated with 20–40% of their weight of chlorine at below 100°, either alone or in conjunction with fatty acids or resin acids; the resultant chlorination products are then treated with a basic agent.

H. S. GARLICK.

Working-up of oil chalks etc. DEUTS. PETROLEUM A.-G., Assecs. of E. H. RIESENFELD, G. BANDTE, and H. PFLUG (G.P. 455,306, 20.10.25).—The material is heated at 150–160° in a current of air diluted with an indifferent gas. Mineral oils which are difficultly convertible into asphalt may be mixed with a mineral substance and treated as above. The products when ground and mixed with asphalts or mineral oil residues form suitable road-construction materials.

A. B. MANNING.

Treatment of materials with binders in the briquetting of such materials. R. LESSING (U.S.P. 1,741,193, 31.12.29. Appl., 6.10.27. U.K., 3.11.26).—See B.P. 286,336; B., 1928, 356.

Purification of gases from organically-combined sulphur. F. FISCHER (U.S.P. 1,741,834, 31.12.29. Appl., 31.10.27. Ger., 24.12.26).—See B.P. 282,634; B., 1929, 233.

Discontinuous distillation of tars. L. KAHL (U.S.P. 1,742,258, 7.1.30. Appl., 26.11.23. Ger., 20.12.22).—See B.P. 208,691; B., 1925, 347.

Conversion of hydrocarbons and mineral oils having a high b.p. into those having a lower b.p.

II. WOLF, Assr. to CARBUROL A.-G. (U.S.P. 1,742,888, 7.1.30. Appl., 28.4.24. Ger., 1.5.23).—See B.P. 215,336; B., 1926, 263.

Purification of benzol, petrol, and the like. K. COX and P. J. McDERMOTT, Assrs. to REFINERS, LTD. (U.S.P. 1,742,343, 7.1.30. Appl., 8.5.28. U.K., 8.12.27).—See B.P. 303,991; B., 1929, 198.

Portable gas producers. J. GOHIN (B.P. 323,147, 8.2.29).

III.—ORGANIC INTERMEDIATES.

Production [from lemons] of citric acid in Brazil. J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 163).—The economic practicability of producing citric acid from lemons is discussed, certain types of which contain 2·15% of the acid. H. F. GILLBE.

Methylation of phenol by dimethyl sulphate. H. F. LEWIS, S. SHAFFER, W. TRIESCHMANN, and H. COGAN (Ind. Eng. Chem., 1930, 22, 31—36).—The alterations in the yields of anisole consequent on changes in relative proportions of reactants or additions of reagents etc. have been studied. In acid or neutral aqueous solution or in alcoholic solution only traces of anisole are obtained, the methyl sulphate being mainly hydrolysed. In the presence of a large excess of water the first methyl group of methyl sulphate reacts rapidly with the phenolic hydroxyl; the sodium methyl sulphate also formed then reacts slowly with more sodium phenoxide. If the water concentration be reduced the second methyl group can be largely used in the formation of anisole; thus 90% of the methyl in methyl sulphate can be used by treating 0·2 mol. of phenol with 0·1 mol. of methyl sulphate, 0·3 mol. of sodium hydroxide, and 0·2 mol. of water for 5 hrs. at 100°. R. J. W. LE FÈVRE.

Syntheses in the diphenyl series. R. L. JENKINS, R. McCULLOUGH, and C. F. BOOTH (Ind. Eng. Chem., 1930, 22, 31—34).—Details are given for the large-scale preparation from technical diphenyl (97%, m.p. 68°) of 2- and 4-chloro-, 2- and 4-nitro-, and 2- and 4-amino-diphenyls. The methods employed are substantially those in the literature, although the authors claim novelty for the use of iron as catalyst in the mono-chlorination, for the use of mixed acids in the nitration, and of iron and water alone for the preparation of the two aminodiphenyls. The following corrected f.p., m.p., and b.p. are recorded for the above derivatives in the order named: 31·2°, 32·2°, 273·7—273·8°/738 mm.; 76·8°, 77·2°, 291·2°/745 mm.; 36·9°, 37·2°, 201—201·3°/30 mm.; 113·8°, 113·7°, 224·1—223·7°/30 mm.; 48·7°, 49·3°, 182°/30 mm.; 52·6°, 54·1°, 211·0—211·2°/30 mm. R. J. W. LE FÈVRE.

Pyroligneous acid. CHARLES. **Decomposition of phenols with phosphoric acid.** MORY.—See II. **Formaldehyde.** SCHWYZER.—See XX.

PATENTS.

Chlorination of hydrocarbons. E. E. AYRES, JUN. (B.P. 320,406, 12.7.28).—In the chlorination of hydrocarbons (especially pentanes) explosion is avoided by introducing the chlorine at a temperature at which the rate of reaction is low (e.g., 150°) into a current of the

hydrocarbon vapour moving so rapidly (e.g., 50 ft. per sec. in a tube of 8 in. diam.) as to prevent ignition or persistence of ignition at the point of entry. Conversely, the hydrocarbon may be added to a rapid stream of chlorine. C. HOLLINS.

Manufacture of halogen derivatives of organic compounds. M. POLANYI and S. VON BOGDANDY (B.P. 289,795, 30.4.28. Ger., 29.4.27).—Sodium is used to initiate the reaction between methane (etc.) and halogen. The mixed gases may be introduced at the base of a vessel where they meet a much slower current of methane charged with sodium vapour at 265°, the ratio of flow being, e.g., 15:1; the reaction vessel is cooled to maintain the gases at about 200°. The exit gases contain 5% of methyl chloride, 90% of methane, and 5% of hydrogen chloride. C. HOLLINS.

Manufacture of primary alcohols [from alkylene oxides]. A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 320,424, 17.7.28).—An alkylene oxide is reduced in vapour form with hydrogen in presence of a catalyst such as reduced nickel containing alkali. Propylene oxide gives *n*-propyl alcohol, mixed α - and β -butylene oxides yield *sec*- and *n*-butyl alcohols, and from styrene oxide β -phenylethyl alcohol is obtained. C. HOLLINS.

Purification of organic liquids [aliphatic alcohols]. I. G. FARBENIND. A.-G., Asscs. of C. MÜLLER, W. FRANKENBURGER, and F. GRASSNER (G.P. 452,066, 31.8.24).—For the removal of traces of iron carbonyl and aldehydes from synthetic methyl or isobutyl alcohol, the liquid is passed through tubes surrounding a mercury-vapour lamp or other source of light of short wave-length and a current of gas containing oxygen or, preferably, ozone is passed through the liquid countercurrent to the flow. The impurities are converted into insoluble compounds or into acids which are readily removed by subsequent distillation of the alcohol. A. R. POWELL.

Manufacture of condensation products from olefines and hydrocarbons of the naphthalene series. R. MICHEL, Assr. to I. G. FARBENIND. A.-G. (Re-issue 17,548, 31.12.29, of U.S.P. 1,667,214, 21.6.27).—See B., 1928, 410.

Manufacture of ethylated naphthalenes. R. MICHEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,741,472, 31.12.29. Appl., 25.1.27. Ger., 4.2.26).—See B.P. 265,601; B., 1928, 740.

Preparation of tetrahydronaphthastyrils. K. SCHIRMACHER, H. SCHLICHENMAIER, and W. KROSS, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,742,322, 7.1.30. Appl., 21.11.27. Ger., 25.11.26).—See B.P. 281,257; B., 1928, 846.

Preparation of benzanthronecarboxylic acids. G. KRÄNZLEIN and H. VOLLMANN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,740,771, 24.12.29. Appl., 9.9.27. Ger., 20.9.26).—See B.P. 277,670; B., 1928, 922.

Oxidation products from hydrocarbons (B.P. 321,494). **Insulating oils** (B.P. 321,187). **Emulsifying agents** (B.P. 321,239).—See II. **Pure acids** (U.S.P. 1,733,152).—See VII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,268, 19.11.28).—Pyranthrone is dibrominated in aqueous suspension, preferably in presence of alkali, to give a vat dye redder than the known dibromopyranthrone. The bromine probably enters the mesonaphthalene ring.

C. HOLLINS.

Manufacture of chromed complexes of chromable azo dyes. COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ÉTABL. KUHLMANN (B.P. 307,871, 9.8.28. Fr., 15.3.28).—Azo dyes are pre-chromed by boiling with chromium salts of sulphonated aromatic hydrocarbons, e.g., benzenesulphonic acid, 1:5-naphthalenedisulphonic acid.

C. HOLLINS.

Manufacture of polyazo [green] dyes [for cotton]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 320,030, 1.5.28).—Tris- and tetrakis-azo dyes giving green shades on cotton are obtained by coupling a diazotised amino- or aminoazo-compound with an amino-ether or -thioether as middle component, rediazotising and coupling with an aminobenzoylaminobenzoyl or aminobenzoylaminobenzoylaminobenzoyl derivative of a 1:8-aminonaphthol (in which substituents may be present in any of the nuclei, and in which the $\cdot\text{CO}\cdot\text{NH}\cdot$ groups may be replaced by $\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}\cdot$ groups), rediazotising and finally coupling with a pyrazolone, an acetoacetic arylamide, 2-methylindole, a sulphazone, a 2:4-dihydroxyquinoline, or a salicylic acid. The last coupling may be effected on the fibre, using an unsulphonated developer of the type mentioned. As middle components ethers of 3-amino-*p*-cresol (cresidine, the sulphatoethyl ether, β -3-amino-*p*-tolylxypropionic acid), or of 1:2- or 1:6-aminonaphthols, or 1-amino-2-methylthionaphthol-6-sulphonic acid, are suitable. Amongst the examples are: H-acid \rightarrow 3-amino-*p*-tolyl ethyl ether \rightarrow *p*-aminobenzoyl-*p*-aminobenzoyl-H-acid \rightarrow 1-*m*-carboxyphenyl-3-methyl-5-pyrazolone; 4:5-dichloroaniline-2-sulphonic acid \rightarrow 2-ethoxy-Cleve acid \rightarrow *p*-aminobenzoyl-*p*-aminobenzoyl-H-acid \rightarrow phenylmethylpyrazolone; 4:5-dichloroaniline-2-sulphonic acid \rightarrow 2-ethoxy-Cleve acid \rightarrow *m*-aminoanisoyl-*p*-aminobenzoyl-H-acid \rightarrow phenylmethylpyrazolone.

C. HOLLINS.

Production of vat dyes. P. NAWIASKY and J. MÜLLER, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,742,317, 7.1.30. Appl., 22.9.27. Ger., 22.9.26).—See B.P. 289,980; B., 1928, 517.

Disazo dyes. R. KIRCHHOFF and M. CANTOR, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,740,819, 24.12.29. Appl., 12.5.26. Ger., 15.5.25).—See G.P. 439,518; B., 1927, 902.

Production of black trisazo dyes. G. WOLESLEBEN, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,740,801, 24.12.29. Appl., 1.9.27. Ger., 6.9.26).—See B.P. 303,424; B., 1929, 239.

Manufacture of [quinone] dyes. J. FRÖHLICH, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,743,034, 7.1.30. Appl., 28.7.26. Switz., 16.9.25).—See B.P. 258,563; B., 1927, 648.

Manufacture of condensation products of the [di]benzpyrenequinone series. G. KRÄNZLEIN and H. VOLLMANN, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,742,358, 7.1.30. Appl., 7.5.26. Ger., 11.5.25).—See B.P. 252,029; B., 1927, 360.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical examination of furs in relation to dermatitis. H. E. COX (Analyst, 1929, 54, 694—703).—The characteristics of the long, fully-developed hairs of the rabbit, hare, cat, peschianiki (Russian cat), opossum, skunk, nutria (coypou or otter), sable (weasel), fox, goat, wallaby, and racoon are described, and their microscopical appearance is illustrated. A number of dyestuffs more commonly used for dyeing these furs, and reactions for identifying them in their aqueous solution (1 pt. in 10,000), are described. The extracts to be tested are prepared by defatting the fur (sometimes the skin also) with light petroleum and extracting with (a) cooled boiled water and (b) 1% acetic acid for at least 24 hrs. Solutions should be kept from atmospheric oxidation and tested for inorganic substances and for acids as well as for dyestuffs and intermediates. D. G. HEWER.

Importance of circulation of liquor in the wood-pulp cooker, and means of producing it. W. SCHMID (Papier-Fabr., 1930, 28, 21—27).—The importance of circulation in producing efficient penetration of the liquor and uniformity of the wood pulp is emphasised. Apparatus for producing circulation with and without pumping, when direct or indirect heating is used, is illustrated and described.

B. P. RIDGE.

Drying of coated papers. J. B. MEYER (Papier-Fabr., 1930, 28, 17—21, 41—46).—Theoretical and practical aspects of the drying of coated papers are discussed in relation to the colloidal properties, hygroscopicity, and particle size of the pulp, dye, size, and filling, the m.p. of the binding agents, and the velocity of the air current. A method of calculating the time of drying, amount of water to be evaporated, etc. from the usual works data and the specific heats of the ingredients is described. The time necessary for a given paper can be calculated from the heat-change number and the heat-balance equation. The former is given by $\alpha = 5.3 + 3.6w$ (for $w \leq 5$ m./sec.), where α is the heat-change number and w the air velocity in m./sec. The usual apparatus employed in drying is described and improvements suggested by patents literature are critically surveyed.

B. P. RIDGE.

PATENTS.

Fibre-preparing treatment of bast or vegetable fibre plants. F. McCaw (B.P. 321,232, 7.8.28).—The plants are dried by heat and at the same time subjected to a jiggling action to level the root ends. They are afterwards de-seeded, decorticated, broken, scraped, and combed to remove the shive from the fibre. Suitable apparatus is described.

F. R. ENNOS.

Bandaging material. WOLFF & Co. KOMMANDIT-GES. AUF AKT., E. CZAPEK, and J. REITSTÖTTER (B.P. 298,606, 26.9.28. Ger., 12.10.27).—Therapeutically active substances other than glycerin are incorporated in a suitable manner with self-supporting viscose foil.

F. R. ENNOS.

Felting of textile materials. I. G. FARBENIND. A.-G., Assecs. of F. JUST, K. DACHLAUER, and E. THIEL (G.P. 457,057, 18.9.25. Addn. to G.P. 381,418; B., 1924, 90).—Felting is accelerated by the use of sulphonic acids (or their salts) of low mol. wt., obtained by treatment of aromatic and/or hydroaromatic hydrocarbons and methyl or ethyl alcohol with sulphonating agents. B. P. RIDGE.

Apparatus for testing the strength of yarns, threads, or the like. BRIT. CELANESE, LTD., H. EWING, and R. P. ROBERTS (B.P. 321,227, 4.8.28).—An increasing or decreasing load is applied to a length of the material, one end of which is fixed while the other is capable of movements relative to a record chart which are proportional, respectively, to the applied load and to the extension produced. F. R. ENNOS.

Preparation of cellulose, particularly for nitration. H. C. HEIDE. From J. J. SCHAUB (B.P. 322,997, 16.6.28).—Wood cellulose is subjected by means of a hollander to long-continued disintegration in water, to which small quantities of acids, alkalis, or salts may be added, if desired, the length of treatment being predetermined by means of a test described. After freeing the pulp from excess of water on a papermaking machine, the sheets or strips produced are twisted into cord or cut into pellets. F. R. ENNOS.

Conditioning cellulosic material for preparation of cellulose derivatives. Preparation of raw material for cellulose derivatives. [A, B] G. A. RICHTER and M. O. SCHUR, and (A) R. H. RASCH, Assrs. to BROWN Co. (U.S.P. 1,729,628—9, 1.10.29. Appl., [A] 9.10.26, [B] 5.8.27).—(A) A wood fibre of high α -cellulose content is rendered less absorptive by beating it to the slowness of pulp used in bond-paper manufacture, or by addition of a cellulose coating material such as viscose. (B) The fine, short, and broken fibres which are separated from pulp by washing on a fine-mesh sieve are detersinified and cleansed by digesting with soap solution, yielding a substantially pure product. F. R. ENNOS.

Manufacture of cellulose derivatives. C. G. SCHWALBE (G.P. 456,929, 9.7.21).—Cellulose is swollen in the cold with dilute solutions of organic acids, or their salts, which produce no hydrolysis on drying. The swelling agent is left in the fibre, which is dried if necessary, and the product is used for the preparation of the derivatives. Cellulose to be used for the production of alkali- or cuprammonium-cellulose, or viscose, is pretreated with dilute caustic soda in the cold. Cotton steeped in 1% acetic acid, or 2% sodium acetate solution, hydro-extracted, and dried at moderate temperature is acetylated in half the time otherwise taken.

B. P. RIDGE.

Manufacture of nitrocellulose. H. F. MOULTON and E. TSCHUDIX (B.P. 322,998 and 323,019, 16.6.28).—Wood pulp is prepared for nitration by (A) subjecting it to long-continued agitation with water in a hollander, and forming the pulp into strips which are spun into string-like form and cut into pellets; (B) treating it with soda lye and carbon disulphide and pressing the gelatinous paste so formed through dies to yield strings or pellets, which, after treatment with glycerin if desired, are immersed in a hardening solution.

The pellets or strings are then nitrated with a mixture of acids in which the proportion of nitric acid considerably exceeds that of sulphuric (*e.g.*, nitric acid 81%, sulphuric acid 16%, water 3%), and finally stabilised. F. R. ENNOS.

Manufacture of acidylcellulose. HEBERLEIN & Co. A.-G. (B.P. 298,087, 1.10.28. Ger., 30.9.27).—Cellulose material is treated with an acid or neutral swelling agent (*e.g.*, sulphuric or nitric acid, zinc chloride) and, after removal of the latter, either partially by pressing, centrifuging, etc. or, if necessary, completely by washing with an indifferent organic solvent (glacial acetic acid) which does not cause coagulation of the material, is acylated in the usual manner.

F. R. ENNOS.

Production of compositions including cellulose derivatives soluble in organic solvents. R. BROWNLOW (B.P. 322,464, 30.5.28).—Cellulose ester or ether products of improved clarity, suitable for conversion into cinematograph films or transparent foil for wrapping foodstuffs, are obtained if in the preparation of a solution of the cellulose derivative phosphorus oxychloride or sulphur oxychloride in association with metal halides (calcium or zinc chloride) is added. Quantities up to 5% (on the weight of cellulose derivative) of a 2:1 mixture of sulphur oxychloride and zinc chloride may be used. D. J. NORMAN.

Dry-spinning of artificial silk. H. WADE. From SYNTHETA A.-G. (B.P. 323,031, 13.7.28).—The spinning solution is kept at a temperature below the b.p. of the solvent of the cellulose derivative up to the time it leaves the spinning nozzles, but the extruded filaments within the spinning cell are subjected to a much higher temperature—up to 140°; in this way an increased spinning speed of the order of 600 ft./min. may be employed. F. R. ENNOS.

Spinning of artificial silk from cuprammonium solutions of cellulose. C. R. LINKMEYER (B.P. 297,060, 13.9.28. Ger., 13.9.27).—The formation of air bubbles in the precipitating bath is prevented by subjecting it, before use, to a vacuum slightly greater than that prevailing in the spinning funnel, and at the same time heating it above the spinning temperature.

F. R. ENNOS.

Manufacture of artificial threads. I. G. FARBENIND. A.-G. (B.P. 300,584, 13.11.28. Ger., 15.11.27).—In the manufacture of threads of high titer (staple fibre) a uniform product is obtained by arranging the spinning nozzles so that they lie either on a circular arc, all points of which are equidistant from the common thread guide, or on a spherical surface of which the common thread guide is the centre, and so that the axis of each nozzle passes through the thread guide. After neutralising etc., several such batches of thread are collected together during their conversion into the usual commercial forms (skeins, cops, etc.). F. R. ENNOS.

Manufacture of artificial filaments, threads, films, etc. BRIT. CELANESE, LTD., and S. A. WELCH (B.P. 322,764, 6.7.28).—Before extrusion through the spinning nozzles, the solution of cellulose or of the cellulose derivative is passed through a number of capillary

tubes of uniform cross-section (0.2–0.02% of the diameter of the spinning orifices) which are arranged parallel to one another and to the general line of flow taken by the solution after leaving them.

F. R. ENNOS.

Casting bed for production of films from solutions of plastic masses, especially for obtaining viscose films. H. HAMPEL (B.P. 322,784, 10.9.28).—A layer of paraffin or mineral wax, beeswax, resins, tars, bitumen, soft kinds of rubber, or mixtures of these with one another or with suitable fillers, is mounted on a roller.

F. R. ENNOS.

Manufacture of shaped masses [linoleum etc.] from cellulose derivatives of higher fatty acids. I. G. FARBENIND. A.-G. (B.P. 289,063, 20.4.28. Ger., 22.4.27).—The insoluble, higher, saturated or unsaturated aliphatic esters of cellulose (cf. Grün and Wittka, B., 1922, 94 A), in which the fibrous structure is retained, are thermoplastic and may be moulded, in absence of solvents and plasticisers, at temperatures below the m.p. by mechanical pressure, a support of paper, jute, etc. being used if desired. The same process may be applied to soluble higher fatty esters of cellulose (cf. Gault and Ehrmann, B., 1923, 826 A) and mixed esters. Examples are: cellulose laurate, claidate, acetate-stearate; cellulose esters of coconut oil acids; ethyl cellulose laurate; ethyl cellulose mixed with the mixed cellulose esters of the acids.

C. HOLLINS.

Saccharification of wood and other cellulosic materials. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 311,695, 22.1.29. Belg., 14.5.28).—Dry cellulosic material is treated with highly concentrated formic acid in the presence of a catalyst and preferably at 70–80°. The resulting product is then hydrolysed by the gradual addition of water while maintaining the temperature at 70–80° until the reaction mixture gives no precipitate when mixed with a large volume of water. Conversion of the dextrins thus formed into sugars is effected by further hydrolysis with, e.g., dilute sulphuric acid at 130°. The formic acid may be recovered by (a) distillation with a liquid capable of giving an azeotropic mixture therewith, (b) distillation with an alcohol which gives a volatile formate, or (c) extraction from aqueous solution by means of organic solvents working on the countercurrent principle.

D. J. NORMAN.

Waterproofing of paper and the like. A. F. CAMPA, Assr. to A. MEEHAN (U.S.P. 1,731,969, 15.10.29. Appl., 16.4.26).—Paper which has been saturated with liquid petroleum and freed from excess of the latter by pressure is passed at least once through a mixture containing 100 pts. of gum lac, 800 pts. of alcohol, 50 pts. of glycerin, and 50% of distilled water, and afterwards dried.

F. R. ENNOS.

Adhesive paper or fabric. I. G. FARBENIND. A.-G. (B.P. 302,588, 15.12.28. Ger., 17.12.27).—The sticking together of gum- or rubber-coated paper or fabric when reeled may be prevented, without deleteriously affecting the adhesive, by coating the reverse side of the strip with a cellulose ester, mixed ester, or ester-ether of a higher fatty acid, particularly cellulose laurate.

D. J. NORMAN.

Decortication [machinery] for preparation of vegetable fibres. D. W. DRON (B.P. 323,083, 7.11.28).

Manufacture of chenille and other fabrics. J. MORTON (B.P. 323,260, 26.9.28).

[Rotary] apparatus for preparing bamboo and kindred material for pulp extraction. J. L. JARDINE and I. T. NELSON (B.P. 323,611, 22.11.28).

Activated charcoal (B.P. 322,185).—See II. Rubber conversion products (B.P. 323,012).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Furs and dermatitis. COX.—See V. Bleaching power of sodium hypochlorite. MINAEV and others. —See VII. Myrobalans. CHOUDARY and NAYUDU.—See XV.

PATENTS.

Wool dyeing. SOC. ANON. PROGIL (F.P. 569,991, 22.8.23).—Blue-black shades, fast to light and fulling, are obtained by dyeing wool in a bath containing Chrome Black, Chrome Blue Black, Alizarin Black, Chrome Blue, potassium dichromate, lactic acid, and blue-wood extract or hæmatin.

B. P. RIDGE.

Manufacture of selvedge warps and effect threads in woollen fabrics. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 322,831, 24.9.28).—"Immunised" cotton yarns are used instead of wool, or silk, or cotton yarns for effect purposes in the manufacture of wool fabrics, since these become less tinged with the dyes with which the wool has been dyed in washing, milling, and finishing processes.

A. J. HALL.

Production of dyeings by means of dyes containing metal. SOC. CHEM. IND. IN BASLE (B.P. 298,091, 1.10.28. Switz., 30.9.27. Addn. to B.P. 297,331; B., 1929, 891).—The process of the prior patent is extended to dyeings on the fibre; e.g., wool is dyed fast navy-blue shades from a bath containing (on the weight of the wool) 7% of prechromed 2-amino-1-naphthol-4:8-disulphonic acid \rightarrow β -naphthol, 1% of prechromed 1:2:4-aminonaphtholsulphonic acid \rightarrow 1-*m*-aminosulphonylphenyl-3-methyl-5-pyrazolone, and 2% of 4-chloro-*o*-aminophenol \rightarrow H-acid.

C. HOLLINS.

Coloured cellulose films. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,044, 2.4.28).—Cellulose film (e.g., "Cellophane") is impregnated with a solution of a leuco-vat dye (Anthra Red) containing a small amount of an alkynaphthalenesulphonic acid (1 g. in 10 litres), and, after air-oxidation, is washed and dried to give transparent coloured film.

C. HOLLINS.

Dyeing of ethers or esters of cellulose or transformation products thereof. I. G. FARBENIND. A.-G. (B.P. 293,766, 10.7.28. Ger., 11.7.27).—Acetate silk etc. is dyed with water-soluble dyes, e.g., 1-amino-4-cyclohexylaminoanthraquinone-2-sulphonic acid or Orange II, in presence of cyclohexylamine, its *N*-alkyl derivatives and homologues, or hydrogenated 1:5-naphthylenediamine. The addition of these substances results in deeper shades than are otherwise obtained.

C. HOLLINS.

Dyeing cellulose acetate products in [oxidation] black shades. SILVER SPRINGS BLEACHING & DYEING

Co., Ltd., and A. J. HALL (B.P. 322 893, 11.12.28. Addn. to B.P. 258,699; B., 1926, 976).—Cellulose acetate silk is dyed with 2:4-dinitrodiphenylamine, which is afterwards reduced to the corresponding diamine on the fibre and then oxidised to black as described in the chief patent. Alternatively, the 2:4-dinitrodiphenylamine is reduced at or about the time of its application to the silk by means of agents (e.g., sodium hyposulphite) present in the dye-bath. A. J. HALL.

Treatment of raw silk, fabrics thereof, or mixed fabrics containing the same. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 320,327, 6.7. and 8.10.28).—Raw silk, which may have been hardened and/or dyed, is scoured by means of a protease (papain, bromelin) activated with hydrocyanic acid and/or hydrogen sulphide, preferably in presence of an electrolyte; the scoured silk may, before rinsing, be treated with a dilute solution of an electrolyte. The time required is 40–120 min. C. HOLLINS.

Treatment [filling] of woven and like fabrics. E. J. WILKINSON, H. M. SCOTT, and A. B. HENSHILWOOD (B.P. 322,766, 7.9.28).—Fabrics are impregnated (not coated) with fibrous filling substances such as cellulose, wool, silk, and asbestos, while passing over the perforated surface of a drum rotating in an aqueous suspension of the filling substances, and are then dried. A. J. HALL.

Colouring of products comprising cellulose acetate. G. H. ELLIS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,740,890—1, 24.12.29. Appl., 27.12.26. U.K., 27.1.23).—See B.P. 291,349; B., 1924, 906.

Stripping of [coloured] rags with sulphur dioxide. F. W. BINNS, Assr. to VIRGINIA SMELTING Co. (U.S.P. 1,741,496, 31.12.29. Appl., 6.8.27).—See B.P. 295,007; B., 1929, 849.

Treatment of yarns or threads. C. W. ADDY, J. BILLING, and H. HALKYARD, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,742,568, 7.1.30. Appl., 23.8.26. U.K., 4.6.26).—See B.P. 264,382; B., 1927, 24.

Production [improvement] of vegetable textile material. L. LILIENTHAL (U.S.P. 1,741,637, 31.12.29. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,804; B., 1925, 801.

Preserving the lustre of organic derivatives of cellulose. C. DREYFUS and H. PLATT, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,740,889, 24.12.29. Appl., 30.12.26).—See B.P. 282,722; B., 1929, 353.

Apparatus for dyeing or analogous treatment in wound form of yarns and threads. J. BRANDWOOD (B.P. 323,246, 25.7.28).

Dyeworks' effluents (G.P. 457,467).—See XXIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Stability, bleaching power, and decomposition products of sodium hypochlorite solutions. V. MINAEV, P. FOMIN, and G. JAKIMOV (Leipziger Monatsschr. Textil-Ind., 1929, 44, 72–74, 125–126; Chem. Zentr., 1929, ii, 798–799).—Hypochlorite solutions prepared

from 9% or 13% alkali are the most stable. Excess of chlorine causes decomposition, which takes place particularly in neutral solutions exposed to light. Neutral solutions possess the strongest bleaching power; sodium hypochlorite is a more active bleaching agent than bleaching powder. A. A. ELDRIDGE.

Manufacture of zinc chloride and zinc sulphate. F. CHEMNITZ (Chem.-Ztg., 1929, 53, 994–995).—Zinc chloride is made by dissolution of the commercial metal in hydrochloric acid to neutrality followed by treatment of the solution first with barium peroxide to remove iron etc., then with barium chloride to remove sulphates derived from the acid used; the solution is evaporated with a small quantity of potassium chlorate to obtain a granular mass of zinc chloride. The sulphate is made by dissolving the metal in sulphuric acid, removing the iron with permanganate and zinc oxide, evaporating to d 1.21, and allowing the liquor to crystallise. A. R. POWELL.

Technical production of anhydrous aluminium chloride. M. NAPITALI (Petroleum, 1930, 26, 55–56).—McAfee's process is described. Bauxite containing at least 57.5% Al_2O_3 , up to 5% SiO_2 and not more than 3% Fe_2O_3 is used, since silicic acid leads to the formation of silicon chloride, causing chlorine losses, and excess of iron pollutes the finished product. Bauxite is calcined at 968° in a rotary oven and then mixed (3:1) with a good coking coal. The mixture is pulverised, compounded with, e.g., molten asphalt or paraffin residues, briquetted under pressure, and heated to 833° to form briquettes consisting of 82% of bauxite and 18% of carbon. These are then air-blown for 15 min. and chlorinated for 8–10 hrs. at 860°. The aluminium chloride passes first to an air cooler and then to a vertical iron tube in which the chloride is deposited as a powder containing up to 6% of chlorides of iron, titanium, and silicon as impurities. Replacement of the coal by petroleum coke and elimination of the air-blowing are suggested as possible improvements. W. S. E. CLARKE.

Leucite industry from the physico-chemical viewpoint. N. PARRAVANO (Anal. Fis. Quím. [tecn.], 1929, 27, 341–367).—See B., 1929, 15.

Synthesis of calcium aluminate. NAGAI and NAITO.—See IX. **Barium aluminate and water purification.** STUMPER.—See XXIII.

PATENTS.

Production of pure acids. H. FRISCHER (U.S.P. 1,733,152, 29.10.29. Appl., 16.5.27. Ger., 4.12.26).—Pure volatile acids are obtained by distilling the appropriate salt with sulphuric or phosphoric acid and passing the hot vapours evolved through hot acid of the same kind while preventing any substantial condensation of the vapour in the hot acid. Thus, by passing hot acetic acid vapour through boiling acetic acid, with or without sodium acetate, any impurities carried over from the still as spray or vapour are retained by the washing acid. A. R. POWELL.

[Apparatus for the storage and] manufacture of sulphuric acid. L. SOLT (G.P. 456,995, 21.8.26).—The apparatus is constructed of or lined with acid-

resistant stoneware luted with a molten mixture of sulphur with up to 40% of acid-resistant inorganic material.

L. A. COLES.

Production of nitric acid. METALLBANK U. METALLURG. GES. A.-G. and DEUTS. SPRENGSTOFF-A.-G., Assecs. of W. MELZER and W. VON BOLTENSTERN (G.P. 457,367, 9.5.26. Addn. to G.P. 410,657; B., 1927, 813).—Finely-divided water or dilute acid is used instead of steam as described in the prior patent.

L. A. COLES.

Manufacture of phosphoric acid. J. KERSTEIN (F.P. 634,117, 10.5.27).—Moist hydrogen chloride is passed over finely-divided phosphorite at a red heat, whereby phosphoric acid volatilises. Sand may be added to the charge to keep the mass porous.

A. R. POWELL.

Production of hydrobromic acid. KALI-FORSCHUNGS-ANSTALT GES.M.B.H., Assecs. of O. F. KASELITZ and P. HÖFER (G.P. 457,059, 30.7.27).—Iron bromide, alone or mixed with absorbent material, *e.g.*, kieselguhr, sawdust, is treated at 350—380° with steam containing a small proportion of air, or, alternatively, iron is treated with a mixture of bromine vapour, steam, and a little air at the same temperature.

L. A. COLES.

[Pre-filter for] preheaters for brine solutions. G. JAUDER and H. BANTHEN (G.P. 455,223, 23.6.26).—The tervalent iron contained in brine solutions occurring in the potash industry is reduced to a bivalent state by passing the solutions through a filter containing suitable reducing material (copper waste, iron filings) before they reach the preheater. The copper tubes do not become corroded thereby since ferrous ions do not react with copper in absence of air.

S. K. TWEEDY.

Manufacture of *blanc fixe* and (A) sodium or (B) potassium nitrate. WOLFF & Co., and F. FROWEIN (G.P. 456,852—3, 26.5.25).—A mixture of (A) powdered Glauber salts or anhydrous sodium sulphate or (B) potassium sulphate with the equivalent quantity of barium carbonate is stirred into a solution containing more than 15% of nitric acid until complete neutralisation is effected. The heat of the reaction allows of the production of a nearly saturated hot solution of alkali nitrate from which the barium sulphate settles readily. After filtration and cooling to separate the nitrate crystals, concentrated nitric acid is added to the mother-liquor, which is then used again in the process.

A. R. POWELL.

Production of nitrites and nitrates [from ammonia]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,080, 31.10.28).—Mixtures of ammonia with, preferably a large excess of, air or oxygen are passed at 250—550° over a catalyst, *e.g.*, platinum, mixtures of iron oxide with bismuth oxide or manganese dioxide, or of copper oxide with manganese dioxide and, if desired, silver, at a rate as rapid as possible with the avoidance of more than traces of undecomposed ammonia in the issuing gases, which gases are brought as soon as possible after leaving the catalyst into contact with basic material, *e.g.*, lime, soda-lime, alkali or alkaline-earth carbonates.

L. A. COLES.

Cyanides [of alkali or alkaline-earth metals] and ammonia. F. BENSA (F.P. 628,616, 15.8.26).—

Ashless carbon is mixed with alkali or alkaline-earth metal compounds (oxides or carbonates), if necessary with oxides of other metals (iron, titanium, nickel, or cobalt) also, and with a binding medium. The mixture is shaped into, *e.g.*, hollow cylinders, which, by being heated in a stream of nitrogen at 900—1000° in the case of alkali metals and at 1050—1200° in the case of alkaline-earth metals, are converted into cyanide; this, on treatment with superheated steam, yields ammonia.

S. K. TWEEDY.

Production of [solid] ammonium formate. F. W. SEFTON-JONES. From LONZA ELEKTRIZITÄTWERKE U. CHEM. FABR. A.-G. (B.P. 323,113, 24.12.28).—Gaseous ammonia and carbon dioxide successively are made to react at 40° with (dry) calcium formate in the presence of a solution of ammonium formate saturated at —10°. The solution is then heated to 60° for 2½ hrs., stirred, and the precipitated calcium carbonate collected and washed to recover the formic acid. The filtrate is cooled to —10°, the calcium-free ammonium formate precipitated and filtered off, and the remaining solution returned for re-use.

P. E. L. FARINA.

Decomposition of sodium salts with hydrofluoric acid and ammonia to produce sodium fluoride and an ammonium salt. T. GOLDSCHMIDT A.-G. (G.P. 457,365, 26.10.24).—Continuous streams of hydrofluoric acid and of a solution containing ammonia and sodium sulphate, chloride, or nitrate are run simultaneously into the mother-liquor previously obtained from a similar operation, in such a manner that the solution is kept neutral or feebly acid in reaction. Portions of the liquor are removed occasionally for the recovery therefrom of the dissolved salts by fractional crystallisation.

A. R. POWELL.

Treatment of crude phosphate. F. G. LILJENROTH (F.P. 633,828, 3.5.27, and 633,866, 4.5.27. Swed., [A] 31.12.26, [B] 8.1.27).—(A) A suspension of finely-ground phosphate rock in water is treated with silicon tetrafluoride, whereby a mixture of silica, calcium fluoride, and calcium fluosilicate is precipitated, leaving a solution of phosphoric acid. The precipitate is collected, mixed with sand and clay, and heated to redness to regenerate silicon tetrafluoride for use again; the residual mass is suitable for the manufacture of cement. (B) Decomposition of the phosphate rock is effected by agitation with dilute sulphuric acid and the precipitated calcium sulphate is converted into carbonate by treatment with ammonia and carbon dioxide. The carbonate is heated with clay and sand for the production of cement.

A. R. POWELL.

Neutral phosphate. MONTAN- U. INDUSTRIEWERKE VORM. J. D. STARCK (F.P. 632,310, 6.4.27. Czechoslov., 25.6.26).—Raw phosphates containing carbonates and fluorides are mixed with the equivalent amount of acid to convert the fluorine into volatile hydrofluoric acid or into stable compounds with added materials containing oxides of iron, aluminium, titanium, etc.

E. HOLMES.

Recovery of the various acid constituents in the gases evolved in the manufacture of phosphates. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assecs. of F. RÜSBERG and F. KLEMA (G.P. 457,271, 21.10.26).—

In the manufacture of phosphates by heating phosphate rock in acid gases, the effluent gases containing silicon tetrafluoride, hydrogen fluoride, hydrogen chloride, and sulphur dioxide are washed with water or alkali salt solutions to remove the three first-named gases and leave a gas with a high content of sulphur dioxide. When water is used for washing the gases the acid solution is filtered to remove silica and the filtrate treated with a solution of an alkali chloride to precipitate the corresponding alkali fluosilicate and/or fluoride.

A. R. POWELL.

Production of calcium cyanamide. J. RAITZYNE (F.P. 628,303 and Addn. F.P. 32,584, 29.11.26).—Ammonia is passed at 600–800° over calcium carbonate containing up to 3% Fe or other catalyst which assists in the decomposition of ammonia, whereby the following reaction takes place: $\text{CaCO}_3 + 2\text{NH}_3 = \text{CaCN}_2 + 3\text{H}_2\text{O}$.

A. R. POWELL.

Manufacture of silica gel and of calcium or sodium silicate. F. RICARD (F.P. 632,509, 9.4.27).—Limestone mixed with powdered coal is burnt at 800–1000° and the resulting lime is boiled with sodium carbonate to produce a solution of sodium hydroxide and a precipitate of calcium carbonate for use again. The solution is boiled with finely-divided silicious material, filtered, and treated with carbon dioxide from the lime kiln. The silica gel is removed and the filtrate boiled with more lime to regenerate sodium hydroxide. Alternatively, the sodium silicate solution may be treated with milk of lime to obtain calcium silicate or evaporated to recover water-glass.

A. R. POWELL.

Manufacture of copper sulphate. P. E. BIGOURDAN and P. BEBIN (B.P. 323,115, 29.12.28).—Scrap copper or alloy etc. containing copper is treated with air in the presence of cupric chloride solution and the oxychloride sludge obtained is treated, after removal of the supernatant liquor, with highly concentrated sulphuric acid in quantity sufficient to yield copper sulphate crystals and cupric chloride. If desired crystallisation may be improved by the admission of steam into the liquor. After removal of the crystals the residual cupric chloride solution is used for the preparation of more oxychloride sludge.

L. A. COLES.

Complex compounds of fluorine [fluosilicates]. W. BACHMANN (Austr.P. 108,677, 18.12.25).—A mixture of fluorspar and calcium fluoride is melted in the electric furnace in the presence of carbon, whereby silicon fluoride is evolved leaving a residue of calcium carbide. The gases are passed into water for the production of hydrofluosilicic acid.

A. R. POWELL.

Treatment of greensand and the like. A. J. MOXHAM, Assr. to J. P. LAFFEY (U.S.P. 1,737,263, 26.11.29. Appl., 1.9.23).—Greensand is mixed with a slight excess of sulphuric acid and about 25% of the weight of the acid in water is added slowly, whereby a vigorous reaction occurs with the formation of anhydrous ferrous and ferric sulphates, alum, and gelatinous silica. When the mass has cooled sufficiently it becomes porous and is readily broken up. The powdered mass is stirred into boiling water, the solution decanted from any heavy residue, and reduced with

metallic iron. After filtration through a sand filter, the surface of which is continuously scraped to prevent clogging, the solution is fractionally crystallised to separate ferrous sulphate and potash alum.

A. R. POWELL.

Manufacture of alumina and salts of aluminium from bauxites or other aluminous products. U. B. VOISIN (B.P. 306,095, 14.2.29. Fr., 15.2.28).—The powdered bauxite, freed from part of its iron (*e.g.*, magnetically), is heated with sufficient fluorspar and sulphuric acid to dispel all the silica present as silicon fluoride, which is converted into hydrofluosilicic acid by passing it into water and the acid used for the manufacture of its metallic salts. The residual sulphates are taken up in water (the solution being easily filterable owing to the absence of colloidal silica), any iron present is oxidised (*e.g.*, by chlorine), and the solution is treated with calcium chloride. From the mixed chloride solution, after removal of calcium sulphate, a mixture of aluminium and ferric hydroxides is precipitated by lime; the regenerated calcium chloride can be re-used. The hydroxides are then suspended in dilute caustic soda, whereupon the aluminium hydroxide dissolves, the solution is filtered, and into the filtrate carbon dioxide is passed to re-precipitate the aluminium hydroxide. This is filtered off and the sodium carbonate present in the filtrate is causticised for use in the first part of the process.

P. E. L. FARINA.

Treatment of iron arsenate. O. C. ROBERTS (Austr.P. 3833, 15.9.26).—Roasted scorodite (FeAsO_4) is heated with sulphuric acid and the ferric sulphate caused to crystallise out. The crystals are ignited to ferric oxide and the mother-liquor, containing arsenious oxide and sulphuric acid, is used as a weed-killer.

S. K. TWEEDY.

Manufacture of magnesium arsenate. S. B. HEATH, Assr. to DOW CHEM. CO. (U.S.P. 1,737,114, 26.11.29. Appl., 11.12.24).—A light form of magnesium arsenate is obtained by heating a slurry of magnesium hydroxide with the theoretical quantity of arsenic acid and 15% of the equivalent amount of sodium hydroxide in an autoclave at 180° until the filtrate contains less than 0.2% As_2O_5 .

A. R. POWELL.

Electrolytic oxidation of cerous sulphate solutions. DEUTS. GASLÜHLICHT-AUER-GES.M.B.H. (G.P. 431,308, 19.1.24).—The cold, supersaturated solution is electrolysed in the anode compartment of a divided cell, the cathode compartment being filled with 5% sulphuric acid. Lead electrodes are used with an anode current density of 1 amp./dm.² After about 3 hrs. a quantity of 5% sulphuric acid is added to the anolyte and electrolysis is continued for a further 3 hrs. The yield of ceric sulphate is claimed to be 99% of the theoretical.

A. R. POWELL.

Production [by the Waelz process] of metallic oxides adapted to be used as pigments. METALLGES. A.-G. (B.P. 311,225, 3.4.29. Ger., 7.5.28).—Briquettes consisting of the metal ore, a reducing substance free from tarry matter, *e.g.*, coke, and a binding medium (lime, gypsum, etc.) which does not yield impurities in the form of soot or vapour on thermal decomposition, are

fed into a rotary tubular furnace, which may be additionally heated by a flame which deposits no soot.

S. K. TWEEDY.

Manufacture of metal carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,332, 30.11.28).—Carbon monoxide, or gases containing it, are allowed to act on suitable metals suspended in, or made into a paste with, a liquid or melt, formation of a thin film of the liquid or melt on the metal being prevented, *e.g.*, by agitation of the liquid or by sufficient evaporation of the liquid before treatment with the gas. Suitable liquids are hydrocarbons, preferably of high vapour pressure and having at least a certain solvent power for carbon monoxide, but the metal carbonyls themselves are pre-eminently suitable. Elevated pressure is advantageous. Alternatively, liquid saturated with carbon monoxide may be passed over the metal. S. K. TWEEDY.

Production of metal carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,021, 18.8.28).—Metals in a form suitable for the production of their carbonyls are obtained by heating the metal oxides above 500° in the presence of reducing agents and of material adapted to prevent sintering of the charge. Suitable materials include charcoal, which is introduced by the use of an excess of solid fuel, and alkali, alkaline-earth, or earth oxides or salts, which may be added as such or introduced by the use of fuel yielding a high percentage of a suitable ash. L. A. COLLS.

Apparatus for the production of oxygen. C. KIRCHNER (Swiss P. 123,923, 5.11.26).—The apparatus comprises two vessels, one above the other. From the lower part of the upper vessel a tube passes to the bottom of the lower vessel and is bent slightly upwards at its lower end. The lower vessel is filled with a liquid to which is added a solution of a substance which generates oxygen in contact with the liquid; the gas then forces the liquid into the upper vessel, thus providing the necessary pressure for the delivery of the gas where required. A. R. POWELL.

Prevention of abnormal cooling of crude liquid oxygen in the lower part of the two-column liquid air rectifier. A. MESSER (F.P. 631,663, 26.2.27. Ger., 13.1.27).—The low-pressure nitrogen from the liquefier is heated by the crude liquid oxygen in the high-pressure column before passing to the heat exchanger for cooling further quantities of compressed air. A. R. POWELL.

Production of solid carbon dioxide. W. HESSLING (B.P. 302,359, 12.12.28. Switz., 15.12.27).—Compressed liquid carbon dioxide is supercooled by the evaporation of a cooling agent, such as ammonia, circulating in an enclosed cycle to a temperature below that obtainable by means of ordinary cooling water, *e.g.*, to -30°, and is then expanded by "wire-drawing." The circulating cooling agent may also be used for liquefying the gaseous carbon dioxide so as to dispense with all high-pressure plant. S. K. TWEEDY.

Manufacture of carbon dioxide snow. T. B. SLATE (U.S.P. 1,735,094, 12.11.29. Appl., 23.10.25. Renewed 13.4.29).—Liquid carbon dioxide is forced through a small orifice into an expansion chamber to

obtain a mixture of snow and cold gas, and the latter is passed countercurrent to the liquid near the jet so as to reduce its temperature but not its pressure, and thereby increase the yield of snow (cf. U.S.P. 1,546,681; B., 1925, 759 B). A. R. POWELL.

Production of hydrogen peroxide. J. D. RIEDEL A.-G. (F.P. 634,195, 5.2.27. Ger., 17.3.26).—A solution of ammonium sulphate and persulphate obtained by electrolysis and containing 1 mol. of sulphuric acid for each mol. of salt present is subjected to distillation. A. R. POWELL.

Sulphur furnace. AKTIEBOLAGET KARLSTADT MEKANISKA VERKSTAD (N.P. 43,346, 3.3.25).—The furnace comprises a revolving outer drum surrounding an open cylinder of smaller cross-section. A. R. POWELL.

Purification of bromine. K. KUBIERSCHKY (G.P. 457,209, 28.7.25).—Narrow towers constructed of stoneware or lead are used for the fractional expulsion of chlorine from bromine by treating it with hot bromine vapour, which is generated in spiral tubes constructed of stoneware, porcelain, or silica, and situated below the towers. L. A. COLLS.

Extraction of iodine from its iodide solutions. A. HOLLARD (F.P. 631,634, 24.6.26).—One sixth of the iodide is oxidised to iodate either chemically or by means of an electric current having a density of 350 amp./m.², and the iodine is completely precipitated by acidifying the resulting solution. Any sulphides, sulphites, etc. which might be present (*e.g.*, in the extracts from calcined seaweed) are oxidised to sulphates. S. K. TWEEDY.

Preparation of ammonium sulphate. S. ROBSON (U.S.P. 1,740,837, 24.12.29. Appl., 8.9.27. U.K., 3.2.27).—See B.P. 289,950; B., 1928, 523.

Manufacture of metallic xanthates. C. J. T. CRONSHAW and W. J. S. NAUNTON, ASSYS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,740,809, 24.12.29. Appl., 17.2.26. U.K., 27.3.25).—See B.P. 252,500; B., 1926, 665.

Liquefaction of oxides of nitrogen. C. BECK and H. DIEKMANN, ASSYS. to I. G. FARBENIND. A.-G. (U.S.P. 1,741,906, 31.12.29. Appl., 11.8.28. Ger., 18.8.27).—See B.P. 321,425; B., 1930, 58.

Mica testing apparatus. L. T. FREDERICK (B.P. 323,033, 20.8.28).

Acid-resisting vessels (G.P. 456,994).—See IX. **Litharge and red lead** (B.P. 323,070).—See XIII. **Fertiliser** (F.P. 630,226).—See XVI.

VIII.—GLASS; CERAMICS.

Slagging of refractory materials. III. **Magnetite.** R. MIKSCH and H. SALMANG (Arch. Eisenhüttenw., 1929—1930, 3, 313—318; Stahl u. Eisen, 1929, 49, 1724—1725. Cf. B., 1929, 283).—Fusion tests with various metallurgical and synthetic slags in magnesia crucibles at 1650° showed that phosphoric and boric acid slags are the most destructive, and that basic slags have relatively very little action, especially those containing a mixture of several bases. In a comparison of the action of slags with a constant acid content but

with various basic constituents, those rich in lime were found to be more corrosive than those with a high content of manganese oxide, ferrous oxide, or alkali, but basic slags with a preponderating proportion of manganese oxide were somewhat more destructive. The principal factor governing the resistance of magnesite to basic slags is the amount of impurity present which can act as a flux; porosity is of relatively minor importance. A. R. POWELL.

Coke-oven walls. STEINSCHLÄGER.—See II.

PATENTS.

Manufacture of strengthened glass. II. D. MURRAY and D. A. SPENCER (B.P. 323,301, 27.10.28).—Two sheets of glass each provided with a "substratum" layer are united layer to layer by means of cellulose ester while immersed in an emulsion of two substantially immiscible liquids, one of which is a solvating agent for the cellulose ester and the other is a similar agent for the substratum. The more active swelling agent should be present in the disperse phase. An emulsion of amyl alcohol and water is suitable when the substratum is gelatin; this emulsion may be stabilised by the addition of a trace of iodine, in which case the substrata should preferably contain a de-iodising agent, *e.g.*, 0.5% of sodium thiosulphate. The emulsion may alternatively contain gelatin and acetic acid.

S. K. TWEEDY.

Shaping of fused amorphous quartz. DEUTS. ENGLISCHE QUARZSCHMELZE GES.M.B.H., Assecs. of C. ELSÄSSER (G.P. 455,305, 17.1.26).—Powdered or granular residues from the manufacture of fused silica ware are mixed with acid-resisting silicate cements, *e.g.*, water-glass, the mixture is moulded, the shapes are pickled in acid, and the surface is fused over in known manner.

A. R. POWELL.

Production of green corundum [artificial emerald]. A. KRATKY (Austr.P. 108,704, 22.5.26).—A mixture of pure alumina with a trace of cobalt-zinc oxide green pigment is melted in an oxyhydrogen flame to which is fed a small quantity of air to avoid overheating the charge and consequent volatilisation of the zinc oxide. A similar result is obtained by adding to the alumina small quantities of a colourless substance which reduces the m.p. of the product. A. R. POWELL.

Manufacture of clay articles. J. W. CRUIKSHANK. From F. W. PRESTON (B.P. 322,804, 15.8.28).—In the manufacture of clay articles, in particular glass-melting pots, the plastic body mixture is simultaneously tamped and rolled against a mould surface to a thickness in excess of that required in the finished article. The material is then further compacted and smoothed to reduce it to the required thickness. The process may be carried out under vacuum. Suitable apparatus for carrying out the process is described. F. SALT.

Ceramic material and its manufacture. (Sir) G. C. MARKS. From A.C. SPARK PLUG Co. (B.P. 322,361, 17.12.28).—A mixture of 60–90% by wt. of alumina, 5–35% of clay, and 5% of a lithium compound is completely fused and rapidly cooled in water. The resulting refractory is mixed in approximately equal

proportions with clay in a porcelain-body mixture for making insulators, sparking-plugs, etc. F. SALT.

Manufacture of ceramic products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,739, 21.8.28).—Bricks etc. of a satisfactory dark colour are produced from ordinary clays by the addition of finely-divided ferrosferic oxide. The oxide obtained in the reduction of an aromatic nitro-compound to the corresponding amine may be used for the purpose. A reducing kiln atmosphere is not necessary. F. SALT.

Salt-glazing of bricks and other clay products. S. H. IVERY, Assr. to HYDRAULIC-PRESS BRICK Co. (U.S.P. 1,735,167, 12.11.29. Appl., 24.2.28).—A slip (or engobe) containing kaolin, flint, alkali aluminium silicates, frit, ball clay, and a colouring agent is applied to the surface to be glazed; the articles are then dried and placed in a salt-glaze kiln so that the glazed surface of the ware is exposed to the kiln gases. Salt is added to the fires when the kiln is at, or near, maximum temperature. F. SALT.

[Fire- and damp-proof] bricks, tiles, etc. A. BRUNO (B.P. 321,895, 19.2.29).—A mixture for making bricks etc. contains magnesite, rock alum, magnesium chloride solution, and sawdust. Marble dust, or a suitable substitute, may also be added. The mixture is spread on a glass or other polished surface and fired at 21–38°.

F. SALT.

Enamelling of fibro-cement and similar material. N. ZAVARO and A. SPINGUEZ (B.P. 310,886, 1.5.29. Belg., 2.5.28).—A first coat of enamel, diluted with turpentine, is sprayed on to fibro-cement material and baked. A second coat, more fusible than the first and diluted with water, is then applied in the same way. Gradual preheating and cooling of the layers is essential to prevent cracking.

F. SALT.

Manufacture of refractory compositions. T. S. CURTIS, Assr. to VITREX Co. (U.S.P. 1,741,920, 31.12.29. Appl., 21.12.26).—See B.P. 289,560; B., 1928, 484.

Apparatus for drawing glass in sheets. Soc. ANON. DES ATELIERS DE CONSTRUCTION & FONDERIES DE JEUMONT (B.P. 310,952 and Addn. B.P. 317,067, 14. and 15.3.29. Fr., [A] 5.5.28, [B] 10.8.28).

Manufacture of sheet glass. A. E. WHITE. From PITTSBURGH PLATE GLASS Co. (B.P. 323,151, 19.2.29).

Rolling and annealing of glass plates. N. V. MAATSCHAPPIJ TOT BEHEER EN EXPLOITATIE VAN OCTROOIEN (B.P. 309,179, 4.4.29. Ger., 7.4.28).

Annealing of glass plates. E. HILGERS (B.P. 314,385, 24.6.29. Ger., 26.6.28).

Manufacture of glassware. HARTFORD-EMPIRE Co., Assecs. of K. E. PEILER (B.P. 310,469, 17.4.29. U.S., 26.4.28).

Coatings for metals (F. P. 632,510).—See X. **Glass substitute** (B.P. 307,462).—See XIII.

IX.—BUILDING MATERIALS.

Heat balance in rotary [cement] kilns. A. C. DAVIS (Cement, 1929, 2, 323–327).—The method of calculating the effective heat utilised in burning cement

in a rotary kiln is described. It closely follows the usual method employed in boiler, kiln, or furnace practice.

C. A. KING.

Fixation of lime by the constituents of the clay in progressive heating of cement raw mixture.

H. KÜHL and H. LORENZ (Zement, 1929, 18, 604—611; Chem. Zentr., 1929, ii, 624).—The formation of clinker substances proceeds progressively from those poor to those rich in lime; the compound $2\text{CaO}, \text{SiO}_2$ is chiefly produced at 1000—1250°. Sintering at 1250° causes vigorous fixation of lime, the calcium silicates and aluminates formed immediately passing into the clinker material. The process of fixation of lime accords with the view that alite forms a series of solid solutions between $3\text{CaO}, \text{SiO}_2$ and $3\text{CaO}, \text{Al}_2\text{O}_3$, whilst celite is regarded as forming a series from $2\text{CaO}, \text{Fe}_2\text{O}_3$ and $4\text{CaO}, \text{Al}_2\text{O}_3, \text{Fe}_2\text{O}_3$.

A. A. ELDRIDGE.

Fundamental synthesis of calcium aluminates and their hydration. I.—III.

S. NAGAI and R. NAITO (J. Soc. Chem. Ind. Japan, 1929, 32, 221—223 B, 286—288 B, 295—297 B).—I. The formation of the aluminates $\text{CaO}, \text{Al}_2\text{O}_3$ and $5\text{CaO}, 3\text{Al}_2\text{O}_3$ has been studied by heating mixtures of calcium oxide and alumina at intervals from 950° to 1600°. Both aluminates are formed easily at temperatures much below the fusion point; one heating at 1250° for 7 hrs. gave 90% of $\text{CaO}, \text{Al}_2\text{O}_3$, which sintered at 1500° and had m.p. equivalent to Seger cone 26—27.

II. The formation of $3\text{CaO}, 5\text{Al}_2\text{O}_3$, similarly followed, showed a prior formation of $\text{CaO}, \text{Al}_2\text{O}_3$. Uncombined alumina still existed at 1600°.

III. The hydraulic strength of mortar prepared with $5\text{CaO}, 3\text{Al}_2\text{O}_3$ is less than that with $\text{CaO}, \text{Al}_2\text{O}_3$.

C. A. KING.

Use of calcium chloride or sodium chloride as a protection for mortar or concrete against frost.

W. N. THOMAS (Dept. Sci. Ind. Res., Building Res. Spec. Rep. No. 14, 1929, 30 pp.).—Whilst both calcium and sodium chlorides afford protection against a limited degree of frost during the early setting period of cement mixtures, the probability of other disadvantageous properties offsets any value in this respect: sodium chloride is liable to cause efflorescence, calcium chloride to produce discoloration of the concrete face. Serious diminution in strength due to the use of sodium chloride seems to be established, and the employment of 2—4% of calcium chloride also appears of a risky nature, though results as to its effect on the ultimate strength are not confirmatory. No addition of such salts should be made for reinforced construction.

C. A. KING.

Mixed Portland cements. IV. S. NAGAI (J. Soc. Chem. Ind. Japan, 1929, 32, 288—289 B).—The results of tests of a mixed cement of the "Neo-solidit" type were nearly equal to those of blast-furnace slag cements and showed quick-setting properties. This type of cement is made by mixing blast-furnace slag, clay, or alunite extraction residues with materials such as kieselguhr, silica earth, etc., and grinding about 25—30% of this mixture with an ordinary Portland cement clinker.

C. A. KING.

Durability of Portland cement. T. MERRIMAN

(Eng. News-Rec., 1930, 104, 62—64).—The solubility of lime from cement in a solution of sucrose parallels the disintegration of cement in sodium sulphate solution, and is regarded as a more reliable criterion of the permanence of concrete than are the setting properties or strength tests usually adopted. The solution used contains 15% of sugar, calcium hydroxide being added so that 25 c.c. of the solution is neutralised by 5 c.c. of 0.5*N*-hydrochloric acid. The addition of lime regulates the solubility equilibrium of different brands of cements. To make the test 7.5 g. of cement are shaken with 25 c.c. of the solution for 2 hrs. and the solution is then titrated with hydrochloric acid. When phenolphthalein is used, a value for the total lime in solution is obtained; with methyl-orange, a combined value for lime, silica, iron oxide, and alumina. A low solubility indicates that the cement is resistant to disintegration, and has therefore been well manufactured, and, in general, cements which showed the lower values for differences in the solubilities shown by the two indicators were those which had the lower content of alumina. This difference may be regarded as an index of disintegration. C. A. KING.

Acid-proof cement mortars. I—III.

S. NAGAI and S. MATSUYAMA (J. Soc. Chem. Ind. Japan, 1929, 32, 233—234 B, 239 B, 294—295 B).—The chemical and physical properties of a number of siliceous powders and alkaline silicate solutions are given. Alkali silicates of high silica content, e.g., $\text{Na}_2\text{O}, 3\text{SiO}_2$, are preferred for the mixing of acid-proof cements. For mortars, soluble sulphates, chlorides, etc. should be absent, and the "siliceous" products finely divided. C. A. KING.

Laitance of cement mortars and concrete. I.

S. NAGAI and K. YOSHIKAWA (J. Soc. Chem. Ind. Japan, 1929, 32, 240—242 B).—Mortars were prepared from cement and sand of known composition with water in various ratios, the laitance formed on the surface was scraped off in successive layers, and these latter were analysed. The outermost layer was found to be practically pure calcium carbonate with only traces of silica. The silica content increases steadily from the outer layers inwards, as does also the insoluble residue. The inner layers thus consist of hydrated silicates, free silica, and other impurities.

C. IRWIN.

Road materials.

K. A. CLARK (Ninth Ann. Rept. Sci. Ind. Res. Council of Alberta, 1928, 39—48).—Bitumen emulsion prepared from bituminous sand is rapidly broken by admixture with gravel, though only slowly with fragments of stone. Experiments carried out with different sizes of gravel showed that the smaller the particles the more rapid was the separation, but that satisfactory spreading could be achieved by adding small amounts of kerosene or by heating the materials. It is, however, possible to emulsify bitumen in the presence of sand by using oleic acid or sodium silicate as agents to produce a suspension of oil in water. A large amount of information has also been accumulated with reference to separation of bitumen from different sands, but the results are inconsistent and further work is necessary.

R. H. GRIFFITH.

PATENTS.

Process and oven or kiln for baking pulverulent materials such as limestone. J. BELLAY (B.P.

309,177, 3.4.29. Belg., 7.4.28).—The pulverulent material is continuously supplied to an annular kiln which has a refractory lining through which radially disposed burners extend and which surrounds an annular fuel chamber containing, *e.g.*, coke, and surrounding in turn a central space heated by burners at the bottom; the hot gases from this space are used to generate steam which is introduced into the fuel chamber for the purpose of producing water-gas for supplying to the burners. The material is heated uniformly and, in the case of limestone, the treatment is completed in 4 hrs.

S. K. TWEEDY.

Burning of cement and similar substances in rotary kilns. M. VOGEL-JORGENSEN (B.P. 323,010, 13.8.28).—In the cooler end of a rotary kiln chains are suspended from iron bars projecting into the interior of the kiln. The bars may be shaped or twisted to aid the movement of the charge, and the chain suspension is adjusted so as to minimise the wear on the kiln wall. A considerable weight of chain, *e.g.*, 10 tons, may be introduced, resulting in high heat transmission which necessitates the provision of forced draught to the kiln.

C. A. KING.

Production of Portland cement. G. SNIJDER (G.P. 457,116, 29.3.25).—A charge comprising calcium carbonate, crystalline silica, *e.g.*, sand, quartzite, and metal oxides in quantity such that the product contains not more than 4% of material other than calcium silicate is calcined and the product is ground.

L. A. COLES.

Cement from limestones rich in sand. L. M. BEYLER (F.P. 630,856, 29.12.26).—The carbon dioxide is removed from the limestone by burning and the requisite amount of water for slaking the quicklime formed is added to the residue. The slaked lime and sand are separated by fanning, the former being used for making cement, and the sand for silicate bricks.

S. K. TWEEDY.

Acid-resisting ferroconcrete vessels. J. GLATZ (G.P. 456,994, 12.7.27. Czechoslov., 13.7.26).—A vessel for use with hot acid solutions comprises an outer ferroconcrete casing and an inner lining of the same material, the linings being separated from one another by heat-insulating asbestos packing. The inside of the inner lining is covered with glazed porcelain or glass tiles embedded in acid-resisting cement and extending over the rim of the vessel and for a short distance down but away from the outer casing.

A. R. POWELL.

Binding medium. J. EHRE (F.P. 630,352, 29.4.26).—A (? burnt) finely-ground mixture of 84% of blast-furnace slag, 10% of infusorial earth, 4% of quicklime, and 2% of coke is suitable for making bricks and pipes and for cement structures.

S. K. TWEEDY.

Production from magnesite of active calcination products [for use in the manufacture of oxychloride cements]. OESTERR.-AMERIKANISCHE MAGNESIT A.-G. (Austr.P. 108,257, 25.2.22).—Calcined magnesite containing more than 7% CO₂, but nevertheless capable of yielding hard-setting oxychloride cements, is prepared by heating natural or artificial magnesite in lumps of uniform, predetermined size in rotating kilns at a suitable, uniform temperature and for an appropriate length of time.

L. A. COLES.

Plaster and process of controlling the setting and expansion of setting, an accelerator and expansion controller therefor, and their manufacture. J. D. WIGGIN and M. M. REMMES, Assrs. to H. B. WIGGINS' SONS Co. (U.S.P. 1,732,737, 22.10.29. Appl., 17.7.24).—Powdered plaster of Paris (1000 lb.) is mixed with 84 lb. of dry boric acid powder and, while agitating, a solution of 16 lb. of boric acid in 60 lb. of water is sprayed over the mass. After drying at the ordinary temperature for some days the mass is dried and ground. It may then be added in any desired proportion to fresh plaster as a controller of the expansion on setting.

A. R. POWELL.

Drying of damp masonry. E. MURMANN (Austr.P. 108,275, 15.6.25. Czechoslov., 9.9.24).—A small quantity of potassium sulphate or barium carbonate is added to the mortar used to convert the calcium nitrate it always contains into calcium sulphate or carbonate and leave the non-deliquescent potassium or barium nitrate. Any magnesium nitrate present behaves similarly.

A. R. POWELL.

Forming a pavement or the like. F. B. DEHN. From FLINTKOTE Co. (B.P. 322,835, 3.10.28).—A concrete base while wet is covered with a thick layer of a bituminous composition consisting of clay (fuller's earth, slate dust, ochre, or sienna) 50%, water 35%, asphalt 15%. Soap or sulphonated oils may be added to assist emulsification, and broken stone or other aggregate embedded, if desired.

C. A. KING.

Making of roads, pavings, foundations, etc. BERRY, WIGGINS & Co., LTD., and H. H. HOLMES (B.P. 322,923, 9.1.29).—In the preparation of road-making material with the aid of a bitumen emulsion, a dilute solution of a metallic salt other than a salt of an alkali metal is incorporated to invert the emulsification of the bitumen. Such a solution may be applied to a road which has been treated with bitumen emulsion.

C. A. KING.

Manufacture of macadam and gravel roads. P. B. DE BIE (B.P. 315,846, 11.9.28. Holl., 20.7.28).—Voids between the uncoated aggregate of a road composition are filled with a mixture of fine-grained material, *e.g.*, sand, and bitumen emulsion, so that the road crust will contain not more than 6% by vol. of bitumen.

C. A. KING.

Treatment of the wood of living trees or of the still-living trunks of felled trees. F. A. BRAUSIL (Austr.P. 108,437, 7.6.26).—The wood is impregnated with solutions of ammonium salts in conjunction with the chlorine compounds of manganese, and, if desired, with the further addition of salts of iron, copper, tin, etc., and organic preservatives, *e.g.*, tetrachloroethane, tetralin, amyl acetate, or oxalic acid; dyes may be added also. The solutions form insoluble compounds with the substances present in the wood so that they cannot be removed by washing.

A. B. MANNING.

Increasing the strength of timber and rendering it immune from attack by animal, bacterial, and fungoid organisms. (SIR) G. C. MARKS. From REGENTS OF UNIV. OF CALIFORNIA (B.P. 323,013, 14.9.28).—A solution of a salt of a toxic heavy metal is injected into a living tree for distribution by the circulation of

sap, and subsequently a reducing or precipitating reagent is injected to render the metal insoluble and fixed. Suitable compounds are the salts of copper, mercury, zinc, or lead.

C. A. KING.

Manufacture of composition board or the like. WOODCRETE, LTD., Asses. of E. J. FOSTER (B.P. 300,982, 23.10.28. Austral., 20.8.28.).—Casein, either dried or in pressed curd, is treated at 60–95° with a solution of sodium hydrogen carbonate or similar alkaline substance; a casein solvent, such as ammonia, is then added and finally glycerin (which imparts flexibility), the raised temperature being maintained. Shavings, wood dust, and/or vegetable fibres, etc. are then incorporated. The stiff, plastic product is shaped, partially dried, and treated with an insolubilising agent, such as formaldehyde.

S. K. TWEEDY.

Manufacture of fused cement. B. BRUHN, Assr. to G. POLYSIUS (U.S.P. 1,741,973, 31.12.29. Appl., 9.6.27. Ger., 10.9.24).—See G.P. 416,592; B., 1926, 15.

Heat- and sound-insulating coverings. RHEINHOLD & Co. VEREIN. KIESELGUIR- U. KORKSTEIN-GES. (B.P. 312,631, 26.2.29. Ger., 29.5.28. Addn. to B.P. 305,048).

Heat- and sound-insulating coverings (B.P. 305,048 and 310,950). **Removal of dust from air** (G.P. 456,612).—See I. **Road-making etc.** (B.P. 322,792—3). **Bituminous compositions** (B.P. 322,431). **Oil-chalks** (G.P. 455,306).—See II. **Treatment of crude phosphate** (F.P. 633,828 and 633,866).—See VII. **Tiles, bricks, etc.** (B.P. 321,895).—See VIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Investigation of the blast-furnace process by analysis of samples of the charge. G. BULLE, II. BANSSEN, and G. EICHENBERG (Arch. Eisenhüttenw., 1929—1930, 3, 169—172, 241—248, 325—330; Stahl u. Eisen, 1929, 49, 1760—1762).—Samples have been taken of the charge at various stages in its progress through the blast furnace and analysed for carbon, silicon, manganese, and phosphorus. The granules of iron recovered from the higher parts of the furnace contained 0.5—2% C, whereas the iron discharged from the furnace contained 3.6% C, showing that carbides are probably formed only at the higher temperatures. The silicon content of the iron appears first to increase, especially just below the tuyère zone, but falls again rapidly towards the taphole. The greater part of the melting takes place in front of the tuyères, but the central core remains more or less solid with granules of iron dispersed throughout the mass. The purity of these granules decreases from the edges towards the middle, showing that the zone in front of the tuyères is highly oxidising, but that, owing to the rapidity with which the metal falls through this zone, relatively little oxidation of the carbon and silicon takes place, and this is made up by the molten metal coming into contact with the reduced central core below the tuyères.

A. R. POWELL.

Investigations on a blast-furnace smelting minette with Saar coke. A. WAGNER and G. BULLE (Arch. Eisenhüttenw., 1929—1930, 391—395; Stahl u. Eisen,

1929, 49, 1860—1861).—Samples of the gas taken from a blast furnace during the smelting of minette with Saar coke showed that the hydrogen content increased from about 0.2% at the tuyères to 2% at 12 m. above and to 3.5—4% in the flue gas, which also contained 11% CO₂ and 28% CO. Thus it appears that about 20% of the oxygen content of the ore is removed by indirect reduction by the hot gases. About 81% of the Saar coke reaches the tuyère level compared with 70—74% for Upper Silesian coke, which is much harder and more difficultly combustible. Only 8.5% of the carbon in the coke is used in the direct reduction of the ore and another 4% is burnt by the moisture present. The flue dust obtained in a straight run averages 37% of the weight of iron produced, but this may be reduced to 10% by removing the fine ore (under 12 mm.) and briquetting it with the flue dust. The latter has a high content of ferrous oxide together with some lime, which occurs as silicate in the ore, and particles of metallic iron, showing the highly reducing conditions which prevail in the furnace. Some granules of iron taken from the tuyère level and carefully cleaned contained only 70—90% Fe with 5—0.8% Si, but less carbon, manganese, and phosphorus than the pig iron tapped from the furnace. The slag first formed in the smelting appears to be appreciably less basic than that tapped.

A. R. POWELL.

Advantages and disadvantages of separate and joint working of the gas producers in open-hearth furnace practice. F. WESEMANN (Stahl u. Eisen, 1929, 49, 1853—1859).—The operation of a steelworks in which every open-hearth furnace is coupled to its own series of gas producers is compared with that of a works in which all the producers discharge their gas into a common pipe-line from which it is supplied as required to any furnace. The results summarise the practice of sixteen German steelworks. The first method has the advantages that the gas produced can be regulated to the requirements of the furnace, the valves do not require constant supervision, and the supply pipes are more readily cleaned; on the other hand the producers must not be more than 50 m. away from the furnace to avoid serious heat losses, and the fuel consumption is high unless the furnace has a capacity of more than 30 tons. The joint method gives a more even gas supply, but the standardisation of its heat content is not easy; there is always a loss of pressure in the main pipe-line, and frequently trouble arises due to fouling of the valves, which in this system affects more than one furnace; again, supervision of the pipe-lines and removal of accumulated dust from them is a lengthy operation.

A. R. POWELL.

Difference between adding pig iron in the solid and in the liquid state to the open-hearth furnace charge. E. KILLING (Stahl u. Eisen, 1929, 49, 1821—1826).—Addition of pig iron to the open-hearth furnace charge in the liquid state gives a better output in tons/hr. than addition of solid pig iron, but more ore is required for refining and a larger quantity of slag is produced, showing that there must be a larger amount of iron oxidised in this procedure. The best output is obtained with liquid pig iron when 35% of the charge consists of this material, and with solid

pig iron when 19% is used. The cost of the process with solid iron considerably exceeds that of the process using liquid iron unless 62% or more of the charge consists of pig iron, when the costs are approximately equal; the addition of the pig iron in a molten state is therefore more economical in spite of the greater losses by oxidation. A. R. POWELL.

Absorptive power of pure iron and its alloying elements for hydrogen and nitrogen. E. MARTIN (Arch. Eisenhüttenw., 1929—1930, 3, 407—416; Stahl u. Eisen, 1929, 49, 1861—1863).—The volume of hydrogen absorbed by pure electrolytic iron previously annealed at 1200° in hydrogen increases linearly with rise of temperature to 900°, when there is a sudden sharp increase followed by a second linear increase at a somewhat more rapid rate up to 1200°. The absorption of nitrogen is very small below 900°, but at this temperature 17 c.c./100 g. is absorbed, part of which is slowly evolved on further heating. Chromium absorbs less hydrogen than iron up to 800° and in nitrogen it forms a stable nitride at 600° which is not decomposed even at 1200° under 0.001 mm. pressure. Molybdenum absorbs less hydrogen and nitrogen than iron, but there is a maximum absorption of both gases at 800°. Tungsten does not take up either gas below 1200°. Silicon has little effect on the absorptive power of iron for hydrogen except when it is present to an extent which inhibits the γ -transformation; then the volume of hydrogen occluded by the silicon-iron alloy above 900° is less than that taken up by pure iron. In the α -field silicon-iron alloys absorb much more nitrogen than does pure iron, but in the γ -field the effect of silicon is small. The state in which the absorbed gas is present cannot be definitely established as yet, but some theories to explain the above behaviour are discussed. A. R. POWELL.

Comparative tests on the heat treatment of case-hardened steel. H. LÜPFERT (Stahl u. Eisen, 1929, 49, 1717—1724).—Space diagrams have been constructed for a case-hardened mild steel and a case-hardened nickel-chromium steel showing the variation of grain size with annealing temperature, time of annealing, and rate of cooling. From these diagrams it is shown how the temperature and time of case-hardening must be regulated in order to obtain the desired degree of penetration of the carbon with the minimum coarsening of the grain size. The bending properties and width of the transition zone under varying heat treatment of the steels are recorded, and micrographs of the structure are reproduced. The higher the hardening temperature and the more prolonged the heating, the poorer is the quality of the outer layers but the tougher is the central part of the specimen. When possible it is recommended to give only one case-hardening treatment, in which case the rate of cooling is of little consequence. If, however, a double treatment is necessary the steel should receive an intermediate anneal and should be cooled rapidly after the second hardening operation. A final anneal at 200° is also advisable to remove internal stresses and to improve the wearing properties of the case. A. R. POWELL.

Physical properties of pure chromium steels and tungsten steels. F. STÄBLEIN (Arch. Eisenhüttenw., 1929—1930, 3, 301—305; Stahl u. Eisen, 1929, 49, 1762—1763).—The density of pure chromium-iron alloys decreases by 0.0083 for every 1% Cr and that of tungsten-iron alloys increases by 0.048 for every 1% W. The specific resistance increases linearly up to 2% Cr or 7% W, then remains more or less constant in the tungsten series and increases less rapidly in the chromium series. Chromium reduces the heat conductivity of iron rapidly, alloys with 5—13% Cr having only half the conductivity of pure iron; tungsten, on the other hand, has little effect. The coercivity of chromium-iron alloys rises to an ill-defined maximum with 10% Cr, then falls to a slight minimum at 17% Cr, whereas that of the tungsten alloys rises sharply between 5 and 10% W and reaches a well-marked maximum at 15% W. Other magnetic measurements on tungsten-iron alloys indicate the existence of a non-magnetic compound, probably FeW, in alloys with more than 6% W. The γ -field of iron-chromium alloys with 0.03% C extends to 18% Cr, whereas, according to Oberhoffer and Kreutzer (B., 1929, 285), it extends only to 12.62% Cr with 0.01% C. In the tungsten series the A3 transformation can no longer be detected with the 5% W alloy. In both series the mean coefficient of thermal expansion decreases with increase of the alloying element. A. R. POWELL.

Cold and hot deformation of austenitic nickel steel and transformer iron. F. SAUERWALD [with F. FLEISCHER, A. FISCHNICH, and A. RADENACHER] (Arch. Eisenhüttenw., 1929—1930, 3, 365—368; Stahl u. Eisen, 1929, 49, 1799).—Rapid deformation of austenitic nickel steels at 900—1000° still causes slight hardening, but silicon-iron alloys soften immediately after deformation at this temperature. Spontaneous recrystallisation after deformation occurs just above 900° with nickel steel and at 1000° with transformer iron. Both alloys exhibit maximum hardening when worked at the temperature of blue-brittleness which occurs in all nickel-iron alloys and even in pure nickel as well as in α - and γ -iron. A. R. POWELL.

Technological and metallographic properties of a high chromium-nickel steel. E. GREULICH and G. BEDESCHI (Arch. Eisenhüttenw., 1929—1930, 3, 359—363; Stahl u. Eisen, 1929, 49, 1832—1833).—The mechanical properties and microstructure of a non-rusting steel containing 33.61% Ni, 10.10% Cr, 0.28% C, 0.15% Si, 1.88% Mn, 0.03% P, and 0.03% S have been investigated after heat treatment at 400—1200°. The test-pieces were forged hot, annealed at 1050°, quenched in water, and cold-rolled to 36.1% reduction in thickness; they were then annealed at 50° intervals between 400° and 1200° for 2 hrs. The hardness remained constant at 280 kg./mm.² up to 650°, then fell rapidly to 139 at 900—1050°, and finally more slowly to 107 at 1200°. The impact strength remained constant at 3.3 m.-kg./cm.² up to 650°, then increased steadily to 15 m.-kg./cm.² at 1120° and rapidly to 21 m.-kg./mm.² at 1150°, falling again to 19 m.-kg./cm.² at 1200°. The tensile strength of the cold-worked metal is retained up to 650°, but at 680°

it has fallen to that of the annealed steel. The structure of the steel is austenitic; at 600–700° cleavage of the crystals begins along crystallographic planes, but new crystals do not become visible below 750°. Between 750° and 1100° the average grain size ϕ at t° is given by the equation $\phi = 0.381e^{(t-750)/0.0112}$. Specimens annealed at 750–800° show the presence of numerous particles of precipitated carbides which re-dissolve slowly at higher annealing temperatures, finally disappearing at 1150°. The rate at which the carbide nuclei dissolve at 800–1150° is a logarithmic function of the temperature. The presence of these nuclei in the structure causes a serious reduction in the ductility, especially at 700–750°. A. R. POWELL.

Rusting of copper steel in the atmosphere and in various waters. C. CARUS and E. H. SCHULZ (Arch. Eisenhüttenw., 1929–1930, 3, 353–358; Stahl u. Eisen, 1929, 49, 1798–1799).—The high resistance to rusting of steel with 0.2–1% Cu when exposed to a damp atmosphere is shown to be due to the formation of a tenacious layer of cupric oxide below the iron scale which first forms. This layer is a secondary deposit produced by oxidation of the copper set free when the outer layer of iron oxide is formed. Under water it is possible to arrange conditions which show that the copper liberated is first converted by the ferric ions into cuprous ions, which are immediately reduced so that the steel becomes covered with a film of copper, which then oxidises to cupric oxide. In the presence of chloride ions the deposited copper is spongy and is rapidly converted into cupric hydroxide, which then accelerates rusting of the iron by the formation of local cells. A. R. POWELL.

Rapid determination of carbon in steel magnetically by means of Malmberg's carbometer. P. KLINGER and H. FÜCKE (Arch. Eisenhüttenw., 1929–1930, 3, 347–352; Stahl u. Eisen, 1929, 49, 1725–1726).—Tests made at a steel works on samples of steel containing 0.1–1.4% C taken directly from the bath and hardened in the usual way have shown that the carbon content can be determined in a few minutes with an accuracy of $\pm 0.02\%$ C by means of Malmberg's carbometer (B.P. 184,215; B., 1922, 763 A), in which the test-piece is subjected to the influence of a magnetic field which is varied several times between two fixed values and the change in the magnetic induction is measured by means of a galvanometer. A. R. POWELL.

Supposed influence of copper in the determination of sulphur in iron and steel. H. PINSL (Giesserei-Ztg., 1929, 16, 453–458; Chem. Zentr., 1929, ii, 610).—Copper in quantities normally present in pig iron, cast iron, or steel does not affect the accuracy of the determination of sulphur by the method of treatment with hydrochloric acid. For the determination of copper, hydrogen sulphide is passed into the unfiltered solution after addition of a deficiency of ammonia, the precipitate and residue are together washed with hot water, and ignited at a moderate temperature until the graphite has been burned off. The copper is then extracted by means of hot concentrated hydrochloric acid and determined, e.g., by means of sodium thiosulphate. A. A. ELDRIDGE.

Electrolytic refining of copper using complex salts of cuprous chloride. III, IV. N. KAMEYAMA and T. NODA (J. Soc. Chem. Ind. Japan, 1929, 32, 232 B).—The equilibrium potential of copper and the electrode potentials at various current densities were measured at 25° and 50° in the complex electrolytes previously described (B., 1928, 573). The cathodic polarisation is small and the anodic smaller still. The behaviour of possible impurities in crude copper has been considered in the light of the results obtained. S. K. TWEDDY.

Properties of cobalt-chromium-tungsten alloys. K. LÖBBECKE (Mitt. Forsch.-Inst. Ver. Stahlw. A.-G., 1929, 1, 65–82; Chem. Zentr., 1929, ii, 642).—The average composition of "stellite" alloys is Co 55, Cr 28, W 15, C 2.5%, with small quantities of impurities, particularly iron. The hardness increases with increasing chromium and decreasing cobalt content; the tungsten has little influence. Increase in the proportion of carbon causes at first an increase in hardness, followed by a fall to a constant value, owing to the formation of graphite. The resistance to wear increases generally with diminishing cobalt content; chromium and tungsten have little influence. The alloy most resistant to corrosion by hydrochloric or acetic acid is that of the above metallic composition; the carbon content plays an important part. A. A. ELDRIDGE.

Influence of wolfram on the "combined tin" content of slags produced in the smelting of cassiterite. K. V. CHRISTIE and E. O. JONES (Bull. Inst. Min. Met., 1930, No. 304, 9 pp.).—The replacement of silica by tungsten trioxide in slags produced in tin smelting causes a gradual increase in the stannous oxide content of the slag and an increase in the viscosity. Slags with a high content of tungsten trioxide usually contain, in addition to a high content of stannous oxide, a large proportion of tin in the form of prills. With a suitable adjustment of the amount of reducing agent used, tin can be produced quite free from tungsten and with only a small proportion of iron even when the slag contains 30% FeO, but the slag contains about 12% SnO. The fact that this tin cannot be recovered in a pure form by further addition of reducing agent, but always appears as hardhead, suggests that the removal of tin from silicate slags is a precipitation process, thus: $\text{Fe} + \text{SnO}, \text{SiO}_2 = \text{Sn} + \text{FeO}, \text{SiO}_2$. By using less ferrous oxide and more lime in the smelting the tin content of the slag is appreciably reduced. A. R. POWELL.

Iron kettles tinned with tin-lead alloys. J. T. DUNN and H. C. L. BLOXAM (Analyst, 1930, 55, 34).—The kettles examined were tinned with a tin-lead alloy composed of 1 pt. of tin to 2 pts. of lead. After boiling water of hardness of 7° in them for 24 hrs. the coatings were penetrated and spots of rust appeared. By boiling sections of known areas in such water for 16 hrs., 1.5 mg. of lead were dissolved from 100 cm.² of tinned iron. The action is regarded as electrolytic and the use of such kettles as harmful for potable water. D. G. HEWER.

Complete analysis of antimony ores, regulus, and alloys. H. BLAHETEK (Chem.-Ztg., 1929, 53, 995–996).—The finely-ground sample (10 g.) is boiled

with hydrochloric acid, the solution evaporated to dryness, and the insoluble residue fused with sodium carbonate. After separation of the silica the acid filtrate is united with the main solution and the mixture poured into freshly prepared ammonium sulphide. The precipitate is collected in a glass filtering crucible, washed with ammonium sulphide, and dissolved in hydrochloric and nitric acids; repetition of this procedure removes all the antimony and arsenic. The second ammonium sulphide precipitate is analysed for copper, lead, bismuth, iron, alumina, nickel, manganese, and zinc by any of the well-known methods, the antimony filtrate is acidified with acetic acid, the sulphides are removed, and lime and magnesia determined in the filtrate. Arsenic is determined in 5 g. of the original sample after evaporation to dryness with aqua regia and distillation of the residue with hydrochloric acid, potassium bromide, and hydrazine sulphate. A third portion of the sample is fused with pure sodium peroxide, the mass leached, the solution treated with alcohol and filtered, and the filtrate after acidification evaporated to dryness to remove silica; the solution is then treated with barium chloride for sulphur. Methods for the determination of carbon dioxide, gold, and silver are briefly described.

A. R. POWELL.

Ageing of cold-worked metals. J. GALIBOURG (Compt. rend., 1930, 190, 168—170).—The comparison between the mechanical properties of nickel bars annealed in hydrogen and aged at 15°, 175—180°, and 225—235° may be used to demonstrate the ageing effect produced at high temperatures (cf. B., 1929, 398). The effect is less marked than for steels. Curves are given showing the effects of successive heat treatments for 30 min. and 1 hr. at 220—230°.

J. GRANT.

Metallising of combustible materials [by the spraying process]. M. U. SCHOOP (Chem. Fabr., 1929, 537).—Paper and similar combustible materials may be metallised by means of a modified form of the Schoop pistol in which the metal in the form of a fine powder is blown into the expansion chamber of an oxyacetylene burner the jet of which is disposed between two broad tubes through which compressed air may be blown to moderate the temperature of the flame. Decorative articles and even bank-notes have been made in this way, using nickel or tungsten powder. It is essential, in order to get good adherence, that the individual particles of metal actually strike the paper while in the molten state, and that, to prevent destruction of the paper, the metal particles should be extremely fine.

A. R. POWELL.

Magnetic separation. ORTLEPP. **Electrodeposition of metals.** WARK.—See XI.

PATENTS.

Sintering apparatus. E. E. ELLIS (U.S.P. 1,737,007, 26.11.29. Appl., 5.5.28).—The travelling grate members of an automatic sintering apparatus, in which the material is fed on to an endless chain which passes through an ignition furnace, are coated with a slurry composed of milk of lime, clay, or sodium silicate to protect them from the destructive action of the gases.

A. R. POWELL.

Roasting furnace. A. THESEN, Assoc. of G. THESEN

(Swed.P. 78,778, 2.6.22. Norw., 6.6.21).—The furnace is heated by an internal electrical resistance spaced from the walls and floor of the heating chamber so that the change is heated only in those parts which are in contact with the air supply.

A. R. POWELL.

Shaft furnaces. METALLGES. A.-G., O. HUBMANN, and F. VOERKEL (B.P. 322,819, 15.9.28).—An apparatus for spreading material evenly, section by section, over the area of a rectangular blast-furnace comprises a conveyer running in one direction, which discharges at an adjustable point into other means of transport running at right angles, also to a variable distance. The second means is preferably a hopper with bottom discharge so that a measured quantity is discharged at each point.

B. M. VENABLES.

Revolving-drum furnace for roasting zinc blende and other sulphide ores. R. KESSLER (G.P. 455,394, 5.8.26).—The furnace comprises a horizontal, rotating cylinder with a series of heating flues disposed centrally along the axis, through which the hot gases from the grate pass forwards and backwards through the furnace. About the heating flues are arranged the roasting chambers through which is passed a stream of heated air from a recuperator attached to the firing end of the cylinder and rotating with it.

A. R. POWELL.

Ore-reducing furnace. CAYZER TIN SMELTING CO. (PROPRIETARY), LTD. (B.P. 323,159, 6.3.29. S. Afr., 29.1.29).—In a reducing furnace which has a rotatable hearth and a pervious bed, the normal working surface is maintained below the level of a surrounding rim over which, however, dross is discharged by a spade on the rabble arm into a concentric outer ring in which entrained metal may separate and drain into the bed. The hearth allows the formation of a distinct pool of molten metal, which reduces the tendency for metal to be entrained in the solid residue.

C. A. KING.

Recovery of volatile metals from ores, metallurgical products, and residues. METALLBANK U. METALLURG. GES. A.-G. (F.P. 631,708, 29.3.27. Ger., 12.4.26).—The material is smelted in a rotating furnace, the charge being fed directly into the melting zone and the volatilisation zone being lengthened in the direction of the charging hopper so as to prevent as far as possible volatilisation of the metal.

A. R. POWELL.

Tunnel furnaces [for annealing etc.]. BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of C. L. IPSEN and J. L. McFARLAND (B.P. 301,431, 29.11.28. U.S., 29.11.27).—One end of a horizontal gas-tight furnace is heated to the required annealing temperature, the remaining portion forming a cooling chamber for the articles while still in a neutral or reducing atmosphere. Mechanical means are provided for charging and moving the material under treatment.

C. A. KING.

Metal-hardening furnaces. J. S. RUSSELL, and MANCHESTER FURNACES, LTD. (B.P. 323,616, 1.12.28).—The furnace comprises an annular heating chamber with a rotating, horizontal, ring-shaped platform therein. An inlet for charging the furnace is provided at one side of the chamber and a discharge outlet on the opposite side adjacent to a dividing wall containing outlets from which the heating flames are directed into the furnace in the direction of the outlet.

A. R. POWELL.

Cast iron. BRIT. CAST IRON RES. ASSOC., A. L. NORBURY, and E. MORGAN (B.P. 323,076, 26.10.28).—A cast iron free from growth or scaling at high temperatures contains 4–10% Si and up to 10% Al, and the graphite content is present as finely-divided flakes.

A. R. POWELL.

Malleable iron. SOC. ANON. DES FORGES ET ACIÉRIES DE FIRMINY (F.P. 632,399, 26.7.26).—Cast iron is mixed with 0.5–10% of nickel or copper, which reduces the time required in making the material malleable by annealing and slow cooling.

A. R. POWELL.

Preparation of pure iron and steel. J. LEVOZ (F.P. 632,070, 9.7.26).—Pig iron is treated in such a way that the carbon and other impurities are burnt out completely and the resulting oxide inclusions are reduced by treatment with high-grade ferrosilicon, ferrotitanium-silicon, and, if necessary, aluminium, whereby the temperature rises to 1800°. Carbon in the desired amount is then added to form a pure steel free from sulphur and phosphorus.

A. R. POWELL.

Production of steel. GRANULAR IRON CO. (F.P. 632,888, 15.4.27).—Iron ores containing appreciable quantities of titania are heated with carbon at a temperature at which the iron oxide is reduced to metal without melting and the titania remains unchanged. The product is magnetically separated and the iron fraction which contains only a small proportion of titania is converted into steel in known manner.

A. R. POWELL.

Treating compound steel ingots. H. KURZ (B.P. 322,940, 30.1.29).—Steel ingots composed of two grades of steel, one cast round the other, are heated at a welding temperature and passed through the rolls at such a temperature and rate that after the final pass the temperature of the compound ingot is 1150–1100°. If necessary, intermediate heatings may be given during the rolling; in this case the heating gases are allowed to impinge only on the side of the ingot at which the low-carbon steel is placed.

A. R. POWELL.

Open-hearth [steel] refining process. F. KASPER (Austr.P. 108,417, 21.1.27).—The metal is treated successively with the various refining agents added in a finely-divided form, together with suitable slagging materials, and the slags are removed after every treatment. Thus silicon is removed first by the addition of lime, then manganese by the addition of ferric oxide and carbon, and finally sulphur and phosphorus by means of ferric oxide and lime.

A. R. POWELL.

Case-nitrication of steel. A. B. KINZEL, Assr. to ELECTRO METALLURG. CO. (U.S.P. 1,736,921, 26.11.29. Appl., 13.2.28. Renewed 24.6.29).—The steel article is heated at 700° in an atmosphere of hydrogen to effect surface decarburisation, then embedded in ferro-vanadium and heated at 900° in nitrogen to introduce about 0.3–0.5% V into the surface layer, and finally case-hardened with nitrogen by heating for 16–20 hrs. in ammonia at 500–600°. A surface hardness of over 900 Brinell units may be obtained by suitable adjustment of the above conditions.

A. R. POWELL.

Preparation of pickling baths [for iron or steel]. P. I. MURRILL, Assr. to R. T. VANDERBILT CO., INC.

(U.S.P. 1,736,934, 26.11.29. Appl., 22.11.28).—A mercaptobenzthiazole (25 pts.) is dissolved in 10% sodium hydroxide solution (75 pts.), a small quantity of the solution being added as an inhibitor to the usual sulphuric acid pickling bath.

A. R. POWELL.

[Inhibitor for] pickling baths for iron and steel.

E. A. KOLBE (Austr.P. 108,256, 1.7.25).—The inhibitor comprises the reaction product of sulphuric acid, oleum, bisulphates or mixtures of metal salts, and phosphoric acid, with vegetable or animal oils or fats or their decomposition, oxidation, or hardened products; 1–15% of the material is added to the usual pickling bath.

A. R. POWELL.

Pickling and cleaning steel and iron. Q. MARINO (F.P. 577,816, 26.2.24).—An electrolytic bath for removing grease and scale from iron comprises a solution of sodium or potassium hydroxide (41.1–1.2) containing starch, sugar, cellulose, tartaric acid, or other substance which forms oxalic acid during electrolysis. An iron anode is used and the bath is operated at 70–85° with 4–16 volts.

A. R. POWELL.

Protection of articles of iron, steel, and cast iron. G. NOBILLEAU and J. GUIPET (B.P. 297,832, 20.9.28. Fr., 29.9.27).—A sherardising mixture for coating iron and steel articles with zinc comprises 25% of zinc powder, 5% of barium carbonate, 2% of native zinc carbonate, and 60% of silica.

A. R. POWELL.

Protective coatings for metals. J. DUBOIS (F.P. 632,510, 9.4.27).—To protect metal surfaces from the action of air or sulphurous gases at high temperatures the hot surface is sprinkled with a powdered mixture of sand, clay, felspar, borax, and sodium hydroxide which fuses to a smooth enamel at a red heat.

A. R. POWELL.

Apparatus for spraying metal coatings. METALLISATOR BERLIN A.-G. (G.P. 451,787, 13.4.26. Addn. to G.P. 307,378; B., 1918, 705 A).—The apparatus comprises an inclined, rotating vessel of polygonal cross-section and having its lower part shaped like an inverted, truncated pyramid. The articles to be sprayed are rotated in the vessel while a stream of metal is sprayed vertically downwards on to the lowest part of the vessel from a pistol situated just above the uppermost part.

A. R. POWELL.

Cleaning of metal articles. CARRIER ENG. CO., LTD., A. FOWLER, and K. J. R. ROBERTSON (B.P. 322,858, 1.11.28).—The articles are passed through a vapour bath in such a way that condensation of the vapour occurs on the surface of the article, whereby the grease is removed. After passing through a drying chamber the articles are then passed through an acid pickling bath and finally through wash-water into a drying chamber, the whole operation being conducted automatically.

A. R. POWELL.

Cleaning of articles, particularly metallic goods. CARRIER ENG. CO., and K. J. R. ROBERTSON (B.P. 322,797, 13.6.28).—The greasy articles are sprayed with the vapour of a solvent for the grease under such conditions that the vapour condenses on the article and the resulting liquid washes off the grease. The

liquid obtained passes back to the boiler, where the solvent is distilled off for use again. The process is conducted continuously by the use of an automatic conveyer traversing the vapour bath and by providing refrigerating means for preventing the escape of vapour when the articles are removed from the bath.

A. R. POWELL.

Separation of rust from steel sand. ZOELLNER-WERKE A.-G. F. FARBEN- U. LACKFABRIKATION (G.P. 456,773, 5.6.27).—The steel sand used in sand-blasting may be separated from particles of rust by washing the mixture with a liquid, *e.g.*, petroleum, which does not affect the steel. The rust-oil suspension is allowed to settle, the clear liquor decanted off and used again, and the residual mud centrifuged to separate the remainder of the oil. The clean steel sand is dried and used again in the blast.

A. R. POWELL.

Production of iron alloys. AKTIEBOLAGET FERROLEGERINGAR (F.P. 569,029, 25.7.23. Swed., 26.7.22).—Briquettes composed of iron ore, the ore of the element to be alloyed with the iron, a non-carbonaceous reducing agent, *e.g.*, silicon or aluminium, and a binding material, *e.g.*, water-glass, magnesium chloride, or clay, are used as electrodes in an arc furnace, whereby a carbon-free alloy of iron is produced.

A. R. POWELL.

Compound wire-cable. FELTEN & GUILLEAUME CARLSWERK A.-G. (G.P. 457,636, 16.1.27).—Copper wires and wires composed of iron or steel alloyed with 0.08—1.1% Cu are employed. The latter become covered, on exposure to the atmosphere, with an adhesive red-brown protective coating.

J. S. G. THOMAS.

Austenite nickel-chromium steel alloys. F. KRUPP A.-G. (B.P. 305,654, 14.1.29. Ger., 9.2.28).—The steels contain 7—12% Ni, 18—25% Cr, and less than 0.07% C. The austenite is retained undecomposed even after tempering or welding and the steels are highly resistant to corrosion.

A. R. POWELL.

[Chromium-nickel-iron] alloys. SOC. ANON. COM-MENTRY-FOURCHAMBAULT & DECAZEVILLE (B.P. 302,249, 12.12.28. Fr., 12.12.27).—The alloys contain 21—49% Ni, 16—25% Cr, 1—5% Mn, 0—1% C, 2—8% W, and iron the remainder. In addition, 2—5% Mo may be present together with, or in place of, the tungsten and 0.2—1% V and/or Ti may be added as deoxidisers. Up to 10% of the nickel or iron may be replaced by cobalt.

A. R. POWELL.

[Hard tungsten-chromium-cobalt-iron] alloy. W. MÜLLER and OLGA PRINZESSIN ZUR LIPPE (B.P. 305,942, 11.2.29. Ger., 11.2.28).—An alloy of tungsten, chromium, and cobalt in the ratio 8:2:1 is added to iron so that the final alloy contains 10—55% Fe and 1—4% C. Up to one fourth of the tungsten may be replaced by molybdenum and up to one half of the cobalt by nickel; in addition, up to 4% of hardening elements other than carbon may be introduced.

A. R. POWELL.

Copper alloys. J. VERCRUYSSSE, F. POMIANKOWSKI, and E. MARNETTE (F.P. 629,924, 28.2.27. Belg., 5.3.26).—Fusion of copper with inorganic salts of an undisclosed

nature is claimed to produce an acid-resisting copper alloy.

A. R. POWELL.

Alloys for pressed brass bearings. METALLBANK U. METALLURG. GES. A.-G., Assees. of E. VADERS (G.P. 456,342, 2.12.22).—Alloys containing 58% Cu, 36—37% Zn, 0.6—1.5% Si, 2—2.5% Mn or 0—4% Fe, and 0—2% Al are claimed. The alloys are plastic at a red heat and their structure consists of hard mixed crystals in a soft matrix.

A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 323,353, 18.12.28).—The alloys consists of aluminium with 0.5—5.0% Cu, 0.1—1.7% Mg, 0.2—1.5% Ni, 0.6—1.5% Fe, 0.5—2.8% Si, 0—0.3% Ti, and 0—0.3% Mn.

A. R. POWELL.

Manufacture of aluminium-silicon alloys. SOC. D'ELECTRO-CHIMIE, D'ELECTRO-MÉTALLURGIE, ET DES ACIÉRIES ÉLECTRIQUES D'UGINE (B.P. 305,640, 8.2.29. Fr., 8.2.28).—Aluminium alloys with a high percentage of silicon such as are produced by the reduction of kaolin in the electric furnace are cooled slowly from 1000° to about 600° to cause part of the silicon to crystallise in the form of large crystals, then puddled, and subjected to pressure in the presence of a chloride flux to cause the eutectic aluminium-silicon alloy to flow away from the excess aluminium and other impurities. The liquated alloy may then be converted into silumin by a suitable adjustment of its composition. *E.g.*, an alloy containing 67.87% Al, 28.68% Si, 1.68% Fe, 0.4% Ti, and 0.62% C gives a fusible product containing 83.86% Al, 14.57% Si, 0.58% Fe, 0.1% Ti, and 0.1% C, together with a silicon-aluminium residue which is useful as a reducing agent in other metallurgical processes.

Treatment of [lead-zinc] ores. P. MONTONNIER (F.P. 631,932, 1.4.27).—Carbonate ores containing lead and zinc are leached with a solution of sodium hydrogen sulphate to obtain a neutral solution of zinc and sodium sulphates and in insoluble residue of lead sulphate. Electrolysis of the filtrate affords metallic zinc and a solution which can be used again in the process.

A. R. POWELL.

Heat-treatment of metallic products [nickel or cupronickel] for bright annealing etc. A. SADLER. From AMER. MOND NICKEL Co. (B.P. 323,496, 29.6.28).—The annealing is carried out in an electric furnace through which is passed a very slow stream of nitrogen containing 2—15% of hydrogen so that the residual gas contains practically no hydrogen and is suitable for cooling the annealed articles.

A. R. POWELL.

Apparatus for leaching ores, burnt pyrites, etc. K. PHILIPP (G.P. 455,627, 23.9.22).—The apparatus comprises a long trough subdivided into several sections, all of which are provided with a number of revolving blades of a design arranged to stir the solid thoroughly with the leach liquor and at the same time to move the solid countercurrent to the liquid flow, thus ensuring efficient leaching and washing in one continuous operation.

A. R. POWELL.

Leaching of metals from ores or metallurgical products by treatment with oxidising agents and solvents. SOC. METALLURG. CHILENA "CUPRUM" (G.P. 457,046, 9.8.23).—Oxidised lead, zinc, and copper ores

are leached with brine to remove lead, then roasted with nitre, and again leached with brine to remove silver. The residue is leached with dilute sulphuric acid to dissolve copper and zinc oxides. A. R. POWELL.

Apparatus for treatment of ores and metallurgical products, especially lead and silver materials with concentrated chloride solutions. SOCIEDAD MINERA "CUPRUM" (G.P. 448,148, 7.12.24).—The apparatus comprises two cylindrical vessels with conical bottoms and wide central tubes into which is injected a suspension of the ore in the leach liquor. This injector action effects thorough agitation of the mixture, which then overflows continuously into a series of eight settling chambers arranged in a band around the outside of each agitation vessel. The underflow from the settling chambers passes back to the agitation vessel and the underflow from the first vessel passes to the second and thence to filters. The overflow from each vessel passes to a series of precipitation tanks in which the solution is freed from lead and silver by treatment with iron and the chloride leach liquor is regenerated. A. R. POWELL.

Recovery of antimony from residues. HÜTENWERKE TEMPELHOF A. MEYER (G.P. 456,703, 20.1.26. Addn. to G.P. 431,984. Cf. B.P. 264,139; B., 1927, 528).—The method described in the prior patent is applicable to the recovery of antimony from ashes, residues, etc.; the material is fused with sulphur equivalent to the impurities present, whereby a crude regulus is obtained below a layer of matte. A. R. POWELL.

Preparation of pure cerussite from zinc-lead ores, purification of zinciferous materials containing lead sulphate, and conversion of the latter into chrome-yellow. E. STERKERS (F.P. 554,102, 17.11.21, and Addn. F.P. 27,152, 12.1.22).—(A) The ore is roasted to convert the lead into sulphate, any zinc sulphate formed is leached out with water, and the residue is digested with a concentrated solution of sodium thiosulphate which dissolves lead sulphate, leaving a residue of zinc oxide. The clear solution is treated with the calculated quantity of sodium carbonate or chromate to precipitate lead carbonate or chromate and regenerate the thiosulphate solution for use again. (B) The ore is first subjected to an air-levigation process in a vessel fitted with a rotating blade of corkscrew shape to separate the greater part of the blende from the galena. A. R. POWELL.

Obtaining precious metals [gold and silver] from ores. L. MELLERSH-JACKSON. From G. BÜRG (B.P. 323,466, 29.8.28).—Ores containing finely-divided gold and silver are heated at 600–700° to coagulate the precious metal particles and then subjected to amalgamation or other known treatment for the recovery of the values. A. R. POWELL.

Joining of copper to difficultly-fusible hard metals [e.g., tungsten]. GLAS-GLEICHRICHTERBAU A.-G. (A.G.G.) (Swiss P. 123,770, 26.8.26).—To join a copper wire to a tungsten rod or sheet, a sheath of nickel is melted over one end of the tungsten, the copper wire is placed in contact with the nickel and heated until it alloys therewith. A. R. POWELL.

Soldering of aluminium. J. DIRNAY (F.P. 574,673, 12.3.23).—The carefully cleaned surfaces are rubbed with tin amalgam, coated with a small quantity of grease to prevent oxidation, joined together, and heated to expel the mercury and effect a joint. A. R. POWELL.

Removal of excess solder from soldered joints. H. DITZ and R. MAY (G.P. 457,456, 20.5.27. Czechoslov., 27.5.26).—The articles are placed in hydrochloric acid, with or without the addition of sulphuric acid or salts, until the excess solder has dissolved. A. R. POWELL.

Soldering fluxes [for leading-in wires of electric lamps]. J. BARNETT, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 323,392, 1.2.29).—A suspension of 10 pts. of colophony and 1 pt. of ammonium chloride in 90 pts. of tetrachloroethane or of 60 pts. of colophony and 5 pts. of ammonium chloride in 100 pts. of *n*-propyl alcohol is claimed. A. R. POWELL.

Production of soldering mixtures [fluxes]. G. W. F. F. KNOTH (B.P. 309,029, 23.3.29. Ger., 3.4.28).—A fluxing paste comprises powdered tin solder, a binder, e.g., petroleum jelly, and an amine, e.g., hexamethylenetetramine. L. A. COLES.

Production of regular coatings of metals on articles by cathodic disintegration. W. A. F. PFANHAUSER (G.P. 453,387, 23.10.26, and 456,772, 5.11.26).—(A) The article to be coated is backed with an artificially cooled surface operated from outside the vacuum vessel; by this means paper, textiles, and the like can be coated with a fine film of metal. (B) A heat-conducting material is placed between the article and the cooling surface to prevent deposition of the coating metal on the latter. A. R. POWELL.

Production of electrolytic deposits [of metals or alloys]. H. SCHMIDT (G.P. 457,590, 17.8.26).—A fused salt mixture containing compounds of at least two different metals dissolved therein is electrolysed first at a low current density to deposit one of the metals, then at a higher current density to deposit both metals simultaneously. The process is suitable for the deposition of smooth coatings of nickel, chromium, or iron, either alone or in the form of hard alloys. A. R. POWELL.

Producing electrolytic deposits of chromium which adhere well and penetrate well into any recesses. W. G. POETZSCH (B.P. 296,988, 6.9.28. Ger., 10.9.27).—The bath contains 450 g. of chromic acid, 5 g. of chromic sulphate, and 3.5 g. of magnesia or alumina per litre. A current density of 3–10 amp./dm.² at 3 volts is employed in plating. A. R. POWELL.

Chromium plating and baths therefor. TERNSTEDT MANUF. Co., Assocs. of J. F. K. McCULLOUGH and B. W. GILCHRIST (B.P. 308,771, 12.12.28. U.S., 29.3.28).—The bath comprises a concentrated solution of an alkali chlorochromate with a small proportion of chromium chromate and free acid. Such a bath may be prepared by dissolving 480 g. of sodium dichromate in 1 litre of water and adding 308 g. of concentrated hydrochloric acid, together with 7.5 g. of oxalic acid to reduce part of the chromic acid to chromium

trichloride, which then reacts to form chromium chromate. The bath is operated at 20–70° with 4–13 volts.

A. R. POWELL.

Apparatus for melting electrolytic tin. A. DOSSMANN (G.P. 455,251, 30.12.25).—The cathodes are placed vertically in a cylindrical or rectangular chamber provided with an inclined tubular portion in the lower end for draining off the molten tin. This tube is provided with air-tight doors on either side for cleaning purposes.

A. R. POWELL.

Electrolytic deposition of silver without external current. A. BARTH (G.P. 455,498, 3.11.25. Addn. to G.P. 393,964; B., 1924, 753).—The silver-rich solution obtained by anodic dissolution of impure silver in the anode compartment of a divided cell is passed through the cathode compartment of a cell with an impure silver auxiliary anode, whereby the greater part of the dissolved silver is precipitated. The effluent from this cell is passed into the cathode chamber of another cell having a copper auxiliary anode in order to precipitate the remainder of the silver.

A. R. POWELL.

Alloys [containing iron, chromium, molybdenum, and uranium]. W. BORCHERS and R. W. STIMSON (U.S.P. 1,740,678–9, 24.12.29. Appl., 14.4.24. U.K., [A] 24.4.23, [B] 27.4.23).—See B.P. 223,617 and 223,622; B., 1925, 13.

Production of beryllium. W. KROLL (U.S.P. 1,740,857, 24.12.29. Appl., 26.9.27. Ger., 28.9.26).—See B.P. 278,723; B., 1928, 336.

Production of metal alloys and of articles made thereof. W. SCHROEDORFF (U.S.P. 1,742,417, 7.1.30. Appl., 18.7.27. Ger., 21.7.26).—See B.P. 274,866; B., 1928, 932.

Metallic alloy. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,740,880, 24.12.29. Appl., 2.11.27. U.K., 6.11.26).—See B.P. 284,789; B., 1928, 271.

Metallising the surfaces of insulating bands. E. PFIFFNER, ASST. to RADIO PATENTS CORP. (U.S.P. 1,741,477, 31.12.29. Appl., 25.3.26. Ger., 2.1.26).—See B.P. 263,728; B., 1927, 390.

Lining of metal pipes or cylinders with rubber or like material. C. MACINTOSH & Co., LTD., and H. C. YOUNG (B.P. 323,054, 29.9.28).

[Varnishing] ovens for heat treatment of sheet material. PREMIX GAS PLANTS, LTD., and A. DOCKING (B.P. 323,023, 14.9.28).

Removal of dust from gases (G.P. 456,612).—See I. Metallurgical charcoal (F.P. 632,623). **Producer gas for open-hearth furnaces** (G.P. 455,595).—See II. Melting furnaces (B.P. 323,291). **Annealing furnaces** (B.P. 304,646). **Electrodeposition** (G.P. 456,840). **Protection of metal surfaces** (U.S.P. 1,736,986–7). **Designs on metals** (G.P. 457,333). **Magnetic cores** (B.P. 313,584).—See XI. **Lacquer for metal surfaces** (B.P. 323,525).—See XIII. **Coating metal with rubber** (B.P. 302,935).—See XIV.

XI.—ELECTROTECHNICS.

Alternating current in magnetic separation. J. A. L. ORTLEPP (J. Chem. Met. Min. Soc. S. Afr., 1929,

30, 99–128).—The magnetic material obtained from the ordinary Wetherill type of dry magnetic separator always contains entrained non-magnetic material, especially when the particles have a high magnetic retentivity and tend to bunch together. Hence, in the magnetic separation of roasted pyrites from a concentrate containing cassiterite, a somewhat high proportion of the latter may be lost in the magnetic material, re-treatment of which on the separator rarely leads to any further recovery. By passing the magnetic material, however, in a thin layer on an endless belt over a series of V-shaped magnetic poles excited by alternating current, repulsion of the magnetic material occurs, and it forms two bands, one on each side of the poles, leaving a band of non-magnetic tinstone between; the magnetic material may be picked up by an ordinary direct-current magnet at the far end of the belt, and is then relatively free from tinstone. The theory of this behaviour is discussed at some length, and some results obtained with a separator working on this principle on a South African tin concentrate are recorded.

Energy considerations in the electrodeposition of metals. I. W. WARK (J. Soc. Chem. Ind. Victoria, 1929, 29, 251–273).—The factors determining the energy efficiency of electrolytic processes are reviewed and the application of these general electrochemical principles to nickel plating and the electrolytic refining and extraction of copper is indicated.

H. J. T. ELLINGHAM.

Determination of carbon in steel. KLINGER and FUCKE. **Electrolytic copper.** KAMEYAMA and NODA.—See X.

PATENTS.

Operation of tungsten- and molybdenum-resistance furnaces. W. C. HERAEUS GES.M.B.H., Assees. of E. KILLINS (G.P. 457,516, 3.11.25).—Furnace heating-tubes wound with tungsten or molybdenum wire are surrounded by a jacket filled with methyl alcohol vapour.

J. S. G. THOMAS.

Electric [induction] melting furnaces. ELECTRIC FURNACE Co., LTD., and D. F. CAMPBELL (B.P. 323,291, 17.10.28).—Two or more electrodes are inserted at the top of the furnace and are arranged so that the slag is heated either by radiation from arcs between the electrodes or by arcs between the electrodes and the bath; the metal of the bath is heated by currents induced by a coil surrounding the bath. If desired, electrodes may be inserted below the surface of the slag.

J. S. G. THOMAS.

Electric [annealing] furnaces. HERAEUS VACUUM-SCHMELZE A.-G., and H. HIEMENZ (B.P. 304,646 and 323,271, 21.9.28. Ger., [A] 23.1.28).—(A) The bottom of the niches or recesses at the bottom of the furnace receiving the heating element is formed of stones terminating in an inverted V, on the upper edge of which the heating element is placed; the diameter of the heating element and the spacing of the side walls of the niches or recesses are arranged so that the heating element is retained without the use of a separate device. (B) The side walls are constructed of stones, inclined to the horizontal, some of which are set back so as to form recesses having inclined sides in which the heating

elements are placed and in which dust and oxide scales collect out of contact with the heating element.

J. S. G. THOMAS.

Apparatus for rapid heating of electrical salt-bath furnaces. ALLGEM. ELEKTRICITÄTS-GES., Assees. of V. PASCHKIS (G.P. 455,696, 22.11.25).—A cage having vertical rods or tubes, made of metal or ceramic material which is a good conductor of heat and electricity, is immersed in the bath. Alternatively, a hollow cylinder may be so employed.

J. S. G. THOMAS.

Electrical precipitation of dust from gases. GEN. ELECTRIC CO., LTD., and F. FORREST (B.P. 323,186, 23.8.28).—Gas is subjected to an electrical dust-precipitation process in a heat exchanger, where it gives up heat which may be used, *e.g.*, to preheat air for combustion.

J. S. G. THOMAS.

Prevention of explosions in electric gas-purification plants. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of F. MÜLLER (G.P. 455,872, 14.8.25).—An electrical device operated by a contact thermometer and selenium cell is provided in the precipitation chamber which automatically works a cut-out switch which stops the discharging conveyer immediately the dust begins to ignite within the chamber.

A. R. POWELL.

Galvanic cell. A. PETIT (F.P. 633,079, 22.4.27).—Metallic salts corresponding to a lower state of oxidation, more especially ferrous chloride and sulphate, are employed as electrolyte; the positive electrode is composed of carbon; and magnesium, zinc, aluminium, lead, copper, or, more especially, iron is used as negative electrode. The respective *E.M.F.* of the cell when the various negative electrodes are employed are: 2.25, 1.52, 1.37, 0.82, 0.6, and 0.97 volts.

J. S. G. THOMAS.

[Electrolyte for] secondary electric cells. E. ALMEIDA, and ALMEIDA ACCUMULATORS, LTD. (B.P. 323,191, 21.9.28).—An aqueous solution containing, per litre, zinc chloride (200–300 g.), potassium bromide (400 g.) and sufficient hydrochloric acid to give a slight acid reaction to Congo-red is employed. Zinc bromide (320 g.) or potassium chloride (200 g.) may also be added, together with a small quantity of mercuric chloride, if desired.

J. S. G. THOMAS.

Electrodes [anodes] for electric batteries, electrolytic cells, etc. L. A. LEVY, and ALMEIDA ACCUMULATORS, LTD. (B.P. 323,192, 21.9.28).—Silicon-iron alloy containing about 14–15% Si and preferably having a suitable micrographic structure (*e.g.*, Tantiron or Ironac) is employed. [Stat. ref.]

J. S. G. THOMAS.

Electrical accumulators. COMP. GÉN. D'ELECTRICITÉ (F.P. 631,692, 28.3.27. Ger., 17.4.26).—To the active material incorporated in the negative plates, 0.1–1% of vanadium, nickel, cobalt, tungsten, molybdenum, or copper, in the form of metallic powder, oxide, or salt, is added so that the voltage necessary to complete the charging of the accumulator is reduced.

J. S. G. THOMAS.

Alkaline accumulator. COMP. GÉN. D'ELECTRICITÉ (F.P. 633,211, 14.4.27. Swed., 16.4.26).—Accumulators in which nickel oxide is the active material and a

solution of sodium hydroxide the electrolyte are rendered more permanent by the addition of 1–10 g. of potassium cyanide to 1 litre of electrolyte.

A. R. POWELL.

Accumulator with an alkaline electrolyte. N. MALICHATKA (G.P. 456,423, 22.4.26).—A horizontal sheet-iron grating provided with short, vertical projections and coated with zinc amalgam containing not more than 15% Zn, and a carbon block covered with manganese dioxide and graphite as depolariser serve as negative and positive electrodes, respectively.

L. A. COLES.

X-Ray tubes. SIEMENS-REINIGER-VEIFA (GES. F. MEDIZ. TECHN. M.B.H.) (F.P. 630,410, 7.3.27. Ger., 3.4.26).—The anticathode is provided with a cover of a substance impermeable to X-rays, *e.g.*, copper, and containing at one side a "window" made of a substance which is permeable to X-rays but impermeable to electrons, *e.g.*, beryllium. The cathode is surrounded by a screen of iron which retains X-rays, and which also surrounds the lower part of the copper cover of the anticathode. This arrangement prevents the bombardment of the glass walls of the tube by electrons and the resulting deterioration of the vacuum.

A. R. POWELL.

Luminous electric-discharge tube. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 315,757, 13.5.29. Ger., 17.7.28).—A separating member composed of material permeable, when heated, to hydrogen, *e.g.*, palladium, is mounted in a connecting piece between the luminous tube (filled with neon and a small proportion of hydrogen) and a side tube containing hydrogen.

J. S. G. THOMAS.

Incandescence cathode [discharge] tube. SIEMENS & HALSKE A.-G., Assees. of H. GERDIEN (G.P. 455,364, 18.5.23).—An incandescence cathode is composed of a compound of hafnium, emitting very light electrons and therefore requiring very little heating.

J. S. G. THOMAS.

Incandescence cathode. E. ROUGE (Austri.P. 108,343, 26.6.26. Belg., 14.6.26).—Thin filaments of non-volatile metals, *e.g.*, platinum, platinum-iridium, heated at about 450–600° are coated, *e.g.*, by spraying, with a peroxide of an alkaline-earth metal, or, alternatively, with a compound which is subsequently converted into peroxide. After mounting the filaments in a tube, the peroxide is converted into oxide, oxygen formed is removed by the vacuum pump, and the tube is sealed off.

J. S. G. THOMAS.

Cathode for electron-discharge devices. SOC. DES ETABL. INDUST. DE E.C. ET DE ALEXANDRE GRAMMONT (B.P. 303,369, 29.12.28. Fr., 31.12.27).—A filament or strip of platinum or palladium is made porous by heating in hydrogen and is then impregnated with a substance of high electron-emission power, *e.g.*, barium, thorium, caesium, or their salts. Gas is removed from the impregnated material by heating *in vacuo*. If desired, the palladium or platinum may be used in the form of a coating on a refractory metal, *e.g.*, tungsten.

J. S. G. THOMAS.

Coating of electrodes with sponge nickel. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of P. ALBRECHT

(G.P. 455,821, 4.8.26).—The electrode is plated with nickel sponge at a current density of 1.6 amp./dm.² in a solution made by dissolving 1.7 g. of nickel chloride in 64 c.c. of 25% ammonia and adding 500 c.c. of water. The electrode is preferably previously plated with a smooth coating of nickel. A. R. POWELL.

Production of electrolytic deposits of varying thickness. P. BRUCKMANN & SÖHNE A.-G. (G.P. 456,840, 30.3.26).—The current density at various parts of the cathode is varied by connecting leads to these parts through variable resistances. A. R. POWELL.

Protection of metal surfaces against incrustation and corrosion. W. THALHOFER, ASST. to A.-G. F. CHEM. IND. (IN LIECHTENSTEIN) (U.S.P. 1,736,986—7, 26.11.29. Appl., [A] 19.8.25, [B] 15.12.26. Austr., [A] 21.8.24, [B] 2.4.25. Renewed [A] 15.10.29).—(A) An interrupted pulsating current, the form characteristic of which has been distorted by means of reactance devices, is applied to the metal, *e.g.*, a boiler, to be protected. (B) An electric potential having a frequency of the order of magnitude of radio frequencies is applied to the boiler, which is connected with a second boiler or similar apparatus in such a way that the boilers form the two plates of an air condenser. A. R. POWELL.

Production of etched and precipitated designs [on metals] by electrolysis. J. RIEDER (G.P. 457,333, 15.8.26).—The design is stamped on the metal, *e.g.*, razor blades, in a paste of ferric chloride and tragacanth, and the article is then made the anode for a few seconds in an alkali hydroxide solution. Alternatively, a paste of gum arabic and silver or copper cyanide may be used for stamping and the electrolysis made in dilute cyanide solution, whereby the design appears in silver or copper. A. R. POWELL.

Magnetic [core] materials. ELECTRICAL RES. PRODUCTS, INC., ASSEES. of G. W. ELMEN (B.P. 313,584, 25.9.28. U.S., 15.6.28).—A magnetic core built up of alternate laminations of hard steel and Permalloy containing 30–85% Ni with air gaps between the layers is claimed. A. R. POWELL.

Preparation of magnet cores from a mixture of finely-divided iron and an insulating material. DEUTS. KABELWERKE A.-G. (G.P. 455,542, 17.4.26).—Finely-divided iron is mixed with 6–8% of its weight of ferromagnetic chromic oxide obtained by calcining chromic acid. A. R. POWELL.

Manufacture of electrically insulating materials. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 309,506, 11.4.29. Ger., 11.4.28).—Asbestos fabric or paper is coated with superimposed layers of lacquer or varnish, some of which have a thickness less than 0.01 mm., and dried at 200–270°. J. S. G. THOMAS.

Electrical insulating and impregnating material. J. E. G. LAMOUSSE (F.P. 633,171, 12.8.26).—Metallic resins, especially zinc resin, the m.p. of which is reduced by the addition of substances such as amides, are used. These materials retain a high dielectric value even when heated, and are especially suitable in the construction of condensers. J. S. G. THOMAS.

Dielectric material for submarine cables. STANDARD TELEPHONES & CABLES, LTD., AND M. C. FIELD (B.P. 323,038, 14.9.28).—A mixture of rubber, partly synthetic, and at least 30% of a molten mineral wax, *e.g.*, paraffin wax, m.p. about 60°, is allowed to swell, then masticated, and finally heat-treated at 80–100°.

J. S. G. THOMAS.

Improving the contact between moving electrodes and the bus-bars. LANGBEIN-PFANHAUSER-WERKE A.-G. (G.P. 455,134, 4.6.27).—The rail on which the moving contact runs is sprayed from a perforated tube with a solution containing acids or salts which dissolve the oxides which form on it. A. R. POWELL.

Electric furnace. H. N. SHAW, ASST. to GLOBAL CORP. (U.S.P. 1,742,286, 7.1.30. Appl., 3.9.25).—See B.P. 257,949; B., 1928, 338.

[Building of high-tension] electric insulators [with embedded conductors]. MICANITE & INSULATORS CO., LTD., and G. E. HAEFELY (B.P. 323,343, 8.12.28).

Electric batteries. M. WILDERMAN (B.P. 323,228, 25.9.28).

[Rotational method for] cathode-ray sterilisation. SIEMENS-RHEINIGER-VEIFA GES. F. MEDIZIN. TECHNIK M.B.II. (B.P. 310,806, 29.4.29. Ger., 30.4.28).

Gas-tight vacuum vessels (G.P. 456,719).—See I. **Regulation of gas-producer plants** (B.P. 302,607). **Insulating oils** (B.P. 297,798 and 321,187). **Transformer etc. oils** (Austr.P. 108,434 and 108,438). **Conversion of oils** (F.P. 632,293).—See II. **Purification of aliphatic alcohols** (G.P. 452,066).—See III. **Oxidation of cerous sulphate** (G.P. 431,308). **Hydrogen peroxide** (F.P. 634,195).—See VII. **Pickling etc. of steel and iron** (F.P. 577,816). **Iron alloys** (F.P. 569,029). **Soldering fluxes** (B.P. 323,392). **Coating of metals on articles** (G.P. 453,387 and 456,772). **Electrodeposits of metals or alloys** (G.P. 457,590). **Chromium** (B.P. 296,988 and 308,771). **Melting of electrolytic tin** (G.P. 455,251). **Deposition of silver** (G.P. 455,498).—See X. **Tanning** (B.P. 294,996—7).—See XV.

XII.—FATS; OILS; WAXES.

Grouping of fatty oils, with special reference to olive oil. E. R. BOLTON and K. A. WILLIAMS (Analyst, 1930, 55, 5–11).—The unsaponifiable matter of the oil is prepared by saponifying 2–2.5 g. with 0.5N-alcoholic potash, titrating with 0.5N-hydrochloric acid, adding 5 c.c. of 0.5N-sodium hydroxide, and extracting three times or more with 30–40 c.c. of petroleum spirit. The combined extracts are washed with 20 c.c. of 0.05N-sodium hydroxide followed by 20 c.c. of water, the solvent is evaporated, and the unsaponifiable matter dried and weighed. This is dissolved in 5 c.c. of chloroform and enough pyridine sulphate-bromide reagent added to leave an excess of unabsorbed halogen approximately equal to the amount absorbed. The reagent is prepared by mixing (i) 8 g. of bromine with 20 c.c. of glacial acetic acid, and (ii) 10 g. of concentrated sulphuric acid with 8 g. of pyridine and 20 c.c. of glacial acetic acid, and then mixing (i) and (ii), cooling, and diluting to

1 litre with glacial acetic acid. After 5 min. in the dark, 5 c.c. of 10% potassium iodide solution are added and 40 c.c. of water, and the liberated iodine is titrated with 0.05*N*-sodium thiosulphate. A blank test is run simultaneously. Duplicate determinations did not vary more than about 3% of the observed figure. The oils and fats examined fall into four groups, the iodine values of which are: (a) 64—70, containing the animal fats and the kernel oils of the *Palma* (coconut, palm-kernel, babassu); (b) 90—96, comprising the fish and marine-animal oils (containing less than 2% of unsaponifiable matter) and cacao butter; (c) 117—124 (the largest group), containing the vegetable oils and fats; and (d) 197—206, containing olive oil only. Soya-bean oil must first be refined and bleached, probably owing to the presence of unsaturated matter other than sterols associated with the colouring matter. As little as 10% of foreign oil will bring the figure for olive oil outside the limits, thus enabling it to be distinguished from tea-seed oil in a manner hitherto impossible. D. G. HEWER.

Boron compounds in foods. DODD.—See XIX.

PATENTS.

Production of oils and fats such as whale oil, liver oils. P. M. HEYERDAHL (N.P. 42,639, 7.12.23).—The material is treated with water or steam, the oil and water being withdrawn through a perforated opening and side-tube, and the residual material through a trap in the floor of the vessel. E. LEWKOWITSCH.

Production of oils and fats from animal [fish] material. S. THUNE and E. F. HEYERDAHL (N.P. 43,701, 16.7.24).—The material is reduced and milled in the presence of steam or hot water.

E. LEWKOWITSCH.

Splitting of oils and fats under pressure. VEREIN. CHEM. WERKE A.-G., F. L. SCHMIDT, and E. HOYER (F.P. 630,389, 5.3.27).—Palm oil (2000 kg.) is heated in an autoclave at 130—140° by direct steam along with 12 kg. of Twitchell's reagent, 1000 kg. of water, and 10 kg. of concentrated sulphuric acid for 2—3 hrs. A little steam is allowed to escape all the time to maintain the quantity of fluid constant. After settling, the glycerin water is removed and the oil, 85% split, is similarly treated again for 3 hrs., whereby further splitting (to 95%) occurs. The sweet water from the second treatment is used for a fresh charge. Great saving of time is claimed. W. J. BOYD.

Dehydration and heating of oils and fats. METALL-BANK U. METALLURG. GES. A.-G. (G.P. 455,825, 15.12.26. Addn. to G.P. 419,868. Cf. B.P. 275,536; B., 1927, 821).—The gases and vapours leaving the vessel in which the materials are heated are compressed and used again for circulating the oil. A. B. MANNING.

Sulphonation of fatty acids and fatty acid derivatives. N.V. CHEM. FABR. "SERVO," and M. D. ROZENBROEK (B.P. 293,690, 2.7.28. Holl., 9.7.27).—Sulphuric esters of fatty acids etc. which are soluble in water, salt solution, and dilute acid, and resistant to the action of alkaline-earth salts and alkaline lye, are prepared by sulphonating the material with concentrated or fuming sulphuric acid, chlorosulphonic acid, or mixtures of these, in the presence of substantial

amounts of oxides, chlorides, or oxychlorides of phosphorus. Concentrated acetic acid (80—100%) as a thinning medium may be added with the phosphorous compound to facilitate intensive sulphonation of materials giving very viscous products. The reaction products are washed or neutralised in the usual manner.

E. LEWKOWITSCH.

Apparatus for continuous production of liver oil etc., with simultaneous removal of solid matter. R. ROED (N.P. 42,912, 2.9.25).—The material is reduced in a rotating, grooved cylinder in the presence of steam; the oil is separated from the mash in another vessel through slits in an inner, hollow cylinder.

E. LEWKOWITSCH.

Decolorisation of liver oils with retention of vitamins and improvement of taste. I. G. FARBEN-IND. A.-G., and E. BERNHARD (G.P. 452,646, 23.6.25).—The oil is intimately mixed with active charcoal at moderately high temperature, in the absence of oxidising conditions. Such charcoal is obtained by treatment of carbonaceous matter, previously impregnated, if desired, with oxides, hydroxides, or salts, in a revolving retort, so that it is exposed to the action of the gases burning preferably with a reducing flame with limited supply of oxygen, whereby admixture of extraneous gases may occur, or so that it is heated by the flaming burning of the material itself rather than by furnace flames.

W. J. BOYD.

Extraction of [animal or vegetable] oils from materials containing them. K. HOLTER and S. THUNE (N.P. 43,031, 26.11.25).—The material is treated with tri- or tetra-chloroethylene or tetra- or penta-chloroethane and the mixture is centrifuged to remove solids.

A. R. POWELL.

Manufacture of polishing materials [in powder form]. J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 323,279, 4.10.28).—A mixture of waxes, resins, etc. containing a substantial proportion of bleached montan wax, or conversion product therefrom, obtained, e.g., by amidation, esterification, saponification (cf. B.P. 296,145; B., 1929, 804) and colouring matter, as desired, is melted and sprayed. E. LEWKOWITSCH.

Linoleum from cellulose derivatives (B.P. 289,063).—See V. **Recovery of oily substances** (B.P. 323,094).—See XXIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Drying of exterior paints under various weather conditions and over different woods. F. C. SCHMUTZ and F. C. PALMER (Ind. Eng. Chem., 1930, 22, 84—87).—Five typical ready-mixed paints were applied separately to glass and various wood panels and their drying times observed in specially designed cabinets permitting the reproduction of different weather conditions. Eight different weather extremes were considered and their equivalents in terms of temperature, humidity, and light are tabulated. Excessive humidity causes a retardation of the drying rate at higher temperatures, particularly in the presence of light (which, in general, has an accelerating action on the setting-up process). Low temperature has by far the greatest retarding influence on the drying process, even tending

to outweigh the increase in effectiveness of light due to decrease in humidity and temperature. Of the different woods studied, cypress and redwood were found to accentuate the retarding tendencies of humidity and low temperature. S. S. WOOLF.

Nitrocellulose lacquers. H. WOLFF and G. ZEIDLER (Farben-Ztg., 1930, 35, 750—752).—Parallel types of nitrocellulose lacquers containing ester gum and four different types of shellac as resin components, Sipalin-MOM and -AOM, tritoyl phosphate, and butyl phthalate as plasticisers, and solvent mixtures suitable for ester gum and shellac, respectively, were tested for flexibility and adhesion, sand-abrasion resistance, tensile strength, and rust prevention on exposure. The results, which are tabulated and discussed, show several anomalies, and it appears that absolute evaluation of plasticisers and resins cannot be achieved by this method. Nevertheless, by examination of the results, it was demonstrated that one particular type of shellac and Sipalin-AOM in conjunction showed general superiority, whilst ester gum and butyl phthalate was the poorest combination. S. S. WOOLF.

Physical properties of lacquer liquids. A. W. VAN HEUCKEROTH (Amer. Paint and Varnish Manufs.' Assoc., Jan., 1930, Circ. 359, 74—172).—A summary of the physical properties and uses of a comprehensive range of solvents, diluents, plasticisers, etc. used in lacquers. S. S. WOOLF.

Resins. III. E. STOCK (Farben-Ztg., 1930, 35, 800; cf. B., 1929, 483).—Analytical and solubility details of Hiroe dammar, Borneo dammar, and a range of proprietary "rubber resins" are tabulated. S. S. WOOLF.

PATENTS.

Manufacture of litharge and red lead. R. S. CARRERAS (B.P. 323,070, 20.10.28).—Lead carbonate obtained by electrolysis is decomposed and oxidised in a two-stage furnace comprising an upper chamber operating at 320° and a lower chamber at 500—600°. The litharge produced in the upper chamber may be discharged as such or passed to the lower for conversion into orange minium. S. S. WOOLF.

Recovery of by-products or waste resulting from the manufacture of dry [zinc oxide] pigments for production of oil paints, enamel paints, etc. H. REINHARD (B.P. 321,675, 7.8.28).—Coarse by-products or waste in the manufacture of zinc oxide (*e.g.*, "drop oxide") are dispersed in oily vehicles for pigments, the mixture is centrifuged, and the proportion of fine pigment contained therein is recovered in the form of an oily paste or fluid of desired consistency. The quantity of the dispersing fluid and the viscosity of the mixture may be regulated in order that further recovery is possible. The residue from the first centrifuging may be ground and treated with more oil vehicle and subjected to further controlled centrifuging. S. S. WOOLF.

[Lacquer for] protection of metal surfaces against corrosion. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 323,525, 23.8.28).—The lacquer comprises a solution of a cellulose ether, a heavy-metal salt of an organic acid of high mol. wt., and a softening

agent in an organic solvent, *e.g.*, benzylcellulose 15 pts., copper naphthenate 5 pts. (or ferric stearate 2 pts.), tricresyl phosphate 6—9 pts., benzene 50 pts., xylene 50 pts., and alcohol 10 pts. A. R. POWELL.

Resinous compositions and varnishes made therefrom. BAKELITE CORP. (B.P. 293,453, 22.6.28. U.S., 7.7.27).—The phenolic complex obtained by the reaction between a phenol and a fatty oil, preferably tung oil, in the presence of a catalyst, *e.g.*, phosphoric or boric acid, is heated with a hardening agent containing a methylene linking, *e.g.*, formaldehyde, a non-phenolic resin (colophony or copal) being present in sufficient proportion to function as a blending agent, *i.e.*, more than 20% by wt. of the phenol present. The product is soluble in turpentine, toluene, etc., and gives quick-drying varnishes. S. S. WOOLF.

Manufacture of resinous compositions. H. WADE. From BAKELITE CORP. (B.P. 323,036, 13.9.28).—A permanently fusible or non-reactive natural or synthetic resin (preferably a phenol-methylene product) is heated with a phenol-furfuraldehyde reaction product to a stage where final hardening will take place rapidly under hot-pressing, the resin being completely dehydrated without gelatinising. A methylene-containing hardening agent, *e.g.*, hexamethylenetetramine, is then incorporated to convert the fusible phenol-methylene component of the mixture into potentially reactive composition. The initial condensation of the phenol-furfuraldehyde resin may be effected in the presence of the non-reactive resin, if desired. S. S. WOOLF.

Manufacture of resinous phenolic condensation products. H. WADE. From BAKELITE CORP. (B.P. 323,275, 2.10.28).—A permanently fusible phenol-methylene condensation resin is converted into a potentially reactive resin by first incorporating additional phenolic material, *e.g.*, so that the phenol-methylene ratio of the mixture is 9—12:6, and then adding a methylene-containing hardening agent, *e.g.*, hexamethylenetetramine, formaldehyde, in such quantity that the resulting methylene-phenol ratio of the mixture is greater than that of the initial condensation product. The final mixture preferably contains equimolecular proportions of phenol and methylene. S. S. WOOLF.

Manufacture of reaction products of natural and phenolic resins. A. AMANN, Assr. to CHEM. FABR. DR. K. ALBERT GES.M.B.H. (U.S.P. 1,736,757, 19.11.29. Appl., 20.10.26).—Resinous compositions of value in varnish manufacture are obtained by heating a natural resin with a substantially smaller proportion of a phenolic condensation product containing a reactive hydroxymethylene group at temperatures not above 150° until reaction is complete and a readily soluble, homogeneous product results. S. S. WOOLF.

Moulding compositions and (A) plastic materials or (B) artificial masses. K. RIPPER (B.P. 297,433 and 323,047, [A, B] 20.9.28. Austr., [A, B] 21.9.27).—Moulding compositions are obtained by reacting formaldehyde or a compound capable of yielding it, *e.g.*, trihydroxymethylene, simultaneously with a protein, preferably casein, and (A) urea, thiourea, or derivatives thereof, or (B) dicyanodiamide, with or without the addition of

a catalyst, the condensation products being subsequently subjected to the combined or separate action of heat and pressure.

S. S. WOOLF.

Production of flexible sheet material. [Glass substitute.] BRIT. CELANESE, LTD. (B.P. 307,462, 7.3.29. U.S., 8.3.28).—Netting, gauze, fabric, or other open-work or reticulated material is coated with a homogeneous mixture of a synthetic resin and cellulose acetate or other organic cellulose derivative. Alternatively, the foundation may be covered by a synthetic resin coating (with or without a content of cellulose derivative) insufficient to close the reticulations, this being completed by a further coating containing a cellulose derivative (with or without a content of synthetic resin). The synthetic resins, use of which is claimed, are of the phenol-aldehyde, ketone-phenol-aldehyde, phenol-furfuraldehyde, aromatic amine-furfuraldehyde, or ketone-furfuraldehyde types.

S. S. WOOLF.

Oxide pigments (B.P. 311,225).—See VII. **Chrome-yellow** (F.P. 554,102).—See X. **Insulation materials etc.** (F.P. 633,171).—See XI. **Rubber conversion products** (B.P. 323,012 and 323,028).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rational classification of the principal accelerators of vulcanisation [of rubber]. II. R. THIOLLET and G. MARTIN (Caoutchouc et Gutta-Percha, 1929, 26, 14,722—14,730; 1930, 27, 14,842—14,849. Cf. B., 1929, 755).—Using a mixture of rubber 100, zinc oxide 5, palm oil 1, and stearic acid 0.5, together with accelerator and sulphur, and vulcanising over a range of periods at various temperatures, the temperature which gives rise to the highest tensile strength is described as the "characteristic temperature" of the accelerator; the accelerators may then be classified according to their rapidity of action at this temperature. The use of anhydroformaldehydeaniline with zinc phenylmethyldithiocarbamate retards "setting" of the rubber mixture, but does not otherwise affect the progress of vulcanisation or delay arrival at the maximum mechanical properties. Joint use of mercaptobenzthiazole and ethyldeneaniline or diphenylguanidine gives the effect of a rapid accelerator producing a marked "plateau effect"; with tetramethylthiuram disulphide the plateau effect is small. Oven-ageing tests at 70° demonstrate that ethyldeneaniline, anhydroformaldehydeaniline, and anhydroformaldehyde-*p*-toluidine exert marked protective action; of the ultra-accelerators, zinc phenylmethyldithiocarbamate is also noteworthy for this feature. The arylguanidines do not impart good ageing qualities; but joint use of diphenylguanidine or ethyldeneaniline and mercaptobenzthiazole or of anhydroformaldehydeaniline and zinc phenylmethyldithiocarbamate promotes good ageing.

D. F. TWISS.

Resins. STOCK.—See XIII.

PATENTS.

Manufacture of artificial rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 320,362, 30.3.28).—The complete process for the production of polymerised isoprene from acetylene obtained from natural methane or waste cracked gases rich in methane is described.

The stages are: acetylene, acetaldehyde, aldol, γ -butyleneglycol, methyl ethyl ketone, methyl β -hydroxyisopropyl ketone, β -methyl- γ -butyleneglycol, isoprene, polymerised isoprene.

C. HOLLINS.

Production of conversion products of rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 323,012 and Addn. B.P. 323,028, [A, B] 17.8.28).—(A) Hard artificial masses, such as moulded articles or lacquers, are obtained by heating non-distillable polymerisation products of a butadiene between 100° and 500°; pigments, organic solvents, plasticisers, natural india-rubber, and polymerisation products of other diolefines may be added before heating. By subjecting fine threads of a solution to the heating process an artificial silk is obtainable. (B) The heat treatment is effected in the presence of sulphur, or of substances yielding sulphur, in proportion lower than that necessary for the production of vulcanite. Insoluble, chemically resistant products are obtainable constituting, *e.g.*, useful coatings for protective and insulating purposes.

D. F. TWISS.

Manufacture of rubber articles. SOC. ITAL. PIRELLI (B.P. 292,964, 27.6.28. Italy, 27.6.27. Cf. B.P. 284,608; B., 1929, 612).—In the manufacture of rubber articles from latex by localised heating, with consequent localised thickening and coagulation, the necessary condition in the latex is induced by the addition of an ammonium salt of a strong acid, *e.g.*, ammonium sulphate, and a sparingly soluble oxide of a metal of fixed bivalency of group II, *e.g.*, of magnesium, calcium, or zinc.

D. F. TWISS.

Vulcanisation of finished sheets or articles of rubber. PHIL. PENIN GUMMI-WAAREN-FABR. A.-G., Assces. of E. WLCECK (G.P. 453,900, 23.6.26. Addn. to G.P. 452,466; B., 1929, 257).—In place of the treatment with aqueous hydrogen sulphide, as in the prior patent, aqueous solutions of sulphides, hydrosulphides, or polysulphides may be used.

E. HOLMES.

Improving the strength of articles prepared from rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 323,322, 19.11.28).—The strength of vulcanised natural or synthetic rubber is increased by the incorporation of resinous esters of one or more mono- or poly-basic carboxylic acids, *e.g.*, phthalic acid, abietic acid, with polyhydric alcohols or ethers thereof containing at least one free hydroxyl group, *e.g.*, glycerol, ethylene glycol monoethyl ether.

D. F. TWISS.

Vulcanisation of rubber and accelerator therefor. D. H. POWERS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,732,532, 22.10.29. Appl., 3.6.25).—The accelerator comprises the product obtained by condensing 2 mols. of an aldehyde with 2 mols. of a primary amine and 1, 2, or 3 mols. of carbon disulphide.

A. R. POWELL.

Recovery of rubber and textile material from rubber tyres. J. THIBERVILLE (F.P. 620,346, 23.12.25).—The scrap is treated with chlorinated products of ethane or ethylene in a container provided with an agitator. The solution is removed through an opening in the base, which retains the cotton. The rubber obtained from the solution by distillation of the solvent

is then sheeted. The cotton is washed with light petroleum to remove residual traces of rubber.

D. F. TWISS.

Coating of metal surfaces with rubber. HARZER ACHSENWERKE GES.M.B.H. BORNUM AM HARZ, ASSEES. OF F. AHRENS (B.P. 302,935, 21.12.28. Ger., 21.12.27).—The cleaned metal surface (preferably of iron), after being etched with a strong acid, washed, and dried with hot air or steam, is treated with a solution of rubber, e.g., in amyl acetate to which a swelling agent such as carbon tetrachloride may be added, and again dried; a sheet of rubber, which may contain vulcanising agents, and may have been sheeted hot on to paraffined material, is then applied with pressure and vulcanised.

D. F. TWISS.

Treatment of rubber latex. J. MCGAVACK, ASSR. to GEN. RUBBER CO. (U.S.P. 1,740,994, 24.12.29. Appl., 6.8.27).—See B.P. 294,002; B., 1928, 721.

[Sealing the ends of ebonite]-coated metal rods or tubes. NEW ECCLES RUBBER WORKS, LTD., and C. HEMM (B.P. 323,399, 7.2.29).

Dielectric material (B.P. 323,038).—See XI. Toilet material (B.P. 323,321).—See XX.

XV.—LEATHER; GLUE.

Bark of *Hopea parviflora* as a tanning material. K. S. CHOUDARY and E. YOGANANDAM (Dept. Ind., Madras, 1929, Bull. No. 27, 14 pp.).—Analyses of samples of the bark of the iron-wood tree (*Hopea parviflora*) growing in Puttur Range, India, showed (%): tans 24.1—25.8, soluble non-tans 6.0—7.2, insoluble matter 59.6—60.4, moisture 8.3—8.6%, degrees red 4.6—5.1, degrees yellow 9.6—9.9. This tannin gave the usual reactions of pyrocatechol tannins, and hard ethyl acetate number 38.2, alcohol number 4.8, and gallic acid value 0.16. Practically no variation in the tannin content of the bark from different parts of the trunk or branches was found. The liquors prepared from it showed a low acid content (p_H 4.7) and bad diffusibility, but had a high ratio of tans to non-tans. A loss of 11.8% in tannin was observed in *Hopea* liquors after being kept for 34 days. A brittle solid extract containing (%) tans 65.9, soluble non-tans 21.6, insoluble matter 2.6, moisture 9.9, acidity 0.7, and showing tintometer readings of 3.9 degrees (red) and 8.7 (yellow) was obtained. Of the maximum extractable tannin in *Hopea* bark 75% was extracted at 15—30° and the maximum at 90—100°. Leather tanned with *Hopea* bark alone gave an unsatisfactory product, since the tannin is too astringent; addition of myrobalans to the bark gave products slightly browner than Madras tanned leather, but less brown than Cawnpore tannages. It dyes evenly, carries grease well, bleaches satisfactorily by the usual processes for lining sides, and, on exposure to sunlight, is darkened less rapidly than leather tanned with avaram or wattle barks, becoming a dull brown, whereas the latter turn reddish-brown. A good leather has been produced by tanning light kips with *Hopea* bark (8 pts.) and myrobalans (3 pts.), and the yield was above normal.

D. WOODROFFE.

South Indian myrobalans. K. S. CHOUDARY and E. Y. NAYUDU (Dept. Ind., Madras, 1929, Bull. No. 28,

40 pp.).—Dried myrobalans fruits (*Terminalia chebula*) as used in mixed tannages in Britain improve the colour of the leather, impart mellowness, deposit bloom on its fibres, and yield acid liquors. A small amount is used in the dyeing industry. About 5000 tons are annually produced in the Madras Presidency, much of which is used by the native tanners, not as a tanning material, but in the later stages of bark tannages as a fixer and bleaching agent. The best variety for this purpose is Salem myrobalans. Analysis of samples from various districts for tans and non-tans, calculated on the dry material and for degrees of red and yellow (Lovibond), shows respectively: Ganjam 25.5, 21.6, 0.5, 2.9; Kurnool class IA 20.2, 17.8, 0.7, 4.3; Kurnool class IIB 24.8, 16.3, 0.5, 3.2; Kurnool class III 20.7, 16.3, 1.1, 4.5; Vizagapatam 30.7, 17.5, 0.7, 2.6; Coimbatore 48.7, 15.7, 0.1, 2.2; Godavari 32.2, 16.7, 0.4, 2.2; Madura 51.2, 11.1, 0.3, 2.0; Cuddapah 41.9, 17.0, 3.7, 6.6; Salem 34.4, 16.8, 0.4, 3.6; Vellore 30.0, 21.0, 0.8, 4.4; Kurnool 33.5, 14.0, 0.3, 2.5. Nuts weighing on the average > 5 g. contained > 40% tannin (on dry basis). The ratio of flesh to stone varied from 1.3:1 in Kurnool nuts to 6.0:1 in a sample from Coimbatore. The ratio of flesh to stone was > 2:1 in rich samples of myrobalans. Tans: non-tans varied from 4.6:1 to 1.1:1, and the sugar content from 2 to 8%. The proportion of red to yellow varied from 1:9 for Salem nuts to 1:1.8 for Cuddapah nuts. The lightest coloured products were obtained from Coimbatore and Madura, and were brighter coloured than the best commercial products exported to foreign countries. The greatest amount of acid was produced by the fermentation of the product from Madura, and most of the South Indian myrobalans yielded more acid than the exported products. Most "bloom" was deposited by the Coimbatore product (13 lb.) and least by the Madura (6.2 lb. per 100 lb. of nuts). A loss in tannin of 25—40% was observed when liquors prepared from the different varieties were kept for 10 days. Coimbatore, Madura, and Salem myrobalans are the best products.

D. WOODROFFE.

Lactic acid bacteria from tan liquors. B. SCHWARZBERG and P. GINDIS (Zentr. Bakt. Par., 1929, II, 78, 96—105; Chem. Zentr., 1929, ii, 760).—The bacteria are facultative anaerobes; the optimal temperature is 30—35°. The acid content runs parallel with the peptone content; dextrose, levulose, galactose, maltose, sucrose, xylose, mannitol, dextrin, and starch, but not rhamnose nor arabinose, were fermented. In barley extract with dextrose, peptone, fibrin, asparagine, leucine, alanine, tyrosine, or glycine served as a source of nitrogen.

A. A. ELDRIDGE.

Sensitive method for the determination of the m.p. of gelatin jellies. W. MATTHAES (Chem.-Ztg., 1929, 53, 910).—The air-dried gelatin is dissolved in water at 65° to a 10% solution and 10 c.c. are placed in a metal tube 16 cm. long and 17 mm. in diam. and allowed to set and age for 24 hrs. in a thermostat at 25°. The tube is closed with a cork carrying a loosely fitting, thin metal rod terminating at the lower end in an upright cone and at the upper end in a thin plate, the whole weighing 10 g. The base of the cone rests on the jelly, and when the latter melts to such an extent that the

cone sinks 3 cm. below the surface the plate at the upper end of the rod makes electrical contact with a similar plate on the top of the cork and rings a bell; the temperature at this point is taken as the m.p. and can be reproduced to within 0.1° if the tube is heated in a water-bath at the rate of about 6° in 55 min. by means of a carbon-filament lamp. A. R. POWELL.

Tannin in brewing. FINK and WILDNER.—See XVIII.

PATENTS.

Tanning [of hides]. J. G. KÄSTNER and C. H. TRÖGER (B.P. 294,996—7, 3.8.28. U.S., [B] 3.8.27).—Not more than 35% of a colloidal decoction of the kernels or seeds of carob or like beans is mixed with a tan liquor in which the hides are immersed. Penetration of the liquor into the hides is assisted by using an electric current (at 20–25 volts, with electrodes of lead-antimony alloy) and/or alternations of pressure and vacuum (up to 300 times per min. with a pressure of 1 atm. or more). Use may also be made in the tan liquor of fermentation bates, yeasts, bacterial cultures, gases, or radiation, or the hides may be pretreated with these. D. WOODROFFE.

Tanning of animal hides. REISCHACH & Co. G.M.B.H. (B.P. 306,400, 16.2.29. Ger., 18.2.28).—Hides are tanned in a solution of a ferrous salt which has been oxidised with nitrous oxide practically free from higher oxides, or the hides are immersed in a solution of a ferrous salt, *e.g.*, ferrous sulphate, and the latter is subsequently oxidised with nitrous oxide under pressure. D. WOODROFFE.

Preparation of artificial horn from alcohol-insoluble albumin and nitrocellulose. F. SCHMIDT (G.P. 457,235, 27.9.23).—The materials are mixed with a suitable neutral, volatile solvent, *e.g.*, ethyl formate, ethylene chlorhydrin, which has a b.p. below 150° , a vapour tension above 50 mm./ 17° , and which causes the albumin to swell and the nitrocellulose to dissolve (or swell); the solvent may be diluted with alcohol and/or water. E. LEWKOWITSCH.

Varnishing of [patent] leather and like material. J. PAISSEAU (U.S.P. 1,742,146, 31.12.29. Appl., 19.10.25. Fr., 23.7.25).—See B.P. 255,803; B., 1927, 21.

XVI.—AGRICULTURE.

Brown earths. A. STEBUTT (Z. Pflanz. Düng., 1929, 15A, 134–167).—The course of formation of brown-earth soils is examined and discussed. A chemical examination of soil profiles is recorded and their morphological characteristics are given. A. G. POLLARD.

Nutrient ratios and reaction condition of weathered soils of the crystalline Odenwald. L. SCHMITT (Z. Pflanz. Düng., 1929, 15A, 117–134).—The granite soils of the Odenwald are examined chemically, by the Neubauer method, and by field trials for their nutrient contents. In general, these soils are acidic in character with a high content of easily assimilable potash and a moderate content of phosphate in a difficultly soluble form. Increasing acidity of these soils is associated with lower proportions of available phosphate. A. G. POLLARD.

Nutrient content [of soils] and nutrient requirement [of plants]. R. MEYER (Z. Pflanz. Düng., 1929, 15A, 101–116).—Present conceptions of growth curves, nutrient contents, and nutrient requirements are critically examined from a practical point of view. "Nutrient content" relates to soil only; "nutrient requirement" is a characteristic of the plant alone, its determination being dependent on plant physiology and intimately connected with the growth law. A knowledge of the nutrient content of a soil is unnecessary in establishing the nutrient requirement of the plant. A consideration of the yield law is necessary and sufficient for determining nutrient requirements. The Mitscherlich method, apart from the theory involved, is the most suitable basis for ascertaining plant nutrient requirements.

A. G. POLLARD.

Use of citric acid in the examination of soils and fertilisers. O. LEMMERMANN (Z. Pflanz. Düng., 1929, 15A, 170–171).—Use of citric acid by Dyer as an extractive for phosphates in soils and fertilisers was anticipated by Gerlach (Landw. Versuchs-Stat., 1896, 46, 201).

A. G. POLLARD.

Rapid colorimetric method for determining the phosphate deficiency in arable soils. A. NĚMEC (Z. Pflanz. Düng., 1929, 15A, 167–170).—Comparison is made of crop yields in phosphate fertiliser trials, and the phosphate content of the soils as determined by various methods. Definite crop increases resulted from the use of phosphate fertilisers when the phosphate content of the soil by the Dirks and Scheffer method (B., 1929, 67) corresponded to less than 1 mg. P_2O_5 per kg. of dry soil. Silica values by NĚmec's method (cf. A., 1928, 1162; B., 1928, 938) agree closely with the results of field trials if a limiting value of 12 is accepted. Lemmermann's citrate method gave results agreeing fairly closely with the colorimetric method, but less so with the field trials. A. G. POLLARD.

Mitscherlich's pot experiment method for the determination of potassium requirement [of soils]. LAMBERG (Ernähr. Pflanze, 1929, 25, 169–173; Chem. Zentr., 1929, ii, 475).—Field and pot experiments with barley, wheat, rye, potatoes, and beet are compared. The increased yields predicted by Mitscherlich's method were, with few exceptions, obtained. A. A. ELDRIDGE.

Colorimetric determination of hydrogen-ion concentration of soils by Kolthoff's dialysis method. W. A. J. OOSTING (Chem. Weekblad, 1929, 26, 618–619).—Results are given of a large number of determinations by the electrometric method and by the colorimetric method after dialysis, which show that the latter gives reliable and accurate values. S. I. LEVY.

Significance of determination of hydrogen-ion concentration in estimating the lime condition of the soils between the Danube and the Theiss. A. RÁTH (Mezőgaz.-Kutat., 1928, 1, 121–126; Chem. Zentr., 1929, ii, 629).—The hydrogen-ion concentration is not always a trustworthy indication of lime condition or requirement. A. A. ELDRIDGE.

Rapid determination of calcium in clay soils with Scheibler's apparatus. I. KÜHN (Mezőgaz.-Kut.,

1928, 1, 118—120; Chem. Zentr., 1929, ii, 476).—The reaction is completed in 5 min. if an equal weight of potassium hydrogen fluoride is added to the soil before shaking with 10% hydrochloric acid (25 c.c.).

A. A. ELDRIDGE.

Litter materials, particularly forest litter. F. MACH and R. HERRMANN (Landw. Versuchs-Stat., 1929, 109, 267—316).—The water- and nitrogen-absorbing capacities of numerous types of litter materials, together with their contents of plant nutrients, are recorded. A general scheme of evaluating these materials is given. The relative amounts of water and nitrogen absorbed by litters are not proportional. Peat was the most absorbent material examined. With advancing humification of forest litter, the absorbing capacity and nitrogen content increase, the calcium and magnesium contents decrease, whilst the potash and phosphate contents do not alter appreciably. Ammonia absorbed by litter is firmly held, a portion being chemically combined and incapable of liberation by treatment with sodium hydroxide.

A. G. POLLARD.

Action of various nitrogenous fertilisers on acid soils. D. MEYER and P. OBST (Z. Pflanz. Düng., 1930, 9B, 18—31).—Pot experiments with cereal crops on a number of acid soils are described. With oats, barley, and mustard, sodium and calcium nitrates produced similar effects except on a very acid soil, where the former caused a decrease and the latter had no effect on a mustard crop. Ammonium nitrate was less efficient than sodium or calcium nitrate on all these crops, although the difference was less marked in the case of oats. Ammonium sulphate produced persistently negative results with mustard and barley, but positive effects with oats. These results are not wholly explicable on a basis of differences in acidity or degree of saturation of the soil with bases. Urea and ammonium nitrate were about equally effective on oats and barley. Urea proved superior with mustard, results with which approached those of sodium and calcium nitrates. Calcium cyanamide was generally equal to, and in some cases better than, sodium and calcium nitrates. The beneficial effects of urea and cyanamide may be due to a reduction of soil acidity by the ammonia derived from them. Oats utilised the greatest proportion of the nitrogen added in these trials. Of the fertilisers used, the most highly utilised were sodium and calcium nitrate, followed in descending order by cyanamide, ammonium nitrate, ammonium sulphate. The intakes by plants of nitrogen and calcium were not proportional.

A. G. POLLARD.

Examination by Neubauer's seedling method of the phosphate- and potash-intake of different plants from air-dried and heated soils of varying acidity. K. SCHUMANN (Z. Pflanz. Düng., 1929, 15A, 65—94).—Preheating of soils to 80—100° caused a larger intake of potash and phosphate by various plants than when air-dried soil was used in Neubauer experiments. Soil reaction was not affected by heating to this temperature. The assimilation of nutrients by plants was markedly affected by differences in soil reaction, the effect varying considerably with different

plants. Plants exhibiting considerable tolerance to alkaline conditions were generally very sensitive to acidity and *vice versa*. Changes in assimilation with acidity made their appearance more or less gradually. In alkaline soils marked effects on assimilation were associated with p_H values of about 8.3 in the hot potassium chloride extract of the soil. Comparative field trials and Neubauer experiments are recorded. Results do not correspond with the Mitscherlich theory of growth factors. Rye proved the most suitable crop for Neubauer experiments.

A. G. POLLARD.

Practical significance of Neubauer [soil] analyses; results from the experimental area of Krummhörn. HEMPELMANN (Z. Pflanz. Düng., 1930, 9B, 81—88).—With the marshland soils examined Neubauer's method yielded very uncertain results.

A. G. POLLARD.

[Action of] zeotokol and phosphoric acid [on plant growth]. H. C. MÜLLER (Landw. Versuchs-Stat., 1929, 109, 343—346).—The recorded inefficiency of zeotokol (cf. Reuter, G.P. 365,396; B., 1923, 1087A; also Blanck and Alten, B., 1925, 80) is the outcome of its property of reducing the solubility of phosphates already present in soil, possibly owing to the formation of iron and aluminium phosphates.

A. G. POLLARD.

Determination of assimilable nutrients in soils. GERLACH (Z. Pflanz. Düng., 1930, 9B, 68—81).—Various methods for determining the nutrient contents of soils are examined from the point of view of their possible errors, limiting values, and relation to field trials (cf. Römer and Scheffer, *ibid.*, 1928, 7B, 579; B., 1929, 991). There is poor correlation between Neubauer experiments and field trials. In Neubauer's process all root-soluble material is not taken up by the experimental crop, but subsequent crops continue to extract considerable amounts of nutrients. The limiting values in Neubauer trials depend on the power of assimilation or solubilisation of the individual plant species, the depth of soil, the anticipated crop yield, and on climatic conditions. Although both Neubauer's and Mitscherlich's methods give more rapid results of fertiliser requirements, neither is more reliable than are field trials.

A. G. POLLARD.

[Law of] minimum [in agriculture]. O. NOLTE (Landw. Versuchs-Stat., 1929, 109, 317—342).—A critical discussion of Liebig's "law of minimum" and its modern applications, including the theory of the constancy of growth-factors (Mitscherlich).

A. G. POLLARD.

Influence of nitrogenous manures on the protein content of malting barley. A. STEVEN (Z. Pflanz. Düng., 1930, 9B, 35—43).—Moderate applications of nitrogenous fertilisers (40 kg. N/hectare) increased barley yields without decreasing the protein content. The use of nitrogen fertilisers for barley are important on light, freely permeable soils and, generally, where barley follows a nitrogen-exhausting crop. The position of barley in crop rotations and the effect of climatic conditions on the crop are discussed.

A. G. POLLARD.

Deficiency of magnesium the cause of a chlorosis in corn [maize]. J. P. JONES (J. Agric. Res., 1929,

39, 873—892).—Maize grown in rotation with hay since 1897 in an experimental field at the Massachusetts Agricultural Station has recently shown a marked chlorosis characterised by a green and yellow-to-brown striping of the leaves. Field and greenhouse studies now show this to be due to magnesium deficiency, caused by continued use of magnesium-free fertilisers on a light soil subject to considerable leaching. The chlorosis is worst in seasons of heavy rainfall. An application of 20—40 lb. per acre of magnesium as the sulphate or as magnesium limestone is sufficient to control the chlorosis on such soils. E. HOLMES.

Manuring of fruit trees with lime. J. N. KONOVALOV (Z. Pflanz. Düng., 1930, 9B, 32—35).—Liming of apple trees produced yields of fruit averaging double those of control trees. Proportional increases were not the same for all varieties. Cherry crops were also increased by liming, but the results were less regular.

A. G. POLLARD.

Influence of the addition of a mineral salt mixture to feeding-stuffs on the quantity and total constituents of milk. W. LIEBSCHER (Landw. Versuchs-Stat., 1929, 109, 347—362).—Results are recorded of milking trials of cows receiving a supplementary mineral salt ration, the active constituents of which were calcium phosphate and potassium iodide. The minerals did not influence the live weight of the animals, the total milk yield, nor the total dry matter and fat produced. The amount and density of the fat-free dry matter increased slightly.

A. G. POLLARD.

Hot fermentation of stall manure. P. EHRENBURG (Z. Pflanz. Düng., 1930, 9B, 49—67).—Recorded experimental work concerning the hot-fermentation process is critically discussed. The superiority claimed for this process is not yet adequately supported by sound investigation.

A. G. POLLARD.

Hay-making on Swedish dryers. W. ZIELSTORFF and K. NEHRING (Landw. Versuchs-Stat., 1929, 109, 253—266).—Comparison is made by chemical analysis and feeding trials of hay made by ordinary sun-drying and by dryers. Use of dryers increased the yield of digestible foodstuffs by 40—50%. If stacked on the dryer immediately after mowing, the hay loses notable amounts in dry weight; if stacked, however, from 12—24 hrs. after mowing, good results are obtained. Losses by "sweating" amount to 4—6% and fall mainly on the crude fibre and nitrogen-free extractives. There are no losses of nitrogen.

A. G. POLLARD.

Nitrates, soil reaction, and yield of tobacco after using different fertilisers; field experiments. A. V. OTRIGANIEV (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 53, 29 pp.).—The nitrate content, soil p_H , and relative size of tobacco crop have been determined before and after the application of the following fertilisers to various soils: sodium nitrate, ammonium sulphate, calcium cyanamide, Chili nitrate, lime, potassium sulphate, and superphosphate.

E. A. LUNT.

Effects of soil temperature and reaction on growth of tobacco infected and uninfected with

black root rot. W. L. DORAN (J. Agric. Res., 1929, 39, 853—872).—Black root rot caused by *Thielavia basicola* had little if any injurious effect on tobacco plants grown in a soil of p_H 5.6 or lower at any temperature. At 15—27° injury occurred above p_H 5.7—5.9, but at 30° there was little injury even at p_H 6.0—6.9. Strains of Havana tobacco susceptible to black root rot suffered greatly on heavily limed plots, whilst resistant strains grew normally. In the absence of *Thielavia basicola*, i.e., after soil sterilisation with formaldehyde, lime increased yields of tobacco at the higher soil temperatures, 27—30°, but retarded growth below 24°. Lime had harmful effects when applied to a tobacco seed bed at 24°, the effect being relatively much greater in sterilised soil. E. HOLMES.

Curing of yellow tobacco. I. Physiology of cigarette tobacco leaf starvation. A. I. SMIRNOV and M. A. DRBOGLAV (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 54, 36 pp.).—The metabolism of the tobacco plant during water deprivation has been studied with reference to respiration, transpiration, and changes in the carbohydrate, nitrogen, and enzyme contents of the leaves.

E. A. LUNT.

Sugar-beet culture and purity of juice. BARTELS.—See XVII.

PATENTS.

Fertiliser. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY ET CIREY (F.P. 630,226, 18.5.26).—Phosphorus pentoxide vapours from the burning of phosphorus are treated with gaseous ammonia, if necessary in excess. The product is hydrolysed to ammonium phosphate in the soil.

S. K. TWEEDY.

Plant sprays. I. KREIDL (G.P. 454,569, 12.4.22. Austr., 6.4.22).—A number of minerals and industrial waste products, including ores or compounds of copper, arsenic, sulphur, etc. may, when ground to colloidal dimensions or when in colloidal solution, be used as plant sprays. Satisfactory colloidal solutions may contain 0.08—0.20% As, 0.1—0.3% CuS, or 0.08—0.20% HgS.

E. HOLMES.

Preparation of insecticides and fungicides. HART & CO. PROPRIETARY, LTD., Assees. of F. L. C. HART (Austral.P. 5201, 14.12.26).—Arsenious oxide is dissolved in glycerin at or above 80° and with the solution are mixed emulsifying agents (alcohol, ether, soap solution) and coal-tar distillates (benzene, creosote). The solution is diluted to various extents for different purposes.

S. K. TWEEDY.

Vermin-killers, especially for rats and mice. I. KREIDL (Austr.P. 108,157, 4.11.25).—Satisfactory vermin-killers consisting of poison baits containing bread-crumbs, meat, or grain with salts of thorium, zirconium, or the rare earths, are claimed. An example contains 10% each of sugar and ceric chloride, with bread-crumbs.

E. HOLMES.

Neutral phosphate (F.P. 632,310). Iron arsenate (Austral. P. 3833).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Influence of cultural conditions on the purity of juice and content of objectionable nitrogen in the sugar beet. H. BARTELS (Z. Ver. deut. Zucker-Ind., 1929, 79, 553—590).—A field in Eastern Germany, with a soil of mild, humous loess loam above sand, was divided into plots on which beets were grown under varied conditions, as regards spacing, time of sowing, and nature and amount of fertilisers. It is concluded that conditions of growth should above all be chosen with a view to ensuring that the roots obtained shall be fully matured. In the present case the period of growth was 160—166 days, and the most favourable spacing was found to be about 160 sq. in. per plant (rows 20 in. apart; plants not more than 10 in. apart). Wider spacing lowers the sugar content of the roots and the purity of the juice, as it delays ripening; it is, therefore, only to be recommended where a longer growing period is possible. Only in case of very long growing periods should the space per plant exceed 240 sq. in. Early sowing is always to be recommended. Of the nitrogenous fertilisers tested, urea was found to affect the amount and quality of the crop unfavourably. Ammonium sulphate is satisfactory in all respects with good, humous soil rich in lime. Artificial and natural saltpetre were found equally effective, and most beneficial in amounts up to 6.5 cwt./acre. Excessive and unbalanced amounts of nitrogen in any form impair the quality of beets. Potash fertilisers have a favourable effect on sugar formation, even in soils naturally well supplied with potash. Application of phosphates, however, to a soil not lacking in these does not appear to have any notable effect. J. H. LANE.

Determination of marc in beets by alcoholic extraction after limited aqueous digestion. E. THIELHAPE and P. MEIER (Z. Ver. deut. Zucker-Ind., 1929, 79, 539—552).—The pectin substances of beets, insoluble in the cold, become hydrated and more or less soluble on digestion with hot water. Ehrlich and Sommerfeld (A., 1926, 441) obtained 6.4% of air-dry or 5.7% of water-free marc by extraction of pulped beets with water at 20°, followed by washing with alcohol and ether, but they found that about half the dry matter in this marc became soluble on prolonged digestion in boiling water followed by heating under pressure at 121—135°. This solubilisation of beet pectin by water does not begin below 55°. Aqueous extraction of beets with water at 55° followed by washing with alcohol would leave a marc consisting substantially of cellulose (39%) and pectin (61%) and the method now described aims at determining marc as thus defined. The beets are sampled and chopped into thin slices, 1—2 mm. thick, and 100 g. are introduced into 1 litre of water at 80° and digested for 2—3 min. The liquid is then filtered through a tared filter-thimble into which the solid is afterwards rinsed, and the thimble is transferred to a modified Soxhlet apparatus in which extraction with 80% alcohol is continued for 1—2 hrs. The marc is finally washed with ether and dried to constant weight at 105—110°. The average marc-content of a number of German beets of the 1927—8 and 1928—9 campaigns

was 4.75%, individual values ranging from 3.10 to 5.81%. J. H. LANE.

Hot liming in defecation [of sugar juice]. J. P. F. HEUSE (Archief, Verslagen, 1929, Afl. 5, 216—222).—Juice, which when clarified by liming cold in the ordinary way settled badly and gave rise to the formation of "fatty" scums, was satisfactorily treated by heating to its b.p. and liming continuously. Neither the filter-press loss nor the unknown loss was higher in this preheating process; further, the exhaustion of the massecuites became better than before. Applied at two other factories in Java, however, it failed to show any improvement. J. P. OGILVIE.

Separate clarification of last-mill [sugar] juice. P. HONIG (Verslag van der Vereeniging het Proefstat., 1928, 211—212).—Last-mill juice heated to 75° was sulphited to p_H 7.8, boiled, filtered, limed to p_H 7.4, and heated to 95°. Under these conditions filtration was easy and cakes of low sucrose content were obtained, besides which there was an economy both of sulphur and lime, compared with whole-juice treatment. The first-mill juice was clarified by sulphitation in the ordinary way. J. P. OGILVIE.

Determination of invert sugar in sugars etc. M. VAN DER KREEKE (Archief, deel III, Meded., 1929, No. 13, 781—787).—Using a 300-c.c. flask, a mixture of 25 c.c. each of Luff's reagent (cf. Schoorl, B., 1926, 507) and of a solution of the sample (previously clarified with normal lead acetate, using sodium phosphate to remove excess) is boiled under reflux, heating being so arranged that the contents of the flask are raised to b.p. in 3 min. and remain in ebullition for 5 min. After cooling, the liquid is mixed with 15 c.c. of 10% potassium iodide solution, then with 25 c.c. of 25% solution of sulphuric acid, and titrated with 0.1N-thiosulphate with starch as indicator. Luff's reagent is said to permit of a possible accuracy of 0.01%, to show no auto-reduction, to be reduced by sucrose to a much less extent than Fehling's solution, and to be less affected by small variations in the duration of boiling. J. P. OGILVIE.

Filtration of juices, syrups, and molasses in [beet] sugar manufacture. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1929, 46, 393—413).—Rotary filters of the Vallez type (B., 1922, 619 A; 1923, 961 A; 1927, 864) are in use in a number of beet-sugar factories, for the filtration of juices after carbonatation. They are economical in respect of labour and yield very clear juices and cakes of uniform thickness which can be efficiently sweetened-off. A French factory has six filters with a total surface of 300 m.², sufficing for the working of 1000 tons of beets daily. The complete cycle of operations for a filter, including filtration of juice, washing and removal of cakes, and preparing for the next filtration, occupies about 2 hrs. About 85 litres of hot water is used for sweetening-off 100 kg. of scums, the sugar content of which is reduced to 0.4—0.5%. A periodic cleaning of the filters with dilute alkali and acid is carried out without removing the cloths. The Vallez filter can be adapted to the filtration of syrups by replacing the cloths by gauze, mixing a portion of the

syrup with paper pulp (made as required from ordinary paper in a disintegrator), and depositing this pulp on the gauze to serve as filtering medium. These filters have also been used in a special process in which a massecuite centrifuged hot is made to yield a further crop of sugar by slow cooling to 25–28°, the sugar formed being then collected in a Vallez filter, from which it is afterwards washed out for remelting. J. H. LANE.

Affining of sugars and use of a decolorising earth in sugar factories. P. DEVOS (Bull. Assoc. Chim. Sucr., 1929, 46, 292–301).—A bauxite from the East, containing 40–50% Al_2O_3 , 15–20% SiO_2 , 4–5% TiO_2 , 2–3% Fe_2O_3 , and about 30% of moisture, is suggested as a filtering medium and preliminary decolorising agent for use in sugar factories. It would require roasting in a reverberatory furnace, after which it is easily friable and can be powdered in a ball mill. It has about 30% of the decolorising power of animal charcoal on low-grade sugar products and about 10% on high-grade syrups, and it adsorbs a considerable amount of calcium salts. It can be almost completely revived by heating at 400°. In a discussion following the paper the iron content of the material was pointed out as objectionable. J. H. LANE.

Sugar refining. J. GUILLAUME (Bull. Assoc. Chim. Sucr., 1929, 46, 341–351).—A short account of the main operations in sugar refining, with flow-sheets from some typical Continental refineries. J. H. LANE.

Modification of the determination of reducing sugars. J. DE VILMORIN and E. CAZAUBON (Bull. Assoc. Chim. Sucr., 1929, 46, 414–415).—The methods previously referred to (B., 1929, 489) are further discussed and modifications suggested. J. H. LANE.

Liberation of ammonia at different stages of [beet sugar] manufacture. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1929, 46, 386–392).—A lecture. J. H. LANE.

Mucilages. J. GÍRAL (Anal. Fís. Quím. [tecn.], 1929, 27, 319–340).—Mucilages are defined as naturally occurring substances which yield viscous, fermentable, optically active solutions which are coagulated by alcohol, salts, and acids, but not by heat; chemically they contain polymerised carbohydrate molecules with some molecules of acids derived from pentoses and hexoses, and occasionally methoxyl and acetyl groups. The physical and chemical behaviour of various vegetable and animal mucilages is described, and their modes of formation and technical applications are discussed. H. F. GILLBE.

Viscous fermentation of frozen beetroots. H. COLIN and M. SIMONET (Bull. Assoc. Chim. Sucr., 1929, 46, 505–509).—See B., 1929, 411.

Purification and decolorisation of cane and beet-sugar juices by the combined use of sulphur dioxide and activated carbons. G. MEZZADROLI and E. VARETON (Ind. Sacc. Ital., 1928, Nos. 9–11; Bull. Assoc. Chim. Sucr., 1929, 46, 356–357).—See B., 1929, 617, 695.

Decolorising carbons and their action on mo-

lasses. M. GARINO and G. BENVENUTO (Bull. Assoc. Chim. Sucr., 1929, 46, 326–331).—See B., 1927, 685.

PATENTS.

Extraction of sugar from molasses. W. HOMMEL (G.P. 455,099, 5.8.24).—The molasses (700 g.) containing 350 g. of sugar is diluted with 1500 g. of water and 112 g. of quick lime are added. When this has dissolved, 220 g. of calcium chloride or its equivalent of other soluble calcium salts and 100 g. of caustic soda solution are added. The mixture is warmed to 60–70°, and the precipitate collected on a filter, washed with hot water, and decomposed by carbon dioxide or other agent. The insoluble calcium salt is filtered off and the filtrate evaporated *in vacuo*, whereby 300–315 g. of sugar are obtained. W. J. BOYD.

Utilisation of waste waters from starch manufacture. SOC. INDUST. DE LANDRECIES, and C. MARILLER (F.P. 631,610, 19.6.26).—Residual starch is allowed to deposit from the waters used in the steeping, washing, and extraction processes in starch manufacture, and classified as of "second quality." The supernatant, sugar-containing liquid is fermented to yield ethyl or butyl alcohol, acetone, and butyric or lactic acid. C. RANKEN.

Refining of sugar. E. N. EHRHART (B.P. 320,982, 13.8.28).—See U.S.P. 1,731,237; B., 1930, 30.

Centrifugal machine (B.P. 322,942). Decolorising of solutions (B.P. 297,430).—See I. **Saccharification of wood etc. (B.P. 311,695).**—See V. **Agglomerated beet pulp (B.P. 320,883).**—See XIX. **Glutamic acid (U.S.P. 1,721,820).**—See XXIII.

XVIII.—FERMENTATION INDUSTRIES.

Proteolytic enzymes of green malt. R. H. HOPKINS and J. A. BURNS (J. Inst. Brew., 1930, 36, 9–15).—Green malt contains a protease and a peptidase. The protease degrades Witte's peptone and prevents the coagulation of crystalline egg-albumin by heat at p_H 4.7. The activity of the protease is completely destroyed by alkali and excessive acidity and greatly reduced by heating at 62° at its natural p_H . The p_H of optimum stability approximates to 4.4–5.0 at 40°. The peptidase attacks glycylglycine at slightly alkaline reaction, and is very sensitive to heat, even at p_H 9.0, in the presence of cold-water malt extract. C. RANKEN.

Influence of sediment on yeast and fermentation. C. SCHUSTER (Woch. Brau., 1930, 47, 1–2).—Laboratory experiments confirm Windisch's statement (B., 1929, 833) that the presence of sediment in the wort increases the vigour of the yeast. From brewery observations it is concluded that excessive amounts of relatively coarse sediment ("trub") gives rise to bottom yeasts which are loose and slimy but of normal fermentative power. Fine turbidity ("trübung") is without influence except that the "Kräusen" are weaker. Sediment of intermediate degrees of fineness acts unfavourably on both yeast and fermentation, and the effect is cumulative in successive fermentations. F. E. DAY.

Solubility of maize proteins in mashing. R. H. HOPKINS and J. A. BURNS (*J. Inst. Brew.*, 1930, 36, 16—21).—Flaked maize, when mashed with malt, yields a wort which contains an amount of permanently soluble nitrogen greater than can be accounted for by the malt components of the grist alone. The increased content of soluble nitrogen is probably due to the slower destruction of the malt protease at mashing temperature in the presence of the higher concentration of protein substrates.

C. RANKEN.

Tannin [in brewing]. I. Precipitation of the proteins of wort and beer by tannin. H. FINK and H. WILDNER (*Woch. Brau.*, 1930, 47, 11—16, 23—26).—The addition of tannin to wort in increasing amounts from 0.01—0.1% at ordinary temperature causes increasing precipitation of proteins. The formol nitrogen is little affected, about 33% of the albumoses are precipitated by 0.05%, and little more by 0.1% of tannin; the precipitation of peptones and protein separating on boiling the slightly acidified wort increases throughout to 40% and 87%, respectively, at 0.1% of tannin. When 0.01% of tannin is added during the boiling of the wort the proportions of coagulating protein and formol nitrogen precipitated are the same as in the cold, but increased amounts of peptone and albumose are precipitated. From beers at ordinary temperature coagulable and albumose nitrogen are precipitated, but peptone and formol nitrogen are hardly affected.

F. E. DAY.

[Detection of] wines manufactured from dried grapes. R. MOREDOD (*Ann. Falsif.*, 1929, 22, 524—542).—Ordinary methods of analysis have failed to detect wines manufactured from dried grapes, and certain additional tests are described which facilitate their differentiation from natural wines. Taste is of first importance, but is incapable of exact definition. Wines from dried grapes give a blue fluorescence in ultra-violet light, whilst normal wines give none, or only a slight green appearance. The resorcinol test for invert sugar gives positive results, since the dried-grape wines contain 2—6 g./litre of reducing substances. In this test, which consists in treating the wine, evaporated to a syrupy consistency, with resorcinol dissolved in hydrochloric acid (cf. Fiehe, B., 1928, 834), natural wines give yellow-orange tints, with occasional red colours which do not persist, whilst the "dried-grape" wines give a persistent cherry-red coloration. The acetone test gives similar results and consists in mixing the evaporated wine with 6 c.c. of acetone, filtering, and adding 0.5 c.c. of hydrochloric acid (*d* 1.19). Only red colorations persisting for 24 hrs. should be regarded as positive results, yellow and orange colours being ignored. This test, however, is not conclusive since some white wines from black grapes give positive results if the decolorisation by animal black has not been complete. Natural wines contain only a small proportion of reducing sugars, mainly dextrose and laevulose, but there are also dextrans and gums which give positive results with Fehling's solution after inversion by heating on a brine bath for 1 hr. The difference between the values, before and after inversion, should rarely exceed 0.5 g./litre

for natural wines, but dried-grape wines always give high values. Wines from dried grapes give a persistent deep blue colour with Gautier's reagent, which is prepared by adding a solution of 15 g. of ammonium molybdate in 70 c.c. of water to 130 c.c. of nitric acid and decanting the solution after keeping for 24 hrs. at 35°. 2 c.c. of the reagent are added to 17 c.c. of wine previously decolorised by shaking with animal charcoal and, if necessary, a trace of albumin. The mixture is held in boiling water for 10 min. and is then poured into boiling water. Natural wines produce a greenish colour, and wines containing unfermented sugar give a blue colour, which disappears at the end of several days, but "dried-grape" wines always produce a persistent deep blue coloration.

H. J. DOWDEN.

Significance of benzoic acid and sodium benzoate in the preparation of non-alcoholic fruit wines. A. OSTERWALDER (*Landw. Jahrb. Schweiz*, 1929, 43, 97—119; *Chem. Zentr.*, 1929, ii, 806).—The bacteria are less sensitive than the yeasts; acetic bacteria are particularly resistant. Benzoic acid and its salts are not recommended for the purpose.

A. A. ELDRIDGE.

Rapid determination of iron in white wines. J. RIBÉREAU-GAYON (*Ann. Falsif.*, 1929, 22, 522—524).—The method is based on the blue colour produced in wine by addition of potassium ferrocyanide and hydrogen peroxide. The standard of comparison consists of six test-tubes each containing 10 c.c. of ferric chloride solution of the following concentrations: 3, 6, 9, 12, 15, and 18 mg./litre, respectively. To each of the first three tubes is added 1 drop, and to the remaining tubes 2 drops, of freshly-prepared potassium ferrocyanide solution (10 g./litre), followed by 3 drops of 12-vol. hydrogen peroxide. The sample of wine (10 c.c.) is treated with 3 drops of the ferrocyanide solution and 3 of hydrogen peroxide, and the colour is matched against the scale. When a greenish tint renders comparison difficult, a test-tube of wine is placed behind the standard and another containing water behind the sample. If the wine colour exceeds the 18-mg. standard, the sample is diluted with 10 c.c. of water, and if this dilution is insufficient, *i.e.*, more than 30 mg./litre in the wine, a fresh 10-c.c. sample is diluted with 20 c.c. of water, 5 drops each of ferrocyanide and of peroxide being then added.

H. J. DOWDEN.

Proteins of barleys of Austria and the neighbouring states. A. SZILVINYI (*Woch. Brau.*, 1930, 47, 3).

PATENTS.

Manufacture of sparkling wine. P. M. RIVOLIER and P. O. I. NAVARRE (F.P. 630,441, 27.5.26).—Natural, sweet grape juice is concentrated and added to the wine in place of the customary sugar. C. RANKEN.

Checking of fermentation in the manufacture of sparkling wines. H. BOULARD (Addn. F.P. 32,419, 13.7.26, to F.P. 615,014; B., 1927, 793).—Fermenting wine is raised to a temperature above that required to kill the fermentation organism, but below the usual sterilisation temperature, so that the zymase remains

active. On being bottled, such wines undergo a second fermentation with the desired development of carbon dioxide.

C. RANKEN.

Sulphurisation of wine etc. in bottle. SEITZ-WERKE GES.M.B.H. (G.P. 456,850, 27.5.24).—A volume of a mixture of wine and sulphurous acid of which the content of sulphur dioxide is known is automatically measured in a gauged reservoir furnished with a two-way stop-cock which permits the alternate filling of the reservoir from the main container and the passing of the treated wine into the bottles.

C. RANKEN.

Stabilising of fermented liquids. P. MALVEZIN (F.P. 631,782, 2.7.26).—Liquid with a high content of toxins is obtained by heating to 45° a vigorously fermenting liquid, which, after cooling, is again inoculated with a fresh culture, and the whole process is repeated as often as is necessary to reach the desired degree of toxicity.

C. RANKEN.

Apparatus for the rectification of alcohol, acetone, etc. SOC. DES ETABL. BARBET (F.P. 631,624, 22.6.26).—The first and second of three columns are used for the separation of the first and last runnings, respectively; the third serves for the distillation of the pure fraction. A product of 96% alcohol free from butyl alcohol is obtained.

C. RANKEN.

Waste waters from starch manufacture (F.P. 631,610).—See XVII.

XIX.—FOODS.

Significance of acidity in flour with reference to flour specifications. F. A. COLLATZ (Cereal Chem., 1929, 6, 515—520).—Attention is drawn to the absence of a standard procedure for determining acidity in flour, with special reference to the lack of agreement as to the optimum time and temperature of extraction and the most suitable extracting medium. Twenty samples of flour have been made the object of a collaborative study by (a) the official Greek method, which consists of extraction with 85% alcohol followed by titration with standard alkali against curcuma as indicator, and (b) the tentative official American method, which consists of extraction with water for 1 hr. at 40°. The results showed fairly good agreement by method a, but poor agreement by method b, the correlation coefficient being 0.255 ± 0.149 . Since 85% alcohol inhibits enzymic or bacterial action, it is probable that method a gives a better idea of the actual acid-reacting materials in the flour, but the lack of agreement and unsatisfactory correlation emphasise the need for further study of the methods. (Cf. Brooke, following abstract.)

H. J. DOWDEN.

[Determination of] acidity [in flour] with special reference to the acidity limits imposed by the Greek Government. C. L. BROOKE (Cereal Chem., 1929, 6, 521—529).—The history of Greek legislation relating to the acidity of imported flours is traced to the point where the limit was fixed at 0.160%, with a tolerance of 10% of the maximum during June, July, and August. A search of the literature has revealed that

the adverse physiological effects of flour of high acidity are of little importance. The relative merits of aqueous and alcoholic extraction, together with a list of the limits set by various investigators of the two methods, are discussed and a comparison made of phenolphthalein and curcuma as indicators for the titration.

H. J. DOWDEN.

March of acidity in stored flours. C. C. FIFIELD and C. H. BAILEY (Cereal Chem., 1929, 6, 530—540).—Investigation has been made of the progressive increase in acidity of flours in storage and particularly of the time which elapses before the acidity exceeds the limit prescribed by the Greek Government, viz., 0.15% acidity expressed as sulphuric acid. Samples of flour of different grades were stored at 25°, 30°, and 35° in air of 80% R.H., whilst further samples were kept in a refrigerator at 3—4°. The change in acidity was determined at intervals by the Greek method (alcohol extraction) and by the tentative official American method (aqueous extraction), and it was found that the change of acidity is a function of temperature. Samples of durum first-clear flour, the acidity of which soon after milling was 0.057%, reached the 0.15% limit after storage periods of 90, 110, and 140 days at 35°, 30°, and 25°, respectively, whereas the sample in cold storage had not reached the limit in 190 days. Determinations of p_H were also made, as well as measurements of the amount of protein decomposed as shown by the nitrogen content of compounds peptised by 5% potassium sulphate. There was no indication of decomposition, and subsequent baking tests showed no evidence of unsoundness in the flour even when its acidity exceeded 0.20%. Similar observations on a spring wheat second-clear flour of initial acidity about 0.1% showed that the acidity increased more rapidly than in the case of the durum flour, the 0.15% limit being reached by the samples at high temperatures in about 40 days, and by the refrigerated sample in 50 days. Again there was no indication of unsoundness in the flour. With a patent flour none of the samples had reached 0.15% acidity at the end of 150 days' storage. Graphical representations of the change of acidity show that the ratio of acidity as determined by the Greek method to that determined by the American method tends to decrease as the acidity rises.

H. J. DOWDEN.

Fermentation tolerance [and flour quality]. E. B. WORKING (Cereal Chem., 1929, 6, 506—512).—Fermentation-tolerance curves alone do not form an adequate measure of flour quality. More useful information can be secured by studying separately gas production and dough quality, for it is then possible to adjust the fermentation conditions so that the peak of gas production may coincide with that of dough quality. Before a complete understanding of dough quality can be reached, further study must be made of the influences of such factors as mechanical action, water, acids, alcohols, and fermentation end-products, oxidising agents, phosphatides, and enzymes.

H. J. DOWDEN.

Relation of hydrogen-ion concentration and buffer value to the baking quality of flour. II.

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A correction. E. A. FISHER and P. HALTON (*Cereal Chem.*, 1929, 6, 504—505; cf. B., 1929, 491).—The note acknowledges the priority of Wood's investigation (cf. B., 1907, 1290) on the isolation of gassing power as a separate factor in "strength" determinations.

H. J. DOWDEN.

Nature of the protein extracted from wheat flour by hot alcohol. M. J. BLISH and R. M. SANDSTEDT (*Cereal Chem.*, 1929, 6, 494—503).—Additional evidence has been collected on the source and nature of the excess protein extracted by hot alcohol over that extracted by cold alcohol (cf. J. Assoc. Off. Agric. Chem., 1927, 10, 465). When samples of crude gluten (2 g.) were extracted with (a) 200 c.c. of 70% alcohol for 3 hrs. at room temperature, and (b) with 200 c.c. of boiling alcohol for 1 hr., the protein contents of the extracts were 52.8% and 67.8%, respectively. Since most of the non-gluten protein had been removed, the "excess" protein is probably derived largely from the glutenin. A sample of high-protein flour from which "soluble proteins" had been removed by extraction with 5% potassium sulphate solution was subjected to three successive extractions with boiling 70% alcohol, the protein in these extracts being hydrolysed with strong hydrochloric acid. The percentages of ammonia-nitrogen were 26.1, 23.5, and 20.24, respectively, all of which figures are higher than those previously reported for pure glutenin. The residue after the three extractions was then treated with alcoholic soda, and the extract on hydrolysis yielded 20.1% as ammonia-nitrogen. It appears, therefore, that sharp separation of proteins by extraction with boiling 70% alcohol is not possible, since the alcohol has a "peptising" action on the "glutenin" fraction, which may not be a distinct chemical individual. A sample of purified glutenin was subjected to the repeated action of boiling 70% alcohol, the nitrogen partition (Van Slyke), before extraction, in the extracts and in the residue being determined. The proportions of glutenin peptised were 9.8, 6.2, 4.3, 2.1, 1.9, and 1.6%, respectively. The nitrogen distributions showed that the chemical constitution of the material peptised differs from that of either gliadin or glutenin, and it therefore appears likely that "glutenin" is a derived and not a naturally occurring protein.

H. J. DOWDEN.

Comparing protein determinations in grain with the quick method. H. KUEHL and P. G. GOTTSCHALK (*Cereal Chem.*, 1929, 6, 512—514).—Various modifications in Kjeldahl nitrogen determination have been investigated with a view to shortening the time required for digestion and distillation. (a) Samples of flour (1 g.) mixed with 15 c.c. of phosphorus pentoxide-sulphuric acid mixture (100 g. P_2O_5 in 200 g. of acid), 1 drop of mercury, and potassium sulphate required heating for 15—17 min. to complete the digestion. (b) A suspension of 1 g. of flour in 10 c.c. of 30% hydrogen peroxide was treated with 8 c.c. of phosphorus pentoxide-sulphuric acid mixture. Although the violent reaction produces a colourless solution, digestion is not complete in the cold after keeping for 18 hrs., but when heated digestion is complete in 10 min. The best results were obtained when the flour was digested with peroxide and

1.5 g. of mercury-potassium sulphate mixture, digestion being complete in 2—4 min. The ammonia distillation normally requires 25—30 min., but this time can be reduced to 10 min. by adding to the mixture after digestion 100 c.c. of water, 5 c.c. of 4% potassium sulphide solution, and 50 c.c. of 32% caustic soda.

H. J. DOWDEN.

Phenols in sterilised milk. A. T. R. MATTHEW (Analyst, 1930, 55, 37—38).—Numerous samples of sterilised milks were found during the summer of 1929 to give a positive test for *p*-cresol, and on microscopical examination showed the presence of slender, granulated bacilli. The bacillus was spore-forming, and on inoculation into untainted sterilised milk produced, after 5 days' incubation at room temperature, milks similar in taste to the tainted samples and giving positive *p*-cresol tests. The presence of phenols in milk in sufficient quantity to make it unfit for human consumption is not therefore a necessary indication of fraudulent addition.

D. G. HEWER.

Change in the refractive index [of the serum] during the souring of milk. A. SCHNECK (*Milch. Zentr.*, 1930, 59, 1—8).—According to Elsdon and Stubbs (cf. B., 1928, 346) determination of the refractive index of the calcium chloride serum for the detection of added water is of little value unless the milk be fresh, but it is now shown that the initial refraction of sour milk can be calculated with a fair degree of accuracy. The acidity of the serum (*y*) follows regularly that of the milk (*x*) and is expressed by $y = 0.8x - 2$. The author has also shown (cf. B., 1928, 942) that an increase of 0.026 in the serum solids corresponds to an increase of 1.0 in the serum acidity, and since from Wiegner's tables a change of 0.026 in the serum solids represents a change in refraction of 0.113°, it is deduced that an increase in the "degree of acidity" of milk of 1.0 causes an average rise in the refraction of 0.09°. This deduction has been confirmed on a number of milk samples, the acidities of which were between 15 and 30. The corresponding increase in refraction per degree of acidity for milk acidities down to 7.0 have been calculated from Grimmer and Benduski's results (cf. B., 1929, 735), the value for acidities of 7—8 being 0.27. By applying these corrections in the case of over 100 samples of milk of various acidities, the refractions of the fresh milk have been calculated and the results show fair agreement with the observed values, the accuracy for acidities below 15 being far greater than for higher acidities.

H. J. DOWDEN.

[Separation of the] sterols in butter [by the van Sillevoldt process]. A. MORE (Analyst, 1929, 54, 735—736).—The filtered fat (15 g.) is saponified with 9.5 c.c. of potassium hydroxide solution (1 kg. in 1400 c.c. of water) and 20 c.c. of 96% alcohol. On cooling, 60 c.c. of water and 180 c.c. of 96% alcohol are added, followed, after mixing, by 10—20 c.c. of 1% Merck's digitonin in 96% alcohol. After 24 hrs. the mixture is filtered, the digitonin-sterol compound washed with alcohol, dried, and weighed, and, after acetylation with 10 times its weight of acetic anhydride, is crystallised from alcohol as in the Bömer method. D. G. HEWER.

Fall in Reichert-Meissl values on keeping butter samples. P. ARUP (Analyst, 1929, 54, 736).—Genuine butters (originally containing between 12.4 and 14.7% of water) kept in bottles fitted with metal screw-caps and cork discs showed falls in their Reichert-Meissl values from 20.7 to 8.0 (free fatty acids 27.0%); 21.8 to 16.5 (14.5%); 20.7 to 15.8 (14.0%); 21.1 to 19.5 (7.5%); and 21.5 to 20.8 (6.0%), suggesting that the amount of free fatty acids might be taken as an index of the Reichert-Meissl value. D. G. HEWER.

Changes with age of the hydrogen-ion concentration of egg white and egg yolk and of the refractive index of egg white. J. C. BAIRD and J. H. PRENTICE (Analyst, 1930, 55, 20—23).—The p_H value of the white of 128 eggs laid by unmated pullets was approx. 7.6, with a rapid rise during the first week of storage to about 9.0, at which latter figure it remained constant. The p_H of the fresh yolk was approx. 6.0, rising in 10 weeks to about 6.2. The refractive index of the white was very constant between 1.369 and 1.356. The p_H value is no guide to the age of commercial eggs.

D. G. HEWER.

Furfuraldehyde in heated honey. L. H. LAMPITT, E. B. HUGHES, and H. S. ROOKE (Analyst, 1929, 54, 736—737).—Further experiments with the heated honeys previously described (B., 1929, 697) lasting over periods of from 8 to 12 months indicated no development of furfuraldehyde or of its derivatives. D. G. HEWER.

A standard for potted meat. C. H. MANLEY and R. W. SUTTON (Analyst, 1930, 55, 11—13).—From a consideration and examination of 22 potted meats in which the water content ranged from 55.3 to 79.7% and starch was present in 5 samples, it is suggested that a reasonable maximum for water would be 70% and that starch should be excluded. D. G. HEWER.

Determination of caramel formed during the roasting of ground coffee. A. RIFÉ and L. MEDRANO (Anal. Fís. Quím. [tecn.], 1929, 27, 313—318).—A modification of Hilger's method for the determination is described. By subtraction of a constant (9.5 units), the results obtained are in fair agreement with those given by the original method. H. F. GILLBE.

Determination of husk in cocoa and cocoa preparations. M. WAGENAAR (Pharm. Weekblad, 1929, 66, 1185—1202).—A résumé of the literature dealing with methods based on counts of fragments of husk visible on microscopical examination is given, and a modified method described, based on the characteristic and easily recognised appearance of the sclereides (cellulose cells of the husk) in polarised light. The strength and permanence of these cells permits of ready recognition after the various treatments of the bean, and of the calculation of a mean figure for total surface of husk cells per g. of fat-free powder composed of husks of all the commoner varieties in the proportions in which they come on the market. S. I. LEVY.

Heat-resistance curve: bacteriological test for pasteurised food. C. DUKES (Analyst, 1930, 55, 14—19).—In order to determine whether a food has been

pasteurised or not, heating at suitable dilutions is carried out at temperatures from 50° to 70° and counts are made of the surviving bacteria. If the food has not previously been pasteurised the number of bacteria per g. or per c.c. will progressively diminish, but if it has been pasteurised there will not be marked diminution in numbers till the pasteurisation temperature is exceeded. Details of manipulation are given and figures obtained for samples of Cheddar cheese and milks. The counts are made of colonies visible to the naked eye, and usually three dilutions at six temperatures each are made for each sample. D. G. HEWER.

Rapid limit test for detection and determination of sulphur dioxide in foods. M. HEND (Analyst, 1930, 55, 35—37).—In order to render the Monier-Williams method more applicable to small quantities of material, water to dilute the sample is omitted and a much stronger acid solution used. A mixture of barium benzenesulphonate and hydrogen peroxide is used and the free benzenesulphonic acid titrated. The liquid is filtered into a Nessler glass and titrated with 0.2N-sodium hydroxide. Hydrochloric acid (35 c.c.) is siphoned into the carbon dioxide flask, 3.5 c.c. of hydrogen peroxide and 1 c.c. of 5% barium benzenesulphonate solution are placed in the small Peligot tube, and 0.5 c.c. each of peroxide and sulphonate in the guard tube. After sweeping the air out with carbon dioxide, 10 g. of sample are added, the contents of the flask are boiled within 1 min., and boiling is continued for 10 min. (15 min. for dried fruits). The water is run out of the condenser and when the latter is hot the contents of the Peligot tube and washings from the delivery tube are mixed and the turbid solution is diluted to 70 c.c. and filtered. A current of carbon dioxide is passed through 4 c.c. of hydrogen peroxide in another Nessler tube, 70 c.c. of water are added, 2 c.c. of 0.1% bromophenol-blue solution are added to each tube, and the contents titrated. Some modifications are necessary for liquids, starches, etc. Acetic acid, higher fatty acids, onions, mustard, nutmeg, and hydrogen sulphide did not interfere; the test takes 15—20 min. and gives reasonably accurate results. D. G. HEWER.

Methods of determining boron compounds in food and drugs. II. Experimental. Effect of fats and other organic substances on the determination. III. Conditions required for quantitative titration. A. S. DODD (Analyst, 1929, 54, 715—725; 1930, 55, 23—33. Cf. A., 1929, 362, 668).—II. Samples containing less than 8% of fat or oil may be ignited directly without previous extraction, and no appreciable loss of boric acid will occur provided the sample has been mixed with alkali and dried. When a large proportion of fat is present the determination of boric acid may be made on the fat-solvent portion without first extracting with alkali, but the exclusion of phosphates must be ensured. Different kinds of fats and oils give rise to different characteristic losses of boric acid on ignition in the presence of alkali, and this may prove useful in identifying and determining the glycerol contained in compounds or mixtures. The only likely cause of loss of boric acid on boiling the acidified solution is too rapid

boiling, but appreciable loss occurs when the solution is concentrated to about one fifth of its original bulk.

III. Experiments relative to the titration of boric acid solutions show that lactose or galactose may be used instead of glycerol or mannitol, but the fact is of merely theoretical interest. Boric acid has a titration curve varying from about p_H 6 to p_H 11, but excessive addition of mannitol increases the acidic intensity from p_H 6 to p_H 3, and the end-point of the acid complex formed by boric acid and mannitol is about p_H 7.2. A preliminary indicator is used to eliminate acidity due to other acids, the most suitable indicators being methyl-red, diethyl-red, sofno I, and bromocresol-purple, with phenol- or cresol-phthalein as final indicator. If concentrated solutions of boric acid and strong acids are used, the end-point is indefinite owing to buffer action, and solutions should not contain more than about 0.1 g. of borate or boric acid and 0.05N- or 0.1N-sodium hydroxide should be used for titration. Mannitol is the most suitable substance for forming an acid complex.

D. G. HEWER.

Use of copper sulphate in place of mercury in Kjeldahl decompositions. F. MACH and W. LEPPER (Landw. Versuchs-Stat., 1929, 109, 363—366).—In the application of the Kjeldahl method to the analysis of feeding-stuffs the use of copper sulphate leads to as rapid decomposition as when mercury or its sulphate is employed, and the results are identical. Potassium sulphate may generally be added at the commencement of the decomposition process. Use of graphite in place of zinc is preferable to minimise "bumping" during distillation.

A. G. POLLARD.

PATENTS.

Determination of the moisture content of cheese, curd, etc. C. HACKMANN (G.P. 456,945, 15.8.26).—An aqueous solution of high density, *e.g.*, a solution of zinc, stannous, or ferric chloride, which is saturated with the water-soluble constituents of the solid substance, is intimately mixed with the solid substance and the change in density determined. C. RANKEN.

Agglomeration process for [beet] pulp from sugar factories and a resulting product intended for cattle feeding. Soc. ANON. DES SUCRERIES TERNYNCK, and L. TERNYNCK (B.P. 320,883, 13.4.28).—Fodder-briquettes which rapidly disintegrate in water are made from dried beet pulp and 20—30% of molasses, in an apparatus comprising skips which automatically deliver requisite charges of pulp and molasses to a mixing vessel, whence the mixture is discharged to a briquetting press operating at 120—150 kg./cm.² (cf. B., 1929, 733).

J. H. LANE.

Manufacture of (A) phosphorus compound, (B) iron compound, from animal proteins. S. and T. POSTERNAK, Assrs. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,743,003—4, 7.1.30. Appl., [A] 21.3.27, [B] 4.1.28. Switz., [A] 31.3.26, [B] 17.1.27).—See B.P. 268,806 and 283,866; B., 1928, 35; 1929, 71.

[Apparatus for] pasteurisation [and deodorisation] of milk, cream, etc. A. H. TEMPLETON (B.P. 323,469, 28.9.28).

Manufacture of [ultra-violet ray-treated] chocolate. E. OPPENHEIM (B.P. 309,601, 11.4.29. Czechoslov., 13.4.28).

Pulverulent mixtures (B.P. 314,941).—See I. Compositions from cellulose derivatives (B.P. 322,464).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Manufacture of formaldehyde and of some of its pharmaceutical derivatives. J. SCHWYZER (Pharm. Ztg., 1930, 75, 28—30, 45—46).—A modern plant for the preparation of formaldehyde from methyl alcohol and air is described. Details are also given for the preparation of paraformaldehyde, hexamethylenetetramine, anhydromethylenecitric acid, and its hexamethylenetetramine salt.

H. BURTON.

Stability of anæsthetic ether in containers of various types. F. VAN DERIPE, L. W. GREEN, and R. E. SCHOETZOW (J. Amer. Pharm. Assoc., 1929, 18, 1228—1229).—Ether stored in internally copper-plated tin cans for 15 months in the dark at room temperature contained neither peroxides nor aldehydes; under similar conditions, ether stored in amber bottles and tin cans contained large amounts of these substances (cf. Nitardy and Tapley, B., 1929, 72). In the aldehyde test the smaller are the particles of potassium hydroxide, the more readily is the coloration produced, but powder must not be used. A solution of potassium hydroxide is not as effective as the solid, and it is essential that the pure material be employed.

E. H. SHARPLES.

Quantitative analytical methods for the study of peroxide formation in ether. R. VAN WINKLE and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1929, 18, 1247—1250).—Methods for determining peroxides and dissolved oxygen in ether based on the determination of the iodine liberated from cadmium-potassium iodide and the Winkler method for dissolved oxygen in water are described. The peroxide method bears a definite relation to the U.S.P. qualitative method. Ether containing large amounts of peroxide gave a white precipitate with the cadmium-potassium iodide. It contained 42.16% Cd and 28.84% I, and was not explosive.

E. H. SHARPLES.

Incompatibility between sodium salicylate and sodium bicarbonate. J. C. KRANTZ, JUN., and C. J. CARR (J. Amer. Pharm. Assoc., 1929, 18, 1250—1251).—The discoloration of mixtures of sodium salicylate and sodium bicarbonate in aqueous solution is not due to impurities in the salicylate. Purified natural sodium salicylate darkens in aqueous solution in the presence of sodium bicarbonate, and the rapidity of the development of darkening is a function of the concentration of either the sodium salicylate or the sodium bicarbonate (cf. B., 1929, 188).

E. H. SHARPLES.

Assay and determination of composition of theobromine-sodium salicylate. H. MATTHES and P. SCHÜTZ (Pharm. Ztg., 1930, 75, 42—44).—Determination of the theobromine content of various samples of theobromine-sodium salicylate and diuretin by the

iometric method (Boie and Lindner, B., 1929, 416; cf. Emery and Spencer, A., 1918, ii, 380) gives values in good agreement with those deduced from the nitrogen content (Kjeldahl). The methods recorded in D.A.B. V and VI give results which are approx. 10 and 15% too low, respectively.

H. BURTON.

Medicinal wood-tar creosote. I. Methoxyl content as a criterion of the composition of creosote. D. V. SICKMAN and R. P. FISCHER (J. Amer. Pharm. Assoc., 1929, 18, 1145—1149).—A method for the evaluation of medicinal creosote based on the density, U.S.P. distillation test, and methoxyl determination is described. A wide variation in composition was shown in the 16 commercial samples of creosote examined. About 50% of creosote is composed of creosol and guaiacol, and there is at least twice as much creosol as guaiacol. The U.S.P. requirements of density and distillation range are sufficient to insure a methoxyl content of at least 11%, which corresponds to a guaiacol and creosol content of 44—49%. There is no distinction between beechwood and other hardwood creosotes.

E. H. SHARPLES.

[Constitution of] commercial "glyzines" [liquorice extracts]. A. BONIS (Ann. Falsif., 1929, 22, 518—522).—Commercial extracts of liquorice contain, in addition to the ammonium salt of glycyrrhizic acid, a bitter-tasting constituent from which it is difficult to free the glycyrrhizin. This material contains nitrogen and the discrepancies between the analyses recorded for glycyrrhizic acid are probably due to the varying proportions of this substance contained by liquorice of different origins. For the removal of this compound, Bertolo (cf. B., 1925, 861) recommends preparing the cadmium salt of glycyrrhizic acid, washing out the impurity with hot water, decomposing the salt with hydrogen sulphide, and recrystallising the free acid from acetic acid. A number of samples of commercial liquorice have been analysed by the methods of Auguet and of Durier (cf. Ann. Falsif., 1909, 387; 1913, 254), which consist of precipitating glycyrrhizic acid with alcohol and transforming it into the ammonium salt. The results varied considerably, for two samples contained only 43.52% and 50.16% of ammonium glycyrrhizate, whilst several others of different origin contained 81—82.5%. To investigate the cause of such variations the filtrates after removal of the acid were neutralised exactly and examined for optical activity and reducing powers. The residues from samples of high glycyrrhizin content gave negative results, but those from the samples assaying 40—50% were found to be dextrorotatory and to have strong reducing powers, suggesting that the samples had been adulterated with dextrin. The usual methods of analysis give high values, which are nevertheless proportional to the true glycyrrhizin content. Calculation of the glycyrrhizin content from the ammoniacal nitrogen displaced by magnesia, however, gives erroneous results. Thus the sample containing 43.5% of glycyrrhizin as determined by the precipitation method gave a value of 79% when calculated from the ammoniacal nitrogen content, whilst determination of total nitrogen by the Kjeldahl method,

and assuming the formula $\text{NH}_4\text{C}_{44}\text{H}_{62}\text{O}_{18}\text{N}$, gives an assay of 100% for the same sample. H. J. DOWDEN.

Standardisation and stabilisation of ergot preparations. Biological methods of assaying ergot preparations and the hydrogen-ion concentration factor. E. E. SWANSON (J. Amer. Pharm. Assoc., 1929, 18, 1127—1137; cf. A., 1929, 955).—The U.S.P. cock's comb and reversal uteri (Broom and Clark) methods give similar results on a number of fluid extracts and solutions of ergot, but the latter method involves more skill and technique. Determinations after 2 yrs. ageing of the samples indicated that the cock's comb method is less reliable as the deterioration increases. The hydrogen-ion concentration has some influence in controlling the deterioration and stabilisation, and a p_{H} value of 3 is necessary to prevent deterioration of fluid extract of ergot. The solution of ergot for hypodermic use deteriorates rapidly. Results obtained from a large number of tests indicate that 1 mg. of ergotoxine base is equivalent in activity to 1.3 mg. of ergotamine base.

E. H. SHARPLES.

Rapid evaluation of fluid extract of ergot. K. IERING (Apoth.-Ztg., 1929, 44, 542; Chem. Zentr., 1929, ii, 463).—The extract (1 g.) is vigorously shaken for 30 sec. with light petroleum (5 g.) and water (5 g.); the aqueous solution, after removal of the petroleum, is similarly extracted with ether (5 g.), and the ethereal solution is poured on to the surface of 2 c.c. of sulphuric acid containing 1 drop of 1% ferric chloride solution. Good samples give a stable blue or bluish-violet ring; otherwise, the ring is accompanied by brown or grey rings.

A. A. ELDRIDGE.

Digitalis assay standards. L. W. ROWE (J. Amer. Pharm. Assoc., 1929, 18, 1138—1142).—Data representing average experiences over a period of years with comparative tests of representative samples of tincture of digitalis by five methods are given. The standard set for the Cat (Hatcher) method is at least 10% higher than that set for the One-Hour (U.S.P.) method and the Four-Hour Frog (Geneva Conference) method, whilst the standard set for the M.L.D. Frog (Houghton) method is not more than 10% lower than the present U.S.P. standard. The M.L.D. Guinea-pig method has probably a correct standard, equal to those of the One-Hour and Four-Hour methods. The present U.S.P. standard for tincture of digitalis, ouabain, is unsatisfactory. It is suggested that the "International Standard" digitalis leaf should be adopted as the standard. The official U.S.P. method is not satisfactory, and it is recommended that an M.L.D. method with a time limit of at least 6 hrs. be substituted.

E. H. SHARPLES.

Nicotine content of tobacco. IV. A. HEIDUSCHKA and F. MUTH (Pharm. Zentr., 1929, 70, 677—680; cf. B., 1929, 869).—Data are given for the nicotine content of the tobacco and tobacco smoke of certain varieties of cigar and cigarette, with particular reference to the so-called nicotine-free varieties.

E. A. LUNT.

p_{H} studies of neoarsphenamine. R. B. SMITH, A. E. JURIST, and W. G. CHRISTIANSEN (J. Amer. Pharm.

Assoc., 1929, 18, 1142—1145; cf. B., 1928, 243).—The method of Elvove and Clark (A., 1924, ii, 592) has been successfully applied for the determination of the p_H of neocarsphenamine. In 20% solution, 9 experimental samples showed a p_H range of 6.31—8.74, and 5 commercial samples a range of 5.78—8.48. Five samples on dilution from 20% to 4.5% showed an increase in p_H of from 0.09 to 0.34 unit, and another sample on dilution from 40% to 0.1% showed a steady rise of p_H from 7.60 to 8.62. E. H. SHARPLES.

Determination of arsenic in Natrium cacodylicum, D.A.B. VI. G. FRIEDLÄNDER (Apoth.-Ztg., 1929, 44, 528—529; Chem. Zentr., 1929, ii., 463).—When carried out under the exact conditions described by the author, the method of D.A.B. VI gives concordant results. A. A. ELDRIDGE.

Fertilisers and tobacco. OTRIGANIEV. **Curing of tobacco.** SMIRNOV and DRBOGLAV.—See XVI. **Determination of boron in drugs.** DODD.—See XIX.

PATENTS.

Administration of anæsthetics. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 322,790, 13.9.28).—The exhaled breath of the patient is passed to a filter containing a highly absorptive mass, such as activated carbon, alone or combined with silica gel, which warms up owing to the heat of the breath and the condensation of moisture and in turn warms the mixture of air and anæsthetic vapour to be inhaled. S. K. TWEEDY.

Manufacture of thymol and menthol. SCHERING-KAHLBAUM A.-G. (B.P. 293,874, 13.7.28. Ger., 15.7.27. Addn. to B.P. 273,685; B., 1930, 136).—The condensation product from *m*-cresol and acetone is acetylated and distilled at 300—320°. The resulting mixture of 4-*isopropenyl-m*-cresol and its acetate no longer tends to polymerise during reduction and may be hydrogenated by the process of the prior patent. Formyl derivatives may similarly be used. C. HOLLINS.

"Fixation" of perfumes. DEUTS. HYDRIERWERKE A.-G. (G.P. 455,824, 9.7.26. Addn. to G.P. 373,219; B., 1923, 953 A).—The perfume is dissolved in an ester or mixture of esters of dicarboxylic acids (other than adipic acid), formed by the oxidation of hydrogenated phenols. Thus the *isobutyl* ester of methyladipic acid, obtained by oxidation of hexahydrocresol, dissolves 30% of its weight of crystalline musk. W. J. BOYD.

Manufacture of toilet material. T. HASHIMOTO (B.P. 323,321, 17.11.28. Addn. to B.P. 322,607; B., 1930, 122).—A thin fluid material for application to the skin is made by first boiling under pressure an emulsion produced by mixing a solution of india-rubber, *e.g.*, in ether or benzene, with one of gum tragacanth; the volatile solvent is then expelled by distillation under reduced pressure. The liquid product, to which antiseptics etc. may be added, has no coagulative property and no adhesive character peculiar to gum.

D. F. TWISS.

Electric vaporisers [for medical purposes]. J. ROBINSON (B.P. 318,214, 5.11.28. U.S., 31.8.28).

Bandaging material (B.P. 298,606).—See V.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Absorption spectra of dye-gelatin layers before and after exposure to daylight. F. LEIBER (Kolloid-Z., 1930, 50, 68—74).—The absorption spectra of light filters made by impregnating gelatin with various dyes have been measured and are given in the form of curves. E. S. HEDGES.

PATENTS.

Manufacture of diazotype [light-]sensitive [photocopying] papers and the like. H. D. MURRAY, and NORTON & GREGORY, LTD. (B.P. 320,395, 14.6.28).—The acid necessary to neutralise the alkali excess in the paper surface is applied to the paper before exposure; *e.g.*, hard-surface papers may be coated with 1% aqueous gelatin containing 4% of oxalic acid, or for more absorbent papers a 10% solution of ammonium chloride may be applied to the back of the paper. C. HOLLINS.

Production of photographic pictures by the aid of diazo compounds and tanning substances. KALLE & Co. A.-G. (B.P. 296,008, 22.8.28. Ger., 22.8.27).—A light-sensitive diazo-compound is applied together with a colloid (gelatin) to a suitable carrier (paper, film, etc.), with or without a stabilising agent, and after exposure under a negative or diapositive and treatment with a tanning agent (*e.g.*, 2% potassium dichromate) the picture is developed with water. Suitable diazo-compounds are *p*-dimethylaminobenzene-diazonium fluoborate, 5-dimethylaminotoluene-*o*-diazonium sulphate, 6-acetamido-4-sulphobenzene-1:2-diazo oxide, 3-carboxynaphthalene-1:2-diazo oxide. C. HOLLINS.

Manufacture of sensitised element and silver halide emulsion therefor. O. MATTHIES, W. DIETERLE, and B. WENDT, ASSRS. to AGFA ANSCO CORP. (U.S.P. 1,742,042, 31.12.29. Appl., 5.1.28. Ger., 7.1.27).—See B.P. 283,223; B., 1929, 577.

Colour photography. SOC. FRANÇ. DE CINÉMATOGRAPHIE ET DE PHOTOGRAPHIE FILMS EN COULEURS KELLER-DORIAN (B.P. 297,773, 21.9.28. Fr., 27.9.27).

Process and apparatus for reproducing kinematograph films in colours. SOC. FRANÇ. DE CINÉMATOGRAPHIE ET DE PHOTOGRAPHIE FILMS EN COULEURS KELLER-DORIAN (B.P. 304,643, 29.6.28. Fr., 23.1.28).

Cellulose compositions (B.P. 322,464).—See V.

XXII.—EXPLOSIVES; MATCHES.

Sensitiveness to detonation and velocity of explosion of gelatin dynamite. H. SELLE (Z. ges. Schiess-u. Sprengstoffw., 1929, 24, 469—474).—The conditions that determine whether gelatin dynamite explodes with a high velocity (about 6000 m./sec.) or a low one (below 3000 m./sec.) are discussed. When a No. 8 detonator is used as the initiating agent, the explosion velocity increases with the diameter of the cartridge, being 900 m./sec. with a 16-mm. cartridge and 2750 with a 30-mm. When 10 g. of compressed picric acid are

used as initiator, the velocity is above 6000 m./sec. in all cases. The explosion velocity decreases with the age of the explosive, falling in the case of a 20-mm. cartridge from 2250 m./sec. for a 2-day old cartridge to 1250 after 400 days. When an old cartridge is kneaded before use it explodes with its original velocity. Although the explosion velocity decreases with age, the strength of the explosive remains practically unaltered after 12 months' storage.

S. BINNING.

PATENTS.

Blasting cartridges. D. HODGE (B.P. 322,837, 5.10.28).—Blasting cartridges containing liquid carbon dioxide are set off by heat obtained by firing an explosive contained in a heater unit in such a way that the combustion in the explosive proceeds only over a predetermined portion of the explosive, thus maintaining the rate of heat evolution at any desired figure. The initiation of the reaction may be effected by fusion of a metal wire, or ignition of a match-head or squib electrically.

S. BINNING.

Primer composition. H. RATHSBURG, Assr. to RHEINISCH-WESTFÄLISCHE SPRENGSTOFF-A.-G. (Re-issue 17,540, 31.12.29, of U.S.P. 1,586,380, 25.5.26).—See B., 1926, 693.

Cellulose for nitration (B.P. 322,997). **Nitro-cellulose** (B.P. 322,998 and 323,019).—See V.

XXIII.—SANITATION; WATER PURIFICATION.

Treatment of spent gas liquor in admixture with sewage at the sewage purification works at Cheltenham. F. R. O'SHAUGHNESSY (Gas J., 1929, 188. Inst. Gas Eng. Suppl., 64—70).—The crude sewage-settling tank was first examined when no gasworks' spent liquor was being admitted to the sewers. It was shown that the Cheltenham sewage was a weak domestic sewage, fairly regular in character during dry weather. The final effluent was of good quality, being well aerated, non-putrefactive, and complying with the Royal Commission Standard; the purification on the 4-hr. oxygen standard was 80—90%. The influence of gas liquor in the proportion of 0.5% of the dry-weather flow of sewage was next investigated. It was found that the samples of sewage containing gas liquor remained inoffensive while kept in the laboratory. Gas liquor would thus help to keep down offensive odours, both in the sewers and while passing through the settling tanks; the final effluent remained bright, with an increase in the oxidised nitrogen due to the larger amount of ammonia in the sewage. All the samples were non-putrefactive and the Royal Commission test gave satisfactory results. Close observation of the bacteria beds during the period of the test showed that their condition was not impaired and the life therein remained vigorous and healthy. Laboratory experiments were also made on the relative "facility of treatment" of spent gas liquor by biological oxidation. On adding an average sample of the gas liquor to the settled tank-liquor in the proportions of 0.0%, 0.5%, and 1% it was found that the more dilute solution of gas liquor

was much more amenable to biological treatment. It is concluded that spent gas liquor, if admitted to the sewers in proportions up to 0.5% of the dry-weather flow, can be satisfactorily purified at the sewage works, without detriment to the beds, and that a satisfactory effluent can be maintained after continued working.

C. B. MARSON.

Operating experiences with a large [Canadian water] filter plant. J. C. KETH (J. Amer. Water Works' Assoc., 1929, 21, 1629—1639).—The Essex Border Utilities Commission plant, which has a nominal capacity of 21,000,000 gals./day, is jointly owned by nine communities with a total population of 120,000. The supply is obtained from the Detroit River and is a typical Great Lakes water. The normal coagulant is aluminium sulphate, but algal troubles, which in June, 1927, reduced the length of filter runs to 1½ hrs., were overcome in the corresponding period of 1928 by the substitution of ferrous sulphate and lime. The added cost was justified by the results obtained, the service hours of the filters being extended to a minimum of 10. The operating costs averaged \$12.58 per million gallons, of which wages accounted for 34.1%, power for 21.7%, and coagulants for 16.7%.

C. JEPSON.

Mechanical developments in water treatment practice. C. T. LEANDER (J. Amer. Water Works' Assoc., 1929, 21, 1619—1659).—Dorr impellor agitators, which consist essentially of a marine propeller revolving slowly on a vertical shaft so as to produce an upward flow in the surrounding cylinder and a downward flow in the outer annular space, are claimed to circulate the contents at a velocity of 0.5—1.0 ft./sec., which will enable the floc to build up readily and at the same time prevent settlement on the floor of the tank. The substitution of continuous, mechanically cleaned sedimentation tanks for those of the intermittently cleaned type results in reduced costs by operating the plant under uniform conditions and so simplifying mechanical and chemical control. In the case of very turbid waters, such as are prevalent in the Mississippi-Missouri region, sedimentation of the raw water prior to chemical treatment is recommended.

C. JEPSON.

Barium aluminate and its use in water purification. R. STUMPER (Chim. et Ind., 1929, 22, 1067—1083).—The use of barium compounds (for precipitating sulphates) and of aluminium salts (for clarification) is familiar in water treatment. The combination of the two reagents as barium aluminate, which would also precipitate calcium and magnesium, therefore appears attractive. Barium aluminate is difficult or impossible to prepare as a definite compound of constant composition. The commercial product is a brown powder containing considerable quantities of barium sulphate, alumina, etc. The solution prepared by adding 1 pt. of the compound to 10 pts. of water, as used in practice, contains 2.04% BaO and 1.17% Al₂O₃, corresponding approximately to the formula 11BaO.8Al₂O₃ for the soluble compound. The apparent solubility and the BaO : Al₂O₃ ratio in solution both vary with the amount of solid "aluminate" present, owing to effects of hydrolysis, variation in *p_H* value, and the tendency of

alumina to form colloidal solutions. The original saturated solution commences to deposit alumina after a few hours, and this precipitation continues for about two months. The solution on addition of calcium or magnesium salts yields precipitates of the composition $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ and $2\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$. The solubility of the former decreases, but that of the latter increases, with rise in temperature. The solution in theoretical quantity precipitates sulphates from solution almost completely even in the cold. About twice the theoretical quantity is required for a 90% precipitation of calcium, but the precipitation of magnesium is more complete; in both cases the anion exercises a modifying influence, elimination being the more complete in the case of sulphate owing to entrainment and adsorption. In the case of solutions containing both sulphates and bicarbonates the reactions are very complex. In general, precipitation is more complete than with the single salts, the barium carbonate first formed reacting again with calcium sulphate. If oil be present in a water treated with barium aluminate the oil will be carried down by the precipitate and carbon dioxide in solution will also be removed by this reagent. As the original complex precipitates formed by addition of barium aluminate to hard water have a further purifying action, they should be left in contact with the water as long as possible. C. IRWIN.

Chloroamines in the disinfection of water. J. J. HINMAN, JUN., and K. C. BEESON (J. Amer. Water Works' Assoc., 1929, 21, 1705—1716).—In clear waters of low organic content the addition of ammonia with the chlorine always retards the rate of disinfection and also reduces the final efficiency unless the quantity of chlorine is greatly in excess of that of ammonia. In turbid waters of high organic content the addition of ammonia increases the germicidal velocity of the chlorine, though after-growths sometimes occur. The method is recommended for the treatment of raw or unfiltered waters, especially if long periods of detention are available after chlorination and before the water reaches the consumer. The values 1:2 and 1:3 for the $\text{Cl}:\text{NH}_3$ ratio were found to give satisfactory results. No loss of free or saline ammonia was detected during the contact period and under the conditions of the experiments. C. JEPSON.

Prevention of chlorophenol tastes [in water] in New York State. C. R. COX (J. Amer. Water Works' Assoc., 1929, 21, 1693—1704).—Observations made on the three methods of dealing with chlorophenol tastes suggested by Houston indicate that (a) superchlorination is effective but costly, although it also improves the general character of the treated water by oxidising some of the organic matter; (b) ammonia treatment applied before the chlorine dose effectively prevents the formation of chlorophenol compounds; the chloroamines produced, however, are less active disinfectants than the corresponding quantities of chlorine, and therefore the method should not be applied to heavily polluted waters without proper safeguards; (c) potassium permanganate may be added to raw, filtered, or filtered and chlorinated water, but is not universally satisfactory owing to the occasional production of secondary

tastes of a bitter nature. The cost is moderate and there is little likelihood of causing stains on clothes etc., especially if added to the raw water. Care is necessary to compensate for the influence of manganese on the o-tolidine test for residual chlorine. C. JEPSON.

Tinned-iron kettles. DUNN and BLOXAM.—See X.

PATENTS.

Aeration of sewage and other liquids. W. BUTLER and E. N. TABOR (B.P. 323,156, 1.3.29).—The long, narrow channels employed in the paddle system of mechanical circulation as applied to the activated-sludge process of sewage treatment are subdivided longitudinally by means of a false bottom so arranged that the sewage during its progress along the channel passes from the upper to the lower sections and *vice versa* at regular intervals. By this means it is claimed that the rate of dissolution of oxygen is increased owing to the regular interchange of the liquid in contact with the atmosphere, and also to the presence in the lower section of an air cushion under pressure, which cushion may be renewed automatically by entrained air or mechanically by being forced through diffusers laid on the floor of the tank. C. JEPSON.

Purification of waste water containing sludge. W. LEONARDY (G.P. 456,977, 14.2.24).—Precipitation and deposition of the solid material are facilitated by the provision of a heating coil in the upper part of a sludge container, and of means for altering the direction of flow of the sludge to cause the solids to fall to the bottom. L. A. COLES.

Clarification and decolorisation of effluents from dyeworks. G. ULLMANN (G.P. 457,467, 15.3.25. Austr., 22.1.25. Addn. to G.P. 438,268. Cf. Austr. P. 100,735; B., 1926, 470).—The wood or straw pulp previously mentioned is replaced by hair, leather residues, or other animal waste material which adsorbs dyes. A. R. POWELL.

Utilisation of waste waters from beetroot sugar molasses [for production of glutamic acid]. K. IKEDA (U.S.P. 1,721,820, 23.7.29. Appl., 17.12.27).—The waste water from which sugar has been wholly or partially removed is treated with sulphuric acid to remove potash, the bulk of the acid is removed as calcium sulphate after heating, and glutamic acid is obtained by crystallising the filtrate from the calcium sulphate (cf. B.P. 248,453; B., 1926, 420). H. J. DOWDEN.

Recovery of oily substances and purification of water containing them. E. B. WARREN (B.P. 323,094, 23.11.28).—When oil-contaminated water is brought into intimate contact with rubber, the grease is absorbed thereby and may be recovered by the use of a suitable solvent. C. JEPSON.

Rendering harmless substances which cause hardness in soaping and washing operations, and production of means suitable therefor. G. ULLMANN (U.S.P. 1,743,054, 7.1.30. Appl., 11.2.26. Austr., 6.3.25).—See B.P. 259,437; B., 1927, 30.

Face masks for protection against dust or poisonous gases. H. GOEKE (B.P. 323,164, 18.3.29).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAR. 14 and 21, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat transfer from a gas stream to a bed of broken solids. C. C. FURNAS (Ind. Eng. Chem., 1930, 22, 26—31).—The transfer of heat from air (of reduced oxygen content to minimise oxidation) to a bed of iron balls of varying sizes and covered with a thin layer of rust was studied. Shielded thermocouples gave the temperature of either solid or gas as required. The balls used were from 1.85 to 4.86 cm. in diam., air flow was from 0.01 to 0.053 litre/sec./cm.², voids 39.5—50.6%, and temperatures up to 700°. It was found that the coefficient of heat transfer from gas to solid varies with the 0.75 power of the gas velocity with constant temperature. With constant flow it is directly proportional to the temperature. Owing to the small size of the apparatus it was not possible to vary the particle size and the percentage of voids separately, but from certain assumptions it was concluded that with constant voids the coefficient per cm.² of surface was independent of the particle size. The conclusions are summarised in equations.

C. IRWIN.

Efficiency of drying by hot air relative to the maximum permissible temperature of the material. O. VAJDA (Chem. Fabr., 1930, 25—27).—Graphs are given showing the relationships for a continuous hot-air dryer between (1) thermal efficiency and exit temperature, (2) total energy efficiency, including that required to move the air, and exit temperature. The total energy requirement falls with increase of temperature, but the permissible maximum temperature is limited by the nature of the material to be dried. The relation between these two temperatures depends on whether the dryer works on the countercurrent principle or not. In the case of hygroscopic materials the degree of saturation of air which will effect drying is reduced in proportion with the reduction of vapour pressure by the material in solution. With the air and material moving in the same direction, the exit-air temperature is the maximum temperature of the material. In a countercurrent dryer, safety under working conditions demands that the inlet-air temperature shall not exceed this maximum, but the reduction in possible saturation by hygroscopicity does not apply. It is shown that for low maximum temperatures the countercurrent dryer is the more economical, but for higher ones it is less so. The more hygroscopic the material, the greater is the advantage of the countercurrent method.

C. IRWIN.

Boiler reactions at high temperatures. W. L. DENMAN and E. BARROW (Ind. Eng. Chem., 1930, 22, 36—39).—Corrosion tests were carried out in a laboratory

boiler operating at a maximum pressure of 16 kg./cm.², using charcoal-iron plates. These were cleaned from oxide by using them as cathodes in an electrolytic cell. The tests were for 24-hr. periods, and in less than that time the dissolved oxygen was exhausted and corrosion had ceased. The amount of corrosion was found to be directly proportional to the dissolved oxygen content. Tests were also run with the addition of pyrogallol, oak extract, and sulphur dioxide. All reduced corrosion, oak extract in alkaline solution being found the most effective. Amongst inhibitors sodium arsenite was found to reduce corrosion, potassium dichromate was difficult to use effectively in the presence of halogens, the film being broken down with local corrosion, and if insufficient potassium dichromate was used corrosion was in any case increased. Some corrosion tests were also run with circulated water of varying hardness etc. The concentration of boiler water can be conveniently determined by the chloride content compared with that of the feed water.

C. IRWIN.

Internal corrosion of hot-water systems by oxygen and carbon dioxide, and exterior injury to boilers by unsuitable fuel. G. OFFE (Gas- u. Wasserfach, 1930, 73, 73—76).—Oxygen and carbon dioxide in the presence of moisture at 70—80° produce visible corrosion on sheet iron in 12 hrs. and on zinc in 24 hrs. Efficient de-aerators are therefore necessary in hot-water plants employing these metals. The correct design of the boiler and arrangement of its connexions with a view to good circulation and elimination of gases is important. Other cases are recorded where failures of small boilers and water heaters were due to sulphur in the fuel (in one case wood waste, in another coke). Iron sulphide and free sulphur were found in the corrosion product, the air supply having been at times deficient. Such corrosion is much more severe than that produced by the equivalent of sulphur dioxide. It is desirable to use good oven coke or gas coke burnt without the employment of steam.

C. IRWIN.

[Boiler-]water softening. J. GORDON (Gas World, 1930, 92, Coking Sect., 10—14).—Figures for hardness of the feed water and boiler water in an installation in which water softened by addition of sodium hydroxide only was evaporated showed that considerable softening occurred within the boiler itself. With feed water at 12° average hardness, the average hardness and average alkalinity (grains/gal.) of the boiler water were 4° and 20.6, respectively. It is concluded that the water after softening contains sodium bicarbonate, which forms sodium carbonate in the boiler, this then reacting with calcium sulphate.

S. I. LEVY.

* The remainder of this set of Abstracts will appear in next week's issue.

R. Fischer's fall viscosimeter. H. L. MATTHIJSEN (Chem. Weekblad, 1930, 27, 20—23).—The apparatus is more suitable than the Engler viscosimeter for determinations with very viscous fluids at 20°. The results obtained with various tars, varnishes, and paints are tabulated.

S. I. LEVY.

The Ostwald colour system. F. SCHOLEFIELD (J. Soc. Dyers & Col., 1930, 46, 5—7).—The method of construction of the Ostwald "isochromatic triangle" is described. Each triangle contains the "bright clear range," the "dark clear range," and every possible flat colour derivable from the particular full colour. The isochromatic triangles may be combined to form a "colour body" in the form of a double cone, of which the periphery passes through all the full spectral colours and of which the axis represents the "unhued" range, with white at one end and black at the other. Colours harmonising with that represented by a particular point within the colour body are found on three straight lines and also on a circle passing through that point.

N. CHAPPELL.

Steam and power in cellulose works. MALM.—See V.

PATENTS.

Furnaces burning pulverulent fuels. A. W. BENNIS (B.P. 323,219, 25.7.28).—The combustion chamber diverges from a grid at the bottom to the boiler at the top, primary air and fuel being admitted in a downwardly inclined direction from the upper part. Part of the secondary air is supplied to the interiors of the hollow bars composing the grid, is thus preheated, and enters the furnace with a constant upward velocity. The grid consists of a pair of squirrel-cage rollers or other moving device to discharge ash into a water-cooled conveyer. Another part of the secondary air may be preheated in the furnace walls.

B. M. VENABLES.

Furnace fronts. [Burners for liquid and pulverised fuels.] (A) W. A. WHITE; (B) W. A. WHITE, and WHITE PATENT OIL BURNING Co., LTD. (B.P. [A] 323,578, 17.10.28, and [B] 323,661, 22.1.29).—Controllable devices are described which admit secondary air around burners for liquid or pulverised solid fuel, and afford provision for rapid change over of the fuel. In (A) reference is mainly to the arrangement of four frusta of cones forming the walls of passages for secondary air, and in (B) to mounting of the Venturi tube, and to flame and air controls upon a double-hinged door, whilst the fuel supply valve is attached to a fixed part.

B. M. VENABLES.

[Tunnel] kiln. H. M. ROBERTSON (U.S.P. 1,737,540, 26.11.29. Appl., 25.2.28).—In a tunnel kiln combustion takes place in side chambers which are constructed close to the trucks of material at the hottest zone. The products of combustion are led through conduits in the side walls of the preheating zone, and transverse air-circulation is maintained over these conduits and the goods. Air is drawn through the side walls of the cooling zones and is used for combustion.

B. M. VENABLES.

Retorts, kilns, dryers, heat interchangers, etc. C. H. LANDER and T. F. HURLEY (B.P. 323,715, 6.7.28).—Apparatus specially suitable for the transmission of heat

into coal for the purpose of low-temperature distillation comprises a vertical retort of which some or all of the walls are composed entirely of tubes through which heating medium is passed from an inlet header at one end to an outlet header at the other: the material travels longitudinally. The walls may be inclined to allow for expansion (or contraction) of the material during treatment, and gases may be permitted to leave (or enter) between the tubes anywhere or only at predetermined points. The walls (if any) not composed of tubes may be constructed of castings or brickwork.

B. M. VENABLES.

Heat exchanger. E. HABER (U.S.P. 1,737,189, 26.11.29. Ger., 31.10.23. Appl., 11.11.24).—Regulating devices are described for the flow of the fluids through a recuperator constructed of steel plates.

B. M. VENABLES.

Heat exchanger. R. E. WILSON, ASSR. to STANDARD OIL Co. (U.S.P. 1,737,347, 26.11.29. Appl., 22.1.27).—A heat exchanger consisting entirely of straight tubes that can be easily opened and cleaned is constructed of a solid block of metal in which the tubes are drilled or cast. They are connected by detachable headers so that alternate ones carry the different fluids and so that the flow is, as far as possible, countercurrent.

B. M. VENABLES.

Heat-exchange device. G. FLINTERMANN (U.S.P. 1,736,906, 26.11.29. Appl., 26.7.27).—A heat exchanger is built up from a number of plates stamped with cup-like projections tapering slightly so that when the cups are nested the plates are spaced apart. The bottom of every cup is perforated with a large central hole and a ring of smaller ones. Through the central holes a tube is threaded tightly, or it may be made from a number of nesting sections. More than two fluids may pass through the apparatus.

B. M. VENABLES.

Prevention of scale deposits in boilers. A. HEINRICH (B.P. 306,145, 14.2.29. Ger., 17.2.28).—A continual equalisation is maintained, through the boiler wall, of the electrical *P.D.* between the earth and the atmosphere. To this end the boiler is earthed at one or more points and connected at one or more points either to several metallic points projecting into the air, or to an antenna preferably arranged parallel to any existing power wires in the neighbourhood.

B. M. VENABLES.

Dehydrator. J. M. YOUNGER (U.S.P. 1,737,068, 26.11.29. Appl., 3.12.27).—The material, *e.g.*, vegetables, is stacked on trays and conveyed on trucks longitudinally through the dryer. It is first subjected to a sweating process in steam-laden air, and then passes through a number of separate drying compartments in each of which a transverse, continuous, and reversible circulation of nearly saturated heated air is maintained by means of fans and louvres at the sides. There is a minor addition of fresh air from conduits beneath the floor.

B. M. VENABLES.

Dehydrator. F. C. CHAPMAN, ASSR. to CHAPMAN DEHYDRATER Co. (U.S.P. 1,737,533, 26.11.29. Appl., 2.7.28).—The apparatus, specially suitable for materials, such as cotton, through which the drying air has to be forced rather than passed over, comprises a tunnel with

side inlet for air (from a fan) near the middle of its length. The material is contained in two longitudinal rows of perforated baskets on trucks and makes substantially air-tight connexion with the floor and roof, but the rows are spaced apart from each other and from the side walls. The trucks are progressed intermittently and are caused to skip across the transverse air inlet. Bulkheads are provided at the leading ends of the central passage and at the trailing ends of the side passages, so that in the first half of the dryer the flow of air is in through the central passage, across the containers, and out through the side passages, whereas in the second half the flow is *vice versa* and consequently in the opposite longitudinal direction. Both streams of exhaust air are mixed with fresh, heated air and taken back to the fan.

B. M. VENABLES.

Dryer. N. PANZIREV (U.S.P. 1,736,980, 26.11.29. Appl., 5.12.28. Ger., 21.3.28).—A number of horizontal conveyers are arranged over each other in a casing, and both runs are operative so that the material takes a downward zig-zag course. On every storey there are two conveyers with spaces both at the centre and sides. The drying gases are admitted through a longitudinal passage at the bottom, pass up the central spaces, divide under a twin-arched roof, and pass down the sides to a pair of longitudinal outlets at the bottom.

B. M. VENABLES.

Attrition mill. E. M. BRENNAN and A. P. DANIEL, Assrs. to BAUER BROS. Co. (U.S.P. 1,737,183, 26.11.29. Appl., 8.3.28).—A disc-grinder or similar type of mill is finely adjusted by means of a differential screw.

B. M. VENABLES.

Shaft packings for grinding mills. HARTSTOFF-METALL A.-G. (HAMETAG), and E. KRAMER (B.P. 323,632, 24.12.28).—A twin bearing for a grinding mill of the dry, high-speed, or other type is arranged in a bearing block that makes an oil- and gas-tight joint with the casing of the mill and forms three chambers, viz., between the outer bearing and the driving means, between the two bearings, and between the inner bearing and the casing of the mill; a large sump chamber is also incorporated. The middle chamber is maintained full of oil by a pump, and oil throwers are situated in the other two, that in the inner one forming an oil screen by centrifugal force, in which any dust escaping from the mill is entrained and run to the sump where it settles and whence the oil is pumped for re-use.

B. M. VENABLES.

Disintegrator or pulveriser and separator for solid products. ATELIERS RÉUNIS, and F. HIPPERT (B.P. 323,472, 29.9.28).—The powdered material is carried away from a disintegrator in a primary air current produced with or without the aid of a fan, and is caused to enter in the form of thin sheets a secondary air current, whence the too-coarse material drops out and is returned to the disintegrator. [Stat. ref.]

B. M. VENABLES.

Mixing or separating apparatus. ANEMOSTAT, LTD., and L. M. HIRCHBERG (B.P. 323,544, 5.10.28).—Various forms of apparatus are described for mixing or scrubbing gases, for mixing solids while suspended in gases, etc. They consist of a straight or T conduit with distributing devices as described in B.P. 283,258, which

cause alternate expansions and contractions of the gas streams. B. M. VENABLES.

Separating apparatus for [wet] treatment of ores, coals, etc. L. HOYOIS (B.P. 303,810, 9.1.29. Belg., 9.1.28).—The apparatus is of the upward-current type; precautions are taken to make the current uniform over the whole section, and the downflowing feed enters in an annular manner.

B. M. VENABLES.

Centrifugal separators. AKTIEB. SEPARATOR (B.P. 299,422, 16.10.28. Swed., 26.10.27).—A bowl-type separator with discs is provided with three discharges, viz., two at the top for the lighter and heavier liquids, and a third downwards and inwards from a point near the bottom of the wall of the bowl for discharge of solids with the necessary quantity of liquid. The wall of the bowl may be conical to assist the travel of solids downwards, and the outlets are preferably in the form of easily changeable jets. [Stat. ref.] B. M. VENABLES.

Safety device for centrifugal machines. H. J. M. C. KRANTZ (U.S.P. 1,736,975, 26.11.29. Appl., 25.10.26).—A device is described for preventing the cover of a centrifuge from being opened unless the brake is on and the rotor at rest or nearly so. The motor switch, brake, and lock are operated by one lever. B. M. VENABLES.

Centrifugal machine. A. U. AYRES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,737,137, 26.11.29. Appl., 15.2.28).—A combined drag bearing and inlet is described for a super-centrifuge. B. M. VENABLES.

Filtering apparatus. J. B. VERNAY (B.P. 314,431, 21.6.29. Fr. 27.6.28. Addn. to B.P. 297,394; B., 1929, 459).—The double-storey apparatus described in the prior patent is provided with a vessel for repulping between the stages of filtration. B. M. VENABLES.

Fractional-distillation apparatus. R. B. CHILLAS, JUN., Assr. to ATLANTIC REFINING Co. (U.S.P. 1,736,845, 26.11.29. Appl., 25.2.26).—Bubbling-caps with downflow pipes are made interchangeable with plain caps so that the downflow of liquor may be distributed as desired; they may be suitable for either round or long, narrow apertures in the plates, and the central down-pipe extends right through the top and has a radial or transverse passage to admit the liquor from a suitable depth on the tray. B. M. VENABLES.

Concentration of liquids at very low temperatures. P. C. LEMALE (Addn. 32,681, 9.6.26, to F.P. 609,295; B., 1927, 241).—The two stages of the vacuum are produced by means of a water ejector which is coupled directly with the inside of the evaporator and, through a tubular apparatus provided with a throttle valve, with a heater for the evaporator. A. R. POWELL.

Crystallising vessel. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 457,731, 15.12.25).—The vessel is provided with an internal cooling coil below which is a compressed-air pipe having a number of air outlets closed by rubber lip valves so that air can be blown through the liquid to assist cooling and crystallisation. A. R. POWELL.

Apparatus for aerating and blending liquids. J. W. BATTERSBY (B.P. 323,242, 28.6.28).—The larger quantity of liquid (e.g., tap water) is caused to flow through a jet within a conduit, beyond the jet are holes

for admission of air which is drawn in automatically and is well diffused, and further on still the smaller quantity of liquid (*e.g.*, flavouring) is admitted at a controlled rate. B. M. VENABLES.

Feeding of liquids under predetermined conditions of speed and/or pressure. C. H. LANDER and T. F. HURLEY (B.P. 323,481, 28.9.28).—An apparatus for delivering definite quantities of a liquid such as fuel oil at a definite rate by means of displacement by a heavier liquid such as mercury, which is supplied through a fine jet under a constant head, comprises three bulbs arranged in a closed circuit with capillary tubes on both sides of at least one of the bulbs, which is calibrated. Besides the closed circuit for lighter fluid there are an inlet jet for the heavier liquid in the highest bulb of all, an outlet for lighter liquid from the middle bulb, and connexions (with stopcocks) for filling and refilling. If the apparatus is inverted a lighter liquid may be used as the displacer. B. M. VENABLES.

Method and apparatus for making emulsions and mixtures, applicable also for other purposes. V. G. WALSH and V. A. COLLINS (B.P. 323,534, 4.10.28).—A mixture which is to be converted into an emulsion, or into a substance of which the viscosity or surface tension is to be changed, is subjected in a closed vessel to a number of impulses from a piston, the main purpose of which is to produce pressure oscillations; at the ends of its strokes, however, it serves also as a pump to draw in fresh material and to force out emulsion through a small aperture. The motion of the piston may be suddenly accelerated when near the end of its in-stroke. Other applications indicated are: altering the viscosity of glucose, lowering the surface tension of castor oil, disintegrating molecular aggregates of tans, milk, etc. B. M. VENABLES.

Mechanical production of froth. E. SCHRÖDER and J. A. S. VAN DEURS (B.P. 306,550, 21.2.29).—A gas and a froth-forming liquid are driven together, under a pressure at least 0.5 atm. above atmospheric, against stationary wires or rotary whipping apparatus. In one form of apparatus the mixture is passed through a vessel containing a number of transverse baffles which are perforated alternately near the centre and near the circumference, and between which beaters rotate. Means may also be provided to hinder the rotation of the whole of the mixture. B. M. VENABLES.

Liquid and gas contact apparatus. BARTLETT HAYWARD Co., Assecs. of F. H. WAGNER (B.P. 307,453, 21.11.28. U.S., 8.3.28).—A tower is provided with perforated trays and the gas is forced up the tower with sufficient force substantially to prevent flow of liquid through the perforations; downflow of the liquid is effected by siphons which maintain a predetermined level on the trays and deliver the liquid on to a series of discs rotated at centrifugal speed in, or just below, apertures which are left in the centres of the trays. Additional fixed and rotating beaters and fixed wire screens may be provided above the trays and gutters around their edges. B. M. VENABLES.

Apparatus for using air or gases as a conveying medium. BRIT. "REMA" MANUF. Co. LTD., P. HOWDEN, and L. KAHLER (B.P. 323,061, 8.10.28).—The con-

duits are divided by longitudinal partitions into a number of separate conduits each provided with a valve at the inlet. When the load is reduced, valves are closed in sufficient number to maintain the velocity in the conduits remaining in use. B. M. VENABLES.

Gas-purification apparatus. N.V. MACHINERIE EN APPARATEN FABR. (F.P. 633,268, 26.1.28. Ger., 23.4.26).—To remove suspended solids or liquids from gases, the mixture is passed through a series of chambers provided with staggered channels the arms of which are at an angle of 50–70° to one another and their distance apart is half their length. A. R. POWELL.

Removal of dust from gases with moving filters. DEUTS. LUFTFILTER-BAUGES. M.B.H., Assecs. of A. SCHREMPF (G.P. 457,430, 27.7.24).—The gas is passed through a series of continuously or intermittently moving filters separated by stationary walls and provided with a central opening in the axis of the apparatus for renewing the filtering medium. A. R. POWELL.

Separation of organic gases and vapours [from air]. I. G. FARBENIND. A.-G., Assecs. of A. ENGELHARDT (G.P. 455,175, 11.1.21. Addn. to G.P. 310,092; B., 1921, 204 A).—The constituents absorbed by activated carbon from the gas mixture are removed from the carbon by superheated steam or the superheated vapour of organic solvents and the carbon is then dried and cooled by a current of flue gas. A. R. POWELL.

Gas washer with rotating centrifugal tubes. W. FELD & Co. G.M.B.H. (G.P. 457,568, 12.3.27).—The apparatus comprises a closed, vertical, cylindrical vessel containing a number of flat bowls one above the other, into each of which dip a series of funnel-shaped concentric tubes carried by a central rotating axle so that the liquid is forced up the tubes by centrifugal force and is sprayed into the next higher bowl in the form of a fine mist. The shape or position of the individual funnels is so arranged that the same volume of liquid passes up the annular space between every pair to ensure an even distribution of liquid throughout the chamber and consequent efficient washing of the gas. A. R. POWELL.

Production of a dry mixture of chemicals suitable for preparing fire foam. K. SCHMIDT, Assr. to PYRENE-MINIMAX CORP. (U.S.P. 1,739,094, 10.12.29. Appl., 18.4.27. Ger., 1.9.26).—Aluminium sulphate, sodium bicarbonate, saponin, and sodium carbonate are dried separately at 60°, then mixed, and ground pumice is added. H. ROYAL-DAWSON.

Machine for testing strength and elasticity of material. GOODYEAR TIRE & RUBBER Co., Assecs. of M. L. KOCHHEISER and S. A. STEERE (B.P. 306,555, 22.2.29. U.S., 23.2.28).—A pair of cords, standard and sample, are stretched equally and simultaneously by means of a screw-operated stretcher. B. M. VENABLES.

Boiler for evaporation or heating of liquids. A. L. HITCHCOCK-SPENCER (U.S.P. 1,743,918, 14.1.30. Appl., 27.3.29. U.K., 23.3.28).—See B.P. 319,380; B., 1929, 963.

Edge filtration. H. S. HELE-SHAW and J. A. PICKARD (U.S.P. 1,744,510, 21.1.30. Appl., 20.11.25. U.K., 17.12.24).—See B.P. 249,202; B., 1926, 423.

[Baffles for flues of boiler] furnaces. Y. HAYASHIDA (B.P. 323,562, 9.10.28).

Combustion product power plant. A.-G. BROWN, BOVERI & Co. (B.P. 292,150, 15.6.28. Ger., 15.6.27).

[Arrangement of burners in boiler] furnaces for burning powdered fuel. CLARKE, CHAPMAN & Co., LTD., and W. A. WOODSON (B.P. 323,484, 1.10.28).

Refrigerating machines. L. SZILARD (B.P. 299,783, 29.10.28. Ger., 31.10.27).

Absorption refrigerating apparatus applicable to ice-making. E. R. MITFORD and L. CRUMP (B.P. 323,815, 13.10.28).

Liquid-distributing devices for gas absorption and like apparatus, particularly for refrigerating. ELECTROLUX, LTD., ASSECS. OF PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAGET (B.P. 303,521, 5.1.29. Appl., 6.1.28).

Spraying apparatus. G. ELLIOT (B.P. 324,117, 1.11.28).

[Asbestos-metal] packing material [for cylinder blocks of internal-combustion engines] and its manufacture. H. REINZ DICHTUNGS-FABR. (B.P. 323,900, 18.12.28. Ger., 3.10.28).

Capsule packing. I. G. FARBERIND. A.-G. (B.P. 306,114, 8.10.26. Ger., 17.2.28).

Drying of materials (B.P. 297,480).—See IX. Gas purification (G.P. 457,888). Automatic titration apparatus (U.S.P. 1,739,230).—See XI. Distillation column (F.P. 632,450).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coal rich in spores. Winter seam, Nostell Colliery, Yorkshire. R. G. H. BODDY (Fuel, 1930, 9, 38—39).—Microscopical examination of this seam, which at Nostell Colliery has a thickness of 2 ft., shows it to consist largely (50—60%) of plant residues, e.g., macerated spores, thick-walled cuticle, and fragments of sporangium wall, which are of the usual type found in durains. The microspores, however, are present mainly in aggregations of crushed spores, quite free from extraneous material.

A. B. MANNING.

Microscopical study of some coals from Szechuan, South-West China. C. Y. HSIEH (Fuel, 1930, 9, 4—19).—Nineteen samples of coal from Szechuan have been examined microscopically by reflected light from a polished surface, after maceration in Schulze's reagent, and also in thin sections by transmitted light when it was desired to show some special structures. The distribution of the constituents, durain, vitrain, and fusain, the mode of occurrence of the ash, and the character of the plant remains observed in each sample are described in detail.

A. B. MANNING.

Coal cleaning, with special reference to Japanese coals. R. LESSING (Fuel, 1930, 9, 20—29).—Nine Japanese coals have been cleaned by a process involving first the removal of the dust and then the separation of the remainder with the aid of solutions of calcium chloride of the requisite density. All samples were subdivided into five fractions of increasing sp. gr., and

the proximate composition of each fraction was determined. All the coals yielded a fraction (70—80%) containing 3—6% of ash. Carbonising tests at 900° in the Lessing assay apparatus were carried out on all fractions. The coals, with one exception, showed caking properties of gradually descending order as the sp. gr. increased to 1.60; fractions of sp. gr. greater than 1.60 were practically non-caking.

A. B. MANNING.

Fuels. E. STANSFIELD, W. A. LANG, J. W. SUTHERLAND, K. C. GILBERT, and S. ZEAVIN (Ninth Ann. Rept. Sci. Ind. Res. Council of Alberta, 1928, 15—19).—The slacking of coals on exposure to the atmosphere has been studied under standard conditions; it is found that bituminous coals lose 1—5% by wt. as fines, but that the losses with sub-bituminous coals are somewhat greater, and with domestic coals are from 40 to 100%. Attention has been paid to a number of binders in briquette making, and it is found that phosphoric acid is not suitable for this purpose. Ash fusibility has been investigated by using a much larger number of cones than is generally taken, and heating these individually to temperatures at 25° intervals; the characteristics of different ashes are emphasised by this method.

R. H. GRIFFITH.

Determination of moisture in coal. T. SOMIYA and S. HIRANO (J. Soc. Chem. Ind. Japan, 1929, 32, 247—248 B).—The loss in weight of a coal sample heated at 105—110° was determined continuously (1) in still air, (2) in currents of air and of commercial and pure nitrogen and carbon dioxide. The curves show the oxidising effect of oxygen even when present only to the extent of 2.5% as in "commercial" nitrogen, and also absorption of carbon dioxide when this latter gas was used. A reliable result is only obtainable with pure nitrogen.

C. IRWIN.

Removal of the ash constituents of coal by density separation. B. MÖRY and I. PÉTER (Szénkis. Közl., 1928 [1927], 2, 113—125; Chem. Zentr., 1929, ii, 815).—Fractional separation was effected by centrifuging the powdered mineral (particles <0.094 mm.) in zinc chloride solution, carbon tetrachloride, and benzene. The ash of lignite is chiefly embedded between the layers.

A. A. ELDRIDGE.

Structure of the ash of coal. B. MÖRY (Szénkis. Közl., 1928 [1927], 2, 107—112; Chem. Zentr., 1929, ii, 815).—A photomicrographic and X-ray study of Hungarian coals.

A. A. ELDRIDGE.

Evaluation of gas coal. H. J. A. DE GOEY and G. A. BRENDER À BRANDIS (Gas- u. Wasserfach, 1929, 72, 1237—1242).—The Geipert method (B., 1926, 938) of determining the gas yield of a coal, using a vertical retort, gives results much closer to actual practice than the determination of volatile matter, using a crucible; there is no constant relationship between the two. The "value" of a coal is the product of the gas yield by the Geipert method and the calorific value of the gas produced. The "specific value" is the value per unit of volatile matter as found by the crucible. These views are illustrated by analytical data for a number of English and German coals. The specific value increases with the age of the coal from cellulose to anthracite or with

reduction in oxygen content. The effect of the oxygen content on gasification is discussed in detail and the conclusion is drawn that the true "value" of a gas coal reaches a maximum with 28% of volatile matter (Geipert method), and then falls to a minimum at 36%. The maximum point is approximately that of maximum methane yield.

C. IRWIN.

Low-temperature treatment of Brazilian coals. F. W. FREISE (Petroleum, 1930, 26, 113—114).—Brazil possesses in the three southern States of Paraná, S. Catharina, and Rio Grande do Sul coal of the lowest Permian formation in five strata, and eight mines are in operation. The evaluation of the raw material by means of low-temperature treatment was investigated, and the yields of semi-coke, gas, and tar are given. In one case washing of the semi-coke gave a product poor in ash which can be compounded with 4% of pitch from the tar distillation to make briquettes containing only 4—7% of ash. Tertiary lignite from the regions of Fonseca and Gandarella has a mean content of 45% of water and volatile constituents, 18% of ash, and 37% of solid carbon. Low-temperature treatment carried out at 450° in a revolving furnace produced sandy coke, tar, and gases of an inferior quality.

W. S. E. CLARKE.

Coke and charcoal. I. Structure of coke and charcoal. II. Nature of carbon composing coke and charcoal. III. Reactivity of carbon materials. Y. OSHIMA and Y. FUKUDA (J. Fac. Eng., Tokyo Imp. Univ., 1929, 18, 125—193).—I. Methods for determining the total, open-pore, and closed-pore porosities are discussed, and a new method for determining the open-pore porosity by measurement of the air contained in the open pores is described. The true sp. gr. was obtained by the pycnometer method, using 250-mesh coke and ether. It was found that the amounts of closed pores in most cokes were small (2—5%), and that the volume of open pores was usually 40—70%. Microscopical examination of wood charcoal indicated that the distribution of its pores was similar to that of the original wood; reasons for the pronounced differences between the microstructures of charcoal and coke are advanced.

II. Finely-ground samples of artificial graphite, retort carbons, oven coke, gas coke, semi-coke, charcoals, pitch carbon, petroleum coke, and active carbon were examined by an X-ray method; all the samples gave indications of the presence of graphite, the amount decreasing as the temperature of carbonisation fell. It is concluded that coke and charcoal consist principally of minute particles of graphite contaminated by and cemented together with hydrocarbon complexes. The size and number of these minute carbon (graphite) crystals increase with rising carbonisation temperature; the nature of coke and charcoal carbon is therefore nearly identical, provided that their carbonisation is effected at the same temperature. Examination of samples of coke taken from various parts of an oven, by the X-ray method, showed that "bottom" coke was in a more developed stage of carbonisation than "top" coke, and that the degree of carbonisation progressively decreased from the centre of the charge to the oven wall.

III. The reactivities of a number of cokes and charcoals have been examined by their gasification, in carbon dioxide, at various temperatures; the amount of carbon dioxide decomposed was determined by a gas interferometer method in which the composition of the gas was obtained from an examination of its refractive index. The conclusions arrived at are the following:—(1) The initial temperature at which coke and charcoal begin to react with carbon dioxide is dependent solely on the maximum temperature of carbonisation at which the sample is produced, and hence on the state of its carbon (as described above in II). (2) Reactivity is also governed by the nature of the reacting surface; hence the reactivity of a coke, in general, is independent of its initial temperature of reaction with carbon dioxide. (3) Reactivity may be affected during the course of an experiment by (a) activation by the reaction itself, due to surface etching and development of microstructure, and (b) deactivation by thermal effects in which the surface atoms tend to arrange themselves in the more stable positions of the graphite lattice, *i.e.*, graphitisation. (4) The surface of high-temperature coke is not as reactive as its inner portion, due to the deposition of stable films from the hydrocarbon vapours evolved during carbonisation; semi-cokes do not exhibit this difference, because their temperature of carbonisation does not effect decomposition of hydrocarbon vapours. (5) The greater reactivity of charcoal is due to its microstructure, which consists of a large amount of surface per unit volume, and to the large number of unstable carbon atoms on this surface. (6) The relationship between the effective surface area of a coke and the rate of flow of carbon dioxide necessary to give a definite amount of carbon monoxide in the reaction products can be expressed in the form of an equation; this can be confirmed experimentally, and indicates that an absolute measure of reactivity can be obtained in terms of effective reacting surface.

C. B. MARSON.

Coke and charcoal. IV. Form of carbon composing coke and charcoal. Y. OSHIMA and Y. FUKUDA (J. Soc. Chem. Ind., Japan, 1929, 32, 208—210 B).—Various kinds of coke and charcoal were investigated by X-ray methods. Retort carbon alone gave the sharp graphite spectrum; the other varieties of carbon gave graphite spectra having one or more broadened bands. It is concluded that coke and charcoal consist principally of graphite in an extremely fine state of subdivision, but are contaminated to a greater or smaller extent with complex polymerised hydrocarbons. The size and number of these minute crystals of graphite increase with rising carbonisation temperature and corresponding decreasing hydrocarbon content. The results obtained support the non-existence of amorphous graphite. By X-ray examination it was found that the coke produced in the lower part of a Kuroda coke oven is more highly carbonised than that in the upper part, and that in each part of the oven the degree of carbonisation is the higher the nearer is the wall of the oven. It is confirmed that the symmetry of graphite is orthohexagonal.

S. K. TWEEDY.

Coke and charcoal. V. Reactivity of graphite. Y. OSHIMA and Y. FUKUDA (J. Soc. Chem. Ind. Japan,

1929, 32, 226—227 B).—The reactivity of artificial graphite to carbon dioxide at 1000° was investigated. Two surface changes occur: (a) the formation of an "etched" surface of highly unsaturated atoms (comparable with Taylor's structure for the surface of a nickel catalyst) tending to raise the reactivity of the graphite; (b) a thermal transformation of the most highly unsaturated surface carbon atoms into a more saturated condition, tending to lower the reactivity. The diminished reactivity of coke is probably due to the deposition of carbon from hydrocarbon vapours on the reactive surface, with consequent destruction of the micro-structure, during coke formation. S. K. TWEEDY.

Coke and charcoal. VI. Reactivity of coke. Y. OSHIMA and Y. FUKUDA (J. Soc. Chem. Ind. Japan, 1929, 32, 251—252 B).—As coke is composed essentially of minute particles of graphite crystal, the effective surface area (A) of a coke sample can be determined by comparing the rates of flow (V , V') of a current of carbon dioxide which the sample will reduce to the extent of 50% to carbon monoxide with that of a current similarly reduced by a graphite rod of area (A') under similar conditions; then $A/A' = V/V'$. The specific effective surface area is this value divided by the weight. A number of such determinations on coke samples are given, and it is claimed that the specific effective surface area is a measure of the reactivity. After the tests the samples showed blackening at the surface, the extent of the blackening being the greater the more reactive was the sample. C. IRWIN.

Temperature changes, heat flow, and heat capacity of coke-oven walls. M. STEINSCHLÄGER (Arch. Eisenhüttenw., 1929—1930, 3, 331—338; Stahl u. Eisen, 1929, 49, 1797).—The thermal conditions in the walls of a coke oven during charging and discharging have been investigated; the results demonstrate the greater heat conductivity of silica brick compared with firebrick and the necessity for rapid charging and discharging in economising heat. A. R. POWELL.

Combustion, wind, and flue equipment. C. A. MASTERMAN (Gas J., 1930, 189, 154—156, 204—208).—Factors of which a knowledge is necessary for the design of correct flue equipment are discussed. Expressions are given for determining, in a flue of given area, the critical height at which the products of combustion from coal gas, coal, coke, or oil will be completely removed and sufficient draught provided to meet normal combustion requirements. C. B. MARSON.

Liquid purification [of gas] by soda ash solution. W. J. G. DAVEY (Gas J., 1930, 189, 157—159).—An account of investigations into the Seaboard process of hydrogen sulphide and cyanogen removal by washing the foul gas with a 3% solution of soda ash (cf. Sperr, B., 1922, 359 A; Cook, B., 1926, 35). Equations are given for the reactions occurring during purification of the gas and activation of the foul liquor by an air-blast. The purification reactions are not so simple as those stated by Sperr, which indicate an efficiency in excess of that actually obtainable; it is suggested that the absorption of hydrogen sulphide might be speeded up by replacement of part of the soda ash by sodium hydroxide. The final product from the removal of

cyanogen is sodium thiocyanate, and profitable recovery of this compound would decrease the cost of the process, and also eliminate a noxious constituent from the ammonia plant effluent; the commercial possibilities of its recovery as the insoluble copper salt are discussed. The foul gases from the actifier may either be burned under boilers or rendered innocuous by passing them through an oxide purifier. D. G. MURDOCH.

Sulphur production and gas purification. T. P. L. PETIT (Chem. Weekblad, 1929, 26, 542—547). J. H. KOERS (*Ibid.*, 599). T. P. L. PETIT (*Ibid.*, 599).—For removing hydrogen sulphide, coal or coke-oven gas is scrubbed by means of concentrated potassium carbonate solution, from which the hydrogen sulphide is driven out again by carbon dioxide and passed through iron oxide. The oxide after repeated regeneration and re-use is treated with solvents for recovery of pure sulphur. The potassium carbonate solution is heated to break up the bicarbonate formed, the carbon dioxide being thus recovered for re-use. Figures for operating cost and some theoretical considerations on the equilibria involved are given. A polemical note by KOERS is followed by a reply by PETIT. S. I. LEVY.

Desulphurisation of gas with Köflach lignite charcoal. F. NEUWIRTH (Berg- u. Hüttenmänn. Jahrb., 1928, 76, 1—13; Chem. Zentr., 1929, ii, 514).—The use and regeneration of the charcoal are described. A. A. ELDRIDGE.

Determination of calorific values of producer gas. H. LÖFFLER (Chem.-Ztg., 1930, 54, 51—52).—A note on the direct-reading Strache-Löffler calorimeter. S. I. LEVY.

Natural gas. E. H. BOOMER (Ninth Ann. Rept. Sci. Ind. Res. Council of Alberta, 1928, 51—53).—Gas from the Viking field, containing 93% CH_4 , 3.5% C_2H_6 , and 3% N, has been used as a raw material for thermal decomposition into carbon-black or to low-boiling hydrocarbons. Direct oxidation processes are found to be of little value, but conversion into water-gas has been achieved. The use of the gas for high-pressure synthesis is considered, and attention is being paid to the removal of sulphur, which is present in large amount. R. H. GRIFFITH.

Microscopical researches on coal-tar solutions. F. J. NELLENSTEYN (Gas World, 1930, 92, 54. Cf. B., 1928, 324; 1929, 231).—A method of determining the "micron number" of tar is described. The tar (0.2—0.3 g.) is dissolved in nitrobenzene to give a 1% solution (by vol.), which is heated, thoroughly mixed, allowed to cool to room temperature, and then filtered through a specified filter paper. The microns in the filtrate are counted in a Thoma haemocytometer, at a magnification of 400; particles consisting of adhering smaller particles are counted as a single body. The method demands exact standardisation, mainly in respect of the filter paper and of the time interval between the preparation and filtration of the solution. Solvents other than nitrobenzene, having a surface tension of at least 40 dynes/cm. at 20°, e.g., quinoline, furfuraldehyde, may be used, but different micron numbers will then be obtained. The micron number, which may vary between zero and 6×10^7 per mm^3 of tar, assists in

evaluating the tar with respect to its binding power, and depends on the origin of the coal, the temperature of carbonisation, and the distillation of the tar, the better tars generally showing the higher numbers. The test may be regarded as complementary to the ordinary "free carbon" test, which is an approximate measure of the micelle content adequate for practical purposes. A more exact value is given by the difference between the values obtained for the precipitates with a liquid of low surface tension, *e.g.*, ether, and one of high surface tension, *e.g.*, nitrobenzene, this difference being the sum of the micronic and ultramicronic micelles.

D. G. MURDOCH.

Russian asphalt. E. GRAEFE (Petroleum, 1930, 26, 101—107).—Petroleum bitumen is produced principally in Baku from Binagadi crude oil, the characteristics of which are tabulated. The oil is blown in a still (capacity 80 tons) at 220—270° with a mixture of steam and air for 24 hrs. External heating is unnecessary as the reaction is exothermic. The yield amounts to 90%, calculated on the residue, and the asphalt yield is 25—30%, calculated on the crude oil; the latter value is reduced to 15% when straight-run asphalt is blown with steam only. When the asphalt has attained the desired degree of hardness it is passed under pressure from the still to the reservoir, and thence is led to the conveyer. The monthly output of asphalt so produced is 3000 tons. Three methods of obtaining bitumen from the residue from the acid tars from petroleum distillates are described. The acid is removed by washing with hot water and the bitumen compounded with lime and used to produce "Mastix"; or the unneutralised tar is decomposed with hot water (85—95°) and mixed with air. After keeping for several hours, the separated acid is removed, the operation repeated twice with the residue, and the product so obtained blown at 110—220° with steam and air. Solvent naphtha can also be used as a means of separating the acid. The bitumen dissolves in this solvent, which can be recovered by distillation, and sulphuric and sulphonic acids remain undissolved. Asphalts so produced are used for electrotechnical purposes, cables, etc., and as road-making material. W. S. E. CLARKE.

Extraction apparatus for bituminous road materials. F. J. NELLESTEYN (Chem. Weekblad, 1930, 27, 19).—The ordinary Soxhlet apparatus is modified by elimination of the siphon tube, so that the solvent runs through continuously. To allow liquid to accumulate in the body of the extractor, if desired, a cock is provided in the connecting tube.

S. I. LEVY.

Action of aluminium chloride on hexahydroaromatic and saturated aliphatic hydrocarbons. R. STRATFORD (Ann. Office Nat. Combust. liq., 1929, 4, 83—108, 317—359; Chem. Zentr., 1929, ii, 1287).—On treatment with aluminium chloride, ethyl-, propyl-, and butyl- or *m*-diethyl-*cyclohexane* afforded, respectively, di-, tri-, and tetra-methyl-*cyclohexane*; *o*- and *p*-dimethyl- and ethyl-*cyclohexane* afforded *m*-dimethyl-*cyclohexane*, whilst the propyl compound afforded 1:3:5-trimethyl-*cyclohexane*. Fission of the side chain is a secondary effect which increases with the length of the chain; diethyl-*cyclohexane* afforded butane, *n*-amyl-

and isoamyl-*cyclohexane* gave different yields of methyl- and tetramethyl-*cyclohexane*, the *iso*-compound also apparently producing the pentamethyl compound. Normal aliphatic hydrocarbons break between the fourth and fifth carbon atoms, giving butane; a chain containing more than eight carbon atoms breaks at both ends, forming butane (2 mols.) and an unsaturated residue. Hydrocarbons containing secondary groups are subject to fission and to transposition of the radicals. The cracking process is attributed to re-arrangement rather than to fission between the nucleus and side chain. Normal aliphatic chains up to $C_{17}H_{35}$ and cyclic hydrocarbons with a side chain of C_6H_{11} or C_5H_{11} did not afford fractions corresponding with those of light petroleum, which were formed only with compounds containing secondary groups, *e.g.*, isobutyl and isoamyl. The fission, but not the rearrangement, depends on the amount (5—100%) of aluminium chloride employed; in the cracking of petroleum, using 10—15% of aluminium chloride, both reactions probably take place. Hydrogenation of aromatic hydrocarbons to *cyclohexane* derivatives by Voorhees and Adams' method was satisfactory unless the side chain contained more than four carbon atoms, when some formation of methyl-*cyclohexane* occurred. Dehydrogenation by Zelinski's method was employed in a study of the structure of certain cyclic hydrocarbons. Values for the m.p. or b.p., d , and n_D of a number of hydrocarbons are recorded.

A. A. ELDRIDGE.

Thermal properties of petroleum products. C. S. CRAGOE (U.S. Bur. Stand. Misc. Publ. No. 97, 1929, 48 pp.).—Various thermal properties of petroleum products, viz., thermal expansion, heats of combustion, sp. vol. of vapour, thermal conductivity, sp. heat, latent heat of vaporisation, and heat content, are given in numerous tables prepared in forms convenient for use in engineering. These tables embody the results of a critical study of the data in the literature, together with hitherto unpublished data. The experimental basis for each table, and the agreement of the tabulated values with experimental results, are given, as well as empirical equations for their derivation. Each table is accompanied by a statement regarding the estimated accuracy of the data and by a practical example of the use of the data.

H. S. GARLICK.

Use of organic solvents for extraction of high-sulphur oils from crude petroleum. G. E. WOODWARD (Ind. Eng. Chem., 1929, 21, 1233—1235).—The miscibility of 61 organic solvents with Inglewood crude petroleum has been investigated qualitatively, and in many cases has actually been determined, together with the relative amounts of sulphur extracted by them. Furfuraldehyde, absolute ethyl alcohol, and acetaldehyde showed the highest concentration of sulphur.

H. S. GARLICK.

Decomposition of paraffin wax on heating. II. H. I. WATERMAN, J. N. J. PERQUIN, and H. A. VAN WESTEN (J. Inst. Petroleum Tech., 1930, 16, 29—33; cf. B., 1928, 512).—A Rangoon paraffin wax, when distilled at 360° and 385° in an apparatus previously described (*loc. cit.*), yielded small quantities of hydrogen at both temperatures and large quantities of unsaturated

hydrocarbons at 385°. Heating of 90 g. of wax for 7 hrs. at 385° yielded quantities of unsaturated gaseous, liquid, and solid hydrocarbons equivalent to 12 g. of bromine. Samples (90 g.) of wax were heated at 360° during several periods of 6 hrs., the gases and distillates being expelled from the heating zone and not subjected to further heat-treatment; in the first three periods unsaturated hydrocarbons equivalent to 2.72, 2.46, and 2.16 g. of bromine, respectively, were formed. It was also shown that the bromine value was a true measure of the degree of unsaturation in the unsaturated compounds.

C. B. MARSON.

Detection and determination of lead in "ethyl" petrol. H. KIEMSTEDT (Z. angew. Chem., 1929, 42, 1107–1108).—Lead tetraethyl may be detected in petrol by moistening a filter paper with the liquid, allowing it to dry exposed to bright sunlight, and exposing it to hydrogen sulphide, when a brown to black coloration shows the presence of lead. Lead may be determined in ethyl petrol by treating 100 c.c. of the petrol with 2–3 c.c. of acetyl chloride and 1–2 drops of water; the mixture is well shaken in a stoppered cylinder until the red liquid turns blue and a precipitate of lead chloride separates in white flocks. After 4–5 hrs., when the petrol has become colourless, the precipitate is collected, washed with light petroleum, and converted into sulphate in the usual way.

A. R. POWELL.

Viscosimeter. MATTHIJSEN.—See I. **Electrically heated stills.** DONY.—See XI. **Petrolatum liquidum.** SCHOU and NIELSEN.—See XX. **Carbon monoxide determination.** WIRTH and KÜSTER. **Determination of benzene in air.** SMYTH.—See XXIII.

PATENTS.

Coke ovens. KOPPERS COKE OVEN Co., LTD., Assees. of H. KOPPERS A.-G. (B.P. 298,945, 11.9.28. Ger., 17.10.27).—The ferroconcrete foundation of a coke oven having passages underneath is divided up into slabs between which expansion joints are provided. The slabs rest on pillars and struts by means of iron shoes which allow of sliding.

A. B. MANNING.

Retorts for the production of gases from wood etc. R. M. A. E. CÉZANNE (F.P. 634,399, 30.8.26).—Apparatus is provided above the retort for supplying a regulated blast of air into a reaction chamber situated preferably in the immediate neighbourhood of the outlet of the hearth for the production of the gas.

L. A. COLES.

Coal and the like crushers. E. TAYLOR, and FERRYHILL FOUNDRY & ENG. Co., LTD. (B.P. 322,641, 29.11.28).—Two crushing rollers with their axes horizontal are arranged within a chamber in such a manner that the coal is crushed first between the upper roller and an inclined plate forming part of a delivery passage to the rollers, then between the rollers themselves, and finally between the lower roller and a resiliently supported breaking surface.

A. B. MANNING.

Pulverising installations for pulverised coal and the like. H. G. C. FAIRWEATHER. From C. PETERS (B.P. 322,695, 14.2.29).—The current of air conveying the powdered material from the mill passes

into a re-collector, situated immediately above the mill, wherein the coarser material is separated from that ready for blowing into the furnace and is returned to the mill. The air return pipe from the cyclone separator is led into the mill in such a manner that the separation of the coarse material in the re-collector is enhanced by an injector effect.

A. B. MANNING.

Apparatus for feeding combustible dust into receptacles under pressure by means of valves. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,356, 19.12.28).—In internal-combustion engines using powdered fuel, or other apparatus in which combustible dust is fed from a reservoir through a valve to a chamber wherein the pressure is continually or intermittently higher than that in the reservoir, the latter and/or the dust feed conduits are provided with return or de-aerating conduits, through which, if necessary, the compressed gases can expand either back into the bunker or to the air outside. Provision may be made for supplying a protecting gas, e.g., carbon dioxide or nitrogen, to the feeding system, preferably in such a manner as to agitate the dust and facilitate its flow.

A. B. MANNING.

Production of decolorising charcoal. A. SCHOLZ (G.P. 455,521, 9.10.24).—In the production of decolorising charcoal from lumps of coal of uniform size in externally-heated vertical retorts, the most intensive reaction occurs near the walls of the retort, so that much carbon is lost as carbon monoxide in this region while the reaction is proceeding into the interior of the charge. It is proposed to charge the retort so that the lumps of the material decrease in size zone-wise towards the interior, whereby the loss of carbon is diminished, the quality of the product is rendered uniform, and the yields are substantially increased. The zones may be defined by the insertion of concentric cylinders in the furnace.

S. K. TWEEDY.

Production of decolorising charcoal. A. SCHOLZ (G.P. 457,164, 30.12.24. Addn. to G.P. 455,521; preceding).—The separate zones in the prior process are charged with different varieties and, if desired, with lumps of varying size of crude charcoal. L. A. COLES.

Gasification of earthy raw brown coal. REISCHACH & Co., G.M.B.H. (G.P. 455,464, 16.8.22).—The material is fed into the generator continuously and regularly in thin layers and at the same time heat from an external source, e.g., a highly heated current of air for gasification, is introduced into the drying zone in such amount that for every kg. of coal gasified not more than 40% of moisture need be driven off by the external heat of the coal.

A. B. MANNING.

Treatment of carbonaceous materials prior to destructive hydrogenation. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 322,917, 31.12.28).—The material, e.g., a mixture of powdered coal with a liquid vehicle, is conveyed over the greater part of the distance between the mixer and the converter in the form of a relatively thin suspension, and is passed into a settling vessel from which the thicker and thinner suspensions are drawn off, the former being supplied to the hydrogenation converter while the latter is returned to the mixer. If desired, the thin

suspension may be passed through a heater on its way to the settling tank.

A. B. MANNING.

Distillation and gasification of solid carbonaceous material. L. C. KARRICK (B.P. 322,765, 7.7.28).—Pulverised carbonaceous material, preferably preheated, is introduced into a current of steam under pressure by which it is conveyed through an externally heated tube, wherein it is raised to a sufficiently high temperature to effect distillation, or distillation and gasification, of the material. The tube, which may be of steel, is arranged in the form of a coil and is contained in a vertical, gas-fired furnace. The coke or ash is separated from the issuing gases and vapours, which are then cooled in a heat exchanger without material reduction in pressure. The heat exchanger may be utilised for the generation of steam for use in a second unit working at a lower pressure. In another arrangement the powdered coal, carried in a stream of superheater steam, is gasified completely and the steam and hot products obtained are passed through a vertical retort containing coarser coal, which is thereby distilled. A complete system, comprising distilling and gasifying units for the fine coal and an internally heated vertical retort for distilling the coarser coal, is described in detail.

A. B. MANNING.

Manufacture of hydrocarbons. I. G. FARBERNID. A.-G. (B.P. 296,429, 16.7.28. Ger., 1.9.27).—Coal pastes, mineral oils, tars, etc., containing sulphur compounds or products of an asphaltic nature, are treated with hydrogen, preferably in the presence of catalysts, under such conditions of temperature and pressure that the sulphur and oxygen present in the initial materials are completely or almost completely eliminated without substantial destructive hydrogenation. The resulting products are then subjected to a cracking treatment, without any additional hydrogen, and with or without catalysts.

A. B. MANNING.

Production of rich combustible gas. METALLGES. A.-G. (B.P. 304,760, 24.12.28. Ger., 26.1.28).—Bituminous fuel is first distilled and then gasified. The gasifying agent used in the latter process contains oxygen and such a high proportion of steam that the bulk of the gasified carbon is transformed into carbon dioxide, so that the hydrogen content of the gas exceeds the carbon monoxide content. The gas may be freed from carbon dioxide prior to use.

A. B. MANNING.

Production of high-grade gas resembling illuminating gas. BAMAG-MEGUIN A.-G. (G.P. 458,516, 11.12.24).—Water-gas and distillation gases produced respectively in a generator and in an annexed retort are passed together through a heat accumulator which receives the waste gases from the generator and serves to gasify the distillation vapours and to heat the retort.

L. A. COLES.

Production of water-gas. H. J. C. FORRESTER. From GAS U. TEER GES.M.B.H. (B.P. 323,042, 19.9.28).—Part of the gas produced in the plant is passed through a regenerating chamber, maintained, e.g., at 1200°, and recirculated through the reaction chamber, to which steam and pulverised fuel are supplied. Two regenerating chambers are provided, one of which is used for preheating the circulating gas while the other is being heated by the combustion therein of part of the gas

produced. After leaving the reaction chamber, but before separation of the part to be recirculated, the gas is cooled in a device which utilises its sensible heat for drying the fuel, or generating steam, etc.

A. B. MANNING.

Purification of coke-oven gas and like gases. UNION CHIM. BELGE SOC. ANON. (B.P. 323,351, 14.12.28. Belg., 19.10.28).—The cold gases issuing from the condensing system are scrubbed in two stages: firstly with liquor which has already been used for the second scrubbing and has subsequently been rendered alkaline by the absorption of gaseous ammonia, and secondly with the cold neutral liquor obtained after expelling the acid components and the volatile ammonia from the impure ammoniacal liquors. The liquor from the first scrubber is combined with that condensed directly from the gas and treated in a suitable distilling apparatus for the recovery of pure gaseous ammonia therefrom. A portion of the neutral liquor from the distilling plant is used for the second scrubbing, the remainder being treated with milk of lime in order to decompose the stable ammonium salts.

A. B. MANNING.

Purification and enrichment of gases and vapours derived from the distillation of inferior fuels, for subsequent catalytic conversion into synthetic fuels. SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (F.P. 632,360, 21.7.26).—The gases are freed from sulphur in purifiers which are charged with metal oxides, e.g., nickel oxide, and which are placed between the gas producer and the catalyst chamber. The purifying material is regenerated by passing a current of hot air through it, followed by a current of preheated water-gas. The sulphur is expelled in the form of sulphur dioxide by the air and the water-gas converts the catalyst into a mixture of metal and oxide which is again able to retain the sulphur and in so doing liberates hydrogen; the latter brings about a partial hydrogenation of the gases.

A. B. MANNING.

Recovery of tar from gases. J. PINTSCH A.-G. (G.P. 456,679, 15.7.26).—Solutions of soap or other substances insoluble in tar are added to the gases from the distillation of coal, shale, etc. while the tar vapours are being condensed.

A. B. MANNING.

Waterproof [asphaltum] cement. J. P. STRASSER (U.S.P. 1,738,022, 3.12.29. Appl., 28.6.24).—An adhesive composition containing asphaltum 50%, dextrin 25%, borax 23.45%, and water 1.5%, by wt., is claimed; formaldehyde is added as a preservative.

H. ROYAL-DAWSON.

Destructive hydrogenation of carbonaceous materials. STANDARD OIL DEVELOPMENT Co., ASSECS. of R. T. HASLAM and R. P. RUSSELL (B.P. 304,301, 1.10.28. U.S., 19.1.28).—Oil or other carbonaceous material capable of being hydrogenated is passed into a mixing zone into which hydrogen or a gas containing it is injected, and the admixed oil and gas are passed through a heating zone at a pressure of not less than 50 atm. (preferably at 100 atm. or more). The mixture then passes into a reaction zone where the temperature is above 400°, and to which, if desired, more hydrogen may be added, the product or products of hydrogenation being recovered therefrom. A portion of the charge in

the reaction zone may be continuously withdrawn, passed through the mixing and heating zones, and returned to the reaction zone, either without or with the addition of a temperature-reducing medium. Furthermore, the gas and vapour products from a primary reaction zone can be passed to a secondary reaction zone which is supplied with hydrogen in larger volume and at a higher temperature. Catalytic material may be suspended in the oil in the reaction zone and recirculated through the heating and reaction zones, or may be in a form capable of being supported in the reaction zone.

H. S. GARLICK.

Production of insulating oil from lignite oil and shale oil. A. RIEBECK'SCHE MONTANWERKE A.-G., Assecs. of T. HELLTHALER (G.P. 458,629, 13.1.26).—The oil, after pretreatment, *e.g.*, by blowing with steam and extraction with alcohol, is treated with the tetrachloride of tin or of another element, other than carbon, of group IV, alone or mixed with fuller's earth etc., after which it is refined and treated to remove paraffin wax.

L. A. COLES.

Catalytic decomposition of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,855, 8.11.28).—Catalysts for use in the thermal decomposition of hydrocarbons, alone or mixed with steam, air, etc.; comprise nickel, cobalt, or iron mixed with aluminium compounds which contain oxygen and at least one of the elements silicon, hydrogen, carbon, boron, phosphorus, and sulphur, and which are not decomposed during the reaction (*e.g.*, kaolin), together with, if desired, magnesium or an alkaline-earth oxide.

L. A. COLES.

Refining of hydrocarbons. L. BOURDELLES (F.P. 632,378, 23.7.26).—The hydrocarbons are mixed with excess of steam and passed over finely-divided metals at 350–450°. The metals, *e.g.*, iron, nickel, or copper, mixed with vanadium or tungsten, are such as decompose steam under the experimental conditions; the hydrogen so produced brings about a partial hydrogenation of the hydrocarbons and an elimination of sulphur. The reaction may be carried out under the ordinary pressure. The catalysts are periodically regenerated by reduction.

A. B. MANNING.

Heating and cracking of [petroleum] oils. FOSTER WHEELER, LTD. From FOSTER WHEELER CORP. (B.P. 323,517, 3.7.28).—Oil is raised to distillation temperature by passing it through the high-temperature tubes of an oil-heating furnace with such rapidity that little or no cracking occurs. The vapours pass to a bubble tower and are fractionated, whilst the less volatile residue is subjected to cracking temperature by passing it through the somewhat cooler tubes of the furnace. The cracked vapours pass to a second vapour-separating device from which the reflux returns to meet the heavy ends from the first tower in quantity sufficient to control the temperature attained in, and the time of flow through, the cracking tubes.

H. S. GARLICK.

Treatment of hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,733,654, 29.10.29. Appl., 12.1.20. Renewed 15.2.27).—Oil is passed through a cracking coil to an adjacent vapour chamber inclined from the horizontal. Vapours are

discharged from the lower end to a dephlegmator and reflux condensate is returned directly thereto. Condensate and liquid residue are discharged from the lower end of this chamber for re-treatment in the heating coil, while the heavy residuum is withdrawn from the bottom without returning to the cracking coil. The whole system is maintained under pressure.

H. S. GARLICK.

Rectifying process [for hydrocarbons]. M. P. YOKER (U.S.P. 1,735,558, 12.11.29. Appl., 15.7.24).—A mixture of hydrocarbons is fractionally vaporised by countercurrent contact between liquid and vapour, and a portion of the vapours is simultaneously rectified. A part of the condensate is then introduced at spaced elevations into the stream of downwardly-flowing reflux, and other condensates are removed separately.

H. S. GARLICK.

Apparatus for pressure-distilling [hydrocarbon] oils. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,729,035, 24.9.29. Appl., 21.2.21. Renewed 18.9.26).—Oil is treated in an expansion chamber consisting of a stationary outer shell with a movable closure element operable to permit the introduction or withdrawal of a concentric, removable, cylindrical inner shell.

H. S. GARLICK.

Apparatus for treating [hydrocarbon] oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,733,655, 29.10.29. Appl., 1.9.20. Renewed 15.10.26).—Oil is fed to a rotating drum heated to produce surface distillation, and the vapours are received in an adjacent, similarly heated, and rotating vapour chamber from which a vapour outlet communicates with a condenser.

H. S. GARLICK.

Cracking of hydrocarbons. E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,733,799, 29.10.29. Appl., 11.6.27).—A charge of oil is circulated from a bulk-supply drum through heating tubes, where it is raised to cracking temperature, and back to the bulk-supply drum from which vapours are withdrawn under pressure. Maintained below the normal liquid level in the bulk-supply drum is a quantity of fuller's earth so arranged that part of the oil charge passes through the fuller's earth while the remainder flows through open liquid paths and back to the heating tubes. Pitch-laden oil is withdrawn from, and raw oil supplied to, the pressure drum during distillation.

H. S. GARLICK.

Purification of hydrocarbon oils. STANDARD OIL DEVELOPMENT Co., Assecs. of H. G. M. FISCHER (B.P. 295,728, 18.8.28. U.S., 19.8.27).—Hydrocarbon oils, especially heavy kerosene and naphtha, are sweetened by agitating with 10–50% of the amount of "doctor" (sodium plumbite) solution required in the absence of air-blowing, blowing a strong current of air through the mixture for about 1 hr., adding the major quantity of sulphur in two batches each not exceeding 0.072 lb. per lb. of litharge, and each followed by about 1 hr. air-blowing. Then sulphur is added in relatively small hourly batches, each followed by air-blowing for about 1 hr. until the oil is sweet.

H. S. GARLICK.

Refining of [hydrocarbon] oils. S. TIJNSTRA (B.P. 318,706, 12.7.28).—Sodium plumbite solution is added to hydrocarbon oils or their cracked products, and to

the treated, separated, and water-washed oil is added an aqueous solution of an alkali polysulphide to remove lead and excess sulphur, the temperature of treatment being 27—32°.

H. S. GARLICK.

Apparatus for treating petroleum oils. A. D. DAVID, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,733,651, 29.10.29. Appl., 7.2.23. Renewed 2.6.27).—Arranged within a furnace are a series of inner and outer concentric heating tubes, through the former of which is passed a relatively more easily cracked oil, while a more refractory oil passes through the other. Both heating tubes are supplied from different sources under pressure and are connected at their outlets to receivers for the heated oil.

H. S. GARLICK.

Distillation of petroleum oils. R. W. HANNA, ASSR. to STANDARD OIL Co. of CALIFORNIA (U.S.P. 1,729,215, 24.9.29. Appl., 4.10.23).—Mixed petroleum vapours are passed at a temperature below cracking point through closed, transverse passages contained within a chamber. A portion of the vapours is withdrawn and returned to the chamber, wherein reflux of the heavier fractions is obtained by a cooling means. The reflux is reheated by heat exchange with, but out of contact with, the first petroleum vapours, and the residual reflux is withdrawn from the system.

H. S. GARLICK.

Combustion of liquid fuel. S. SELMA (B.P. 323,601, 16.11.28).—Fuel is led through a feed pipe terminating in a nozzle extending into broken or crumbled refractory material which partially fills a chamber, e.g., a copper pipe, which is traversed by the fuel to be gasified, the remaining portion of the chamber serving for the collection of the gas developed. This is supplied through a conduit to a burner or burners. The whole is disposed in the interior of a hearth so that the flame of the burner contacts with the entire portion of the tube forming the gas chamber and a part of the gasifier. Auxiliary gasifying means consisting of a helical or tortuous fuel passage suitably heated may be provided as a convenient means for initiating gasification and setting the burner in operation, and is capable of being cut off from the main system as soon as the operation of the burner is started.

H. S. GARLICK.

Manufacture of producer gas. H. NIELSEN and B. LAING (U.S.P. 1,743,930, 14.1.30. Appl., 9.8.24. U.K., 17.8.23).—See B.P. 227,880; B., 1925, 197.

Coke extractors for vertical retorts for the carbonisation of coal and like materials. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT Co. (B.P. 323,894, 14.12.28).

Burner for regenerative furnaces. H. BANGERT and G. HÜHN (B.P. 324,157, 16.11.28).

Gas burners. BRIT. FURNACES, LTD., and E. W. SMITH. FROM SURFACE COMBUSTION Co. (B.P. 323,760 9.10.28).

Acetylene generators. D. A. BROWN (B.P. 323,904, 27.12.28).

Retorts (B.P. 323,715). Combustion product power plant (B.P. 292,150). Separation of coal (B.P. 303,810). Distillation apparatus (U.S.P. 1,736,845). Feeding of liquids (B.P. 323,481).—See I.

Petroleum-like hydrocarbons (Addn. F.P. 32,666). Alkyl halides, glycols, etc. (Addn. F.P. 32,638).—See III. Road-construction material (B.P. 309,070).—See IX. Coating metal articles (B.P. 323,847).—See X.

III.—ORGANIC INTERMEDIATES.

Differentiation of isoamyl, isobutyl, and *n*-butyl alcohols, and of the lower alcohols, and of amyl and butyl acetates, by means of ammonium cobaltthiocyanate. H. WEBER (Chem.-Ztg., 1930, 54, 61).—An account is given of the colour reactions between the aqueous solution of the reagent and various organic solvents. In examining mixtures, separation by distillation is employed in conjunction with the colour test.

S. I. LEVY.

Preparation of dianisidine. S. LANGGUTH (Chim. et Ind., 1930, 23, 34—36).—Full experimental details are given for the preparation of dianisidine (3:3'-dimethoxybenzidine) by the following series of reactions. *m*-Nitrophenol (by diazo-reaction from *m*-nitroaniline) is methylated with methyl sulphate in 10, 30, or 40% alkali and the *m*-anisidine, prepared by subsequent reduction, is fused with sodium hydroxide at 180—200° in an iron vessel, *m*-nitrophenyl methyl ether is added, and the mixture heated at 230° with continual stirring. The warm, fused product is dissolved in 50% alcohol, reduced with zinc dust, and the resulting hydrazo-compound converted into *m*-dianisidine by the benzidine change under the influence of cold sulphuric acid. By a similar process *o*-dianisidine may be prepared. The methods available for the preparation of methyl (and ethyl) sulphate in good yield are critically discussed.

J. W. BAKER.

Action of aluminium chloride on hydrocarbons. STRATFORD.—See II. **Absolute alcohol.** DIETRICH.—See XVIII. **Cresol-rosin soap solution.** KOGAN.—See XXIII.

PATENTS.

Conversion of methane into petroleum-like hydrocarbons. A. GOUDET (Addn. 32,666, 26.5.26, to F.P. 613,146. B., 1928, 844).—Methane is passed at 500—900° under 1000 atm. pressure over nickel, copper, cobalt, or their compounds, together with alkali or alkaline-earth metals, halogens, or sulphur. Conversion is accelerated by the addition of a little mercury vapour and by the action of an electric field.

A. R. POWELL.

Manufacture of alkyl halides, glycols, and alcohols from cracked gases of hydrocarbon oils. Soc. ANON. D'EXPLOSIFS ET DE PROD. CHIM. (Addn. 32,638, 13.4.26, to F.P. 610,434; B., 1929, 10).—The constituents of the gas are fractionated by liquefaction. The monochlorohydrins obtained as described in the prior patent are hydrogenated in the presence of powdered nickel, cobalt, or copper to obtain the corresponding alkyl halide, which on hydrolysis yields the alcohol. Direct hydrolysis of the chlorohydrins yields the corresponding glycols.

A. R. POWELL.

Manufacture of concentrated acetic acid. Soc. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 312,046, 22.3.29. Fr., 19.5.28).—Dilute acetic acid is extracted with an acetic acid solvent (butyl acetate) and the

solution obtained is charged into a surface-heated column containing an entrainer (a petroleum hydrocarbon mixture, b.p. 99–101°) which is insoluble or sparingly soluble in water, which forms with acetic acid a binary azeotropic mixture capable of separation into layers on the addition of a small quantity of water, and which forms with water another binary azeotropic mixture having a b.p. lower than that of the first mixture. Butyl acetate flows out at the bottom of the column; vapour comprising dilute acetic acid and the entrainer passes out at the top. The dilute acid and the entrainer, after condensation of the vapour and stratification of the condensed liquid, are returned to the extraction apparatus and to the column, respectively, and a mixture of vapours comprising acetic acid and the entrainer is withdrawn at an intermediate point above the charging inlet. After condensation of the vapour and separation of the liquids, the entrainer is returned to the column and the acetic acid is passed to a subsidiary column in which final traces of water and the entrainer are removed.

L. A. COLES.

Concentration of lower aliphatic acids. BRIT. CELANESE, LTD., and J. BILLING (B.P. 320,606, 11.7.28).—Dilute acetic acid is concentrated by successive extractions with two (or more) solvents so chosen that the preferential extraction of acid rather than water is increasingly marked in successive solvents. *E.g.*, 15–30% acetic acid is extracted with methylated ether and the resulting 70% acid is concentrated to 97–98% by extraction with methylene dichloride or a mixture of ether and light petroleum.

C. HOLLINS.

Manufacture of organic acids and esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,457, 16.8.28).—In the production of acids and esters from alcohols and carbon monoxide (cf. B.P. 283,989; B., 1928, 223), a catalyst is used which does not sinter or melt at the reaction temperature (380° at 200 atm.) and which contains a difficultly reducible metal oxide and less than two equivalents of an inorganic acid. Examples are chromium metaphosphate with 5% of free acid, cerium metaphosphate with 8–10% of free acid, and a 9:1 mixture of cerium and aluminium metaphosphates with 0.5–1% of free acid.

C. HOLLINS.

Preparation of oxindole-3-propionic acid. CHEM. FABR. AUF ACTIEN VORM. E. SCHERING, Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 451,957, 12.2.26. Addn. to G.P. 431,510; B., 1926, 870).—In the process of the prior patent, isatin is replaced by oxindolealdehyde. *E.g.*, molecular quantities of oxindolealdehyde and malonic acid are melted at 150–160°, and the oxindole-3-acrylic acid is reduced with aluminium amalgam. (Cf. G.P. 433,099; B., 1927, 286.)

R. BRIGHTMAN.

Preparation of adipic acid. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P. 120,518, 30.3.26).—Technical cyclohexanol (80% purity) is slowly added (5 hrs.) to nitric acid, *e.g.*, 65% nitric acid at 20–30°. Adipic acid is obtained in 90% yield. Traces only of oxalic acid are formed. The escaping gas is mostly nitrous oxide.

R. BRIGHTMAN.

Production of unimolecular aliphatic sulphuric acid compounds and polymerised sulphuric acid compounds [Turkey-red oils]. F. SCHLOTTERBECK

(G.P. 451,458, 15.5.24).—Oils such as castor oil are rendered water-soluble by treatment with concentrated sulphuric acid, excess acid is removed, and the residue is treated with feeble hydrolysing agents, *e.g.*, dilute acids or water, until the sulphonic groups attached to the double linking are hydrolysed and the polymerised reaction products are precipitated from solution. The precipitated oil, after removal and neutralisation, yields a Turkey-red oil capable of dissolving a high proportion of hydrocarbons, chlorinated hydrocarbons, and mineral oils; the residual liquor on salting out yields a Turkey-red oil with good acid-resisting properties. Both products are of use for treating textiles and leather.

L. A. COLES.

Manufacture of acetone. CONSORT. F. ELEKTRO-CHEM. IND. G.M.B.H. (B.P. 299,330, 22.10.28. Ger., 22.10.27).—Acetic acid vapour is heated to the reaction temperature (500°) before entering the catalyst chamber, which is a tube of copper, Krupp alloy V2A, microtherm, ferrosilicon, or the like, iron being avoided.

C. HOLLINS.

Preparation of carbamide. STICKSTOFF-WERKE A.-G. RUSE, V. EHRLICH, and A. DOBOCZY (Aust.P. 107,285, 30.5.24).—Cyanamide solutions are evaporated at atmospheric pressure, a faint acid reaction being maintained. *E.g.*, cyanamide solution, from crude calcium cyanamide and carbon dioxide, containing 70 g. N/litre, is acidified with sulphuric acid (2 g.) and evaporated. Several similar additions of acid are made. The product contains 91% of carbamide, 6% of ammonium sulphate, and 3% of other products, representing 67–68 g. of the original nitrogen.

R. BRIGHTMAN.

Manufacture of derivatives of carbazole. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 320,641, 17.5.28).—Carbazole-3:6:8-trisulphonic acid (or other carbazole derivative having a free 1- or 8-position) is nitrated at 0° with mixed acid and the potassium 1-nitrocarbazole-3:6:8-trisulphonate, salted out with potassium chloride, is reduced with iron and dilute acetic acid. The sulphonic groups are then removed by heating the amine with, *e.g.*, 7% sulphuric acid at 180–190°, to give 1-aminocarbazole, m.p. 196–197°.

C. HOLLINS.

Manufacture of water-soluble secondary diazo-amino-compounds and of azo dyes therefrom. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 320,324, 4.7.28).—A diazo-, diazoazo-, or tetrazo-compound is coupled in presence of an acid-fixing agent (alkali carbonates, magnesia, caustic alkali, etc.) with a secondary amine carrying a sulphonic or carboxylic group, *e.g.*, ethyl-2-toluidine-4-sulphonic acid, ethyl-metanilic acid, ethylsulphanilic acid, sarcosine, ethyl-*p*-toluidine-3-sulphonic acid, *p*-sulphophenylglycine, di-(carboxymethyl)amine, dibenzylaminedisulphonic acid, cyclohexylglycine, etc. The products are soluble diazo-amino-compounds stable in dry form or in neutral or alkaline solution, and are used for coupling, *e.g.*, on the fibre.

C. HOLLINS.

Manufacture of anthraquinone and its derivatives [from *p*-benzoquinone or α -naphthaquinone and α -butadienes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,375, 6.7., 28.9., 1.10., 11.10.,

and 10.12.28).—*p*-Benzoquinone is condensed with 2 mols., or α -naphthaquinone with 1 mol., of an $\alpha\gamma$ -butadiene. With molten reagents or in neutral or alkaline diluents (water, alcohols, hydrocarbons, pyridines, etc. with caustic alkali) in presence of oxygen or oxidants and oxygen-transferring catalysts anthraquinones are obtained: mild condensation in absence of oxidising conditions gives 9:10-dihydroxy-1:4-dihydroanthracenes, probably by way of 1:4:11:12-tetrahydroanthra-

quinones, $C_6H_4 \begin{matrix} \diagup CO \cdot CH \cdot CH_2 \cdot CH \\ \diagdown CO \cdot CH \cdot CH_2 \cdot CH \end{matrix}$ Hot alkali alone converts 1:4-dihydroanthraquinone (a product of the condensation under suitable conditions) into anthraquinone and 9:10-dihydroxy-1:4-dihydroanthracene; in presence of air the entire product is anthraquinone. The 9:10-dihydroxy-1:4-dihydroanthracenes are slowly oxidised by air, more rapidly by ferric chloride etc., to coloured substances of a quinhydrone type. If in the condensation 2- or 2:3-halogenated α -naphthaquinone is used, hydrogen halide is readily eliminated and anthraquinones are obtained. From *p*-benzoquinone and 2 mols. of the butadiene under mild conditions the primary product is a 1:4:5:8:11:12:13:14-octahydroanthraquinone, which isomerises to a non-phenolic 9:10-dihydroxy-1:4:5:8:11:12-hexahydroanthracene, convertible by mild oxidation into the phenolic 9:10-dihydroxy-1:4:5:8-tetrahydroanthracene. Amongst the 36 examples are the following: the product from *p*-benzoquinone and isoprene is boiled with 5% aqueous potassium or sodium *m*-nitrobenzenesulphonate, to yield 2:6- and 2:7-dimethylantraquinones, separable by extraction with methyl alcohol; anthraquinone is similarly obtained from α -naphthaquinone and $\alpha\gamma$ -butadiene; 2-methylantraquinone from α -naphthaquinone and isoprene; naphthazarin is heated with $\alpha\gamma$ -butadiene in benzene at 100°, and the yellow product is stirred with aqueous sodium hydroxide at 15° to give the blue-violet sodium salt of 1:4-dihydroxy-5:8-dihydroanthraquinone, the diacetate of which is oxidised by chromic acid to quinzarin diacetate; 2:3-dimethylantraquinone is obtained by heating 2-chloro- α -naphthaquinone and $\beta\gamma$ -dimethyl- $\alpha\gamma$ -butadiene in alcohol at 100–105°; the product from 5-nitro-2:3-dichloro- α -naphthaquinone and $\alpha\gamma$ -butadiene heated together at 130° is boiled with caustic alkali, vatted, and re-oxidised to give 1-aminoanthraquinone.

C. HOLLINS.

Purification of synthetic methyl alcohol. E. TSCHUNKUR and A. KLAMROTH, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,744,180, 21.1.30. Appl., 12.10.27. Ger., 27.10.26).—See B.P. 311,468; B., 1929, 670.

Manufacture of acetic acid. H. DREYFUS (U.S.P. 1,743,659, 14.1.30. Appl., 27.5.26. U.K., 13.6.25).—See B.P. 262,832; B., 1927, 125.

Production of menthol. W. SCHOELLER and H. JORDAN, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,718,666, 25.6.29. Appl., 20.6.27. Ger., 29.6.26).—See B.P. 273,685; B., 1930, 136.

Manufacture of stable diazo-salt preparations. O. STAHLIN, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,744,149, 21.1.30. Appl., 23.7.27. Ger., 2.8.26).—See B.P. 275,245; B., 1929, 12.

Furnace for carbon disulphide production (U.S.P. 1,737,566).—See XI. **Esters of resin acids** (B.P. 298,972).—See XIII. **Vulcanisation accelerators** (U.S.P. 1,739,486).—See XIV.

IV.—DYESTUFFS.

Ostwald colour system. SCHOLEFIELD.—See I.

PATENTS.

Manufacture of acid wool dyes. I. G. FARBENIND. A.-G. (B.P. 294,986. 1.8.28. Ger., 3.8.27. Addn. to B.P. 282,409; B., 1928, 442).—4-Bromo-1-aminoanthraquinone-2-sulphonic acid is condensed with a *p*-aminoformanilide alkylated in the formamido-group.

C. HOLLINS.

Manufacture of solubilised vat dye and dyeing therewith. BRIT. DYESTUFFS CORP., LTD., and A. J. HAILWOOD (B.P. 320,742, 26.9.28. Addn. to B.P. 280,647; B., 1928, 83).—The process of the prior patent is applied to Duranthrene Red BN obtainable according to examples 2 and 3 of B.P. 26,690 of 1913 or by other modified methods.

C. HOLLINS.

Green trisazo dyes for cotton. H. CLINGESTEIN, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,701,717, 12.2.29. Appl., 6.1.28. Ger., 3.1.27).—See B.P. 308,958; B., 1929, 551.

Indigoid dyes. J. HALLER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,709,977, 23.4.29. Appl., 9.12.27. Ger., 7.1.26).—See B.P. 286,359; B., 1928, 328.

Manufacture of vat dyes. E. HONOLD, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,744,163, 21.1.30. Appl., 26.9.27. Ger., 2.10.26).—See B.P. 303,203; B., 1929, 238.

Manufacture of azo dyes from 2:3-hydroxynaphthoic acid arylides. L. LASKA and A. ZITSCHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,744,172, 21.1.30. Appl., 24.5.26. Ger., 28.5.25).—See B.P. 279,146; B., 1928, 8.

Azo dyes (B.P. 320,324). **Anthraquinone etc.** (B.P. 320,375).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Viscose. XXVIII. **Effect of mixing different substances with viscose.** S. IWASAKI and S. MASUDA (J. Cellulose Inst., Tokyo, 1929, 5, 327–337).—The effects on the viscosity of the solution and on the properties of the spun threads, of mixing casein, rubber latex, soap, sodium silicate, etc. with viscose have been determined under the conditions of composition, temperature, and rate of spinning previously employed (B., 1929, 389). Viscosity as determined by the falling-sphere method changes in the usual manner, but the magnitude of the change and the transformation point depend on the nature and amount of the substance added. Addition of 1–5 g. of “carnation” milk to the standard amount of viscose increases the strength, but has no effect on the lustre or extensibility of the threads, whilst larger amounts produce weaker threads of a milk-white colour. Latex (2.5 g.) increases the strength, but larger quantities give white threads with decreased strength and extensibility. The viscose-latex mixture

can be spun earlier than the control without latex. Soaps and sodium silicate effect no improvement in the threads and their lustre remains unchanged.

B. P. RIDGE.

Lilienfeld silk (Nuera silk). H. E. FIERZ-DAVID and A. BRUNNER (Helv. Chim. Acta, 1930, 13, 47—49).—Various photomicrographs of fibres (and cross-sections) of Lilienfeld silk are given which serve to show that the fibres become stretched on being spun into an acid bath; the strong acid-cold precipitation bath is responsible for the plasticity of the fibres, which makes stretching possible. The strength of the fibres depends on the parallel orientation of the "crystallites" in them.

H. BURTON.

Use of sulphite waste liquor for the preparation of sulphate-cellulose. O. ROUTALA and S. WECKMAN (Suomen Kem., 1929, 2, 147—153).—Sulphite waste liquor was treated with sodium sulphate, the precipitated calcium sulphate was filtered off, and the filtrate evaporated to dryness. A causticised solution of the ignited residue contained sodium hydroxide 40.4 g., carbonate 10.6 g., and sulphide 12.1 g./litre. Pine-wood chips heated with this solution for 5½ hrs. at 8.5 atm. maximum pressure gave a 40% yield of a typical sulphate-cellulose which contained extract 1.08%, lignin 5.3%, and pentosan 5.84%. In order to investigate the chemical reactions which occur, the sulphite waste liquor containing calcium was submitted to dry distillation, when methyl mercaptan, methyl disulphide, a substance (m.p. 40°) containing sulphur, and a well-defined crystalline substance of m.p. 119—120° were obtained.

B. P. RIDGE.

Steam and power production in the soda-house of sulphate-cellulose works equipped with vacuum evaporators and waste-gas steam boilers. I. MALM (Papier-Fabr., 1930, 28, 57—60).—The problem of power production should be treated in conjunction with the steam production of the works. The power and steam production and the arrangement of plant in a medium sized sulphate-cellulose works are discussed, and it is shown that by centralisation of steam production in the soda-house and improvement in the plant arrangement on the lines suggested, whereby full use is made of secondary (waste) heat supplemented as required by coal dust and/or gas firing of direct-heated boilers, considerable economy may be effected. B. P. RIDGE.

Distinction between cellon and cellophane. G. SÁNDOR (Z. angew. Chem., 1929, 42, 1108).—If cellophane is exposed to filtered ultra-violet radiation from a quartz mercury-vapour lamp it does not fluoresce at all or only does so very feebly, whereas cellon shows a faint blue fluorescence. If both materials are dipped for 30 sec. in a 0.5—1% solution of extract of pine bark, then washed, dried, and again exposed to the ultra-violet rays, cellophane shows a violet fluorescence, whereas cellon shows its original pale blue.

A. R. POWELL.

Determination of the strength of soda-cellulose. KIENZL and NAGL (Papier-Fabr., 1930, 28, 49—55).—The effects of time of beating, stuff density, amount of material used, rate of beating, moisture content, acidity or alkalinity, and temperature of the stuff have been investigated under laboratory conditions to determine

the most satisfactory procedure for beating soda-cellulose in the ball-mill before testing its strength. The use of 800 c.c. of stuff of 2.5% density, which is beaten at 250 r.p.m. for the first 30 min. and subsequently at 300 r.p.m., is recommended. Wet stuff reaches a high degree of beating sooner than dry, but on prolonged beating approximately the same degree is attained by both. Increasing concentration of mineral acid decreases the degree of beating obtained under standard conditions, whilst weak organic acids and caustic soda have little effect. Increase in temperature causes a slower rate of beating. Use of the ball-mill shortens the time necessary to reach a certain degree of beating of soda-cellulose as compared with that necessary with the test-hollander. Apparatus for preparing circular test-sheets of paper is described, and it is shown that the average variation from the mean of results from such sheets is no greater than that obtained with samples of technically manufactured paper. B. P. RIDGE.

Beating of [paper] pulp. IV. Beaten pulp. I. Beaten stuff. II. M. NAKANO (J. Cellulose Inst., Tokyo, 1929, 5, 264—269, 283—290).—I. The wetness of beaten pulp is increased by stirring, and the higher the pulp consistency the greater is the increase of wetness. Beaten pulp does not combine chemically with water, but the latter is held as water of imbibition. Dilute solutions of acid and alkali and mercuric chloride solution prevent increase of wetness on keeping, and treatment with boiling alkaline solutions of any concentration causes loss of wetness. Brown, mechanical wood pulp increases in wetness after bleaching. The wetness of a mixture of two different pulps cannot be calculated from that of the components.

II. The swelling of cellulith and vulcanised fibre in water has been observed over a period of about 100 days by weighing the water taken up by them, but no syneresis is found. The true degree of beating of the pulp, *i.e.*, its wetness after boiling, depends on the mean dimensions and the mean modulus of elasticity of the fibres, whilst the wetness lost by boiling depends on the mean dimensions of the beaten fibres and on the water of imbibition. B. P. RIDGE.

Beating of [paper] pulp. V. Relation between time and true degree of beating. M. NAKANO (J. Cellulose Inst., Tokyo, 1929, 5, 317—326).—The effect of beating stuff in the ball-mill has been investigated from the point of view of wetness. Apparatus is described with which the degree of wetness may be measured. As previously shown (*cf.* preceding abstract) the apparent wetness, W , may be divided into two parts, *viz.*, the true degree of beating, W_m , and the wetness W_a , due to water of hydration or imbibition, which is readily removed by boiling. The latter has no effect on the beating action. The relation between W_m and the time of beating or number of revolutions of the mill (x) is expressed by straight lines when $W_m - k$ (where k is a constant of the apparatus) is plotted against x on semilogarithmic paper. From such straight lines the equation $W' = W'_0 e^{\alpha x}$ is deduced, where $W' = W_m - k$, W'_0 is the initial degree of beating obtained from the graph, and α is the slope of the straight line. The agreement between observed and calculated values of W'

is good, but observed values at the beginning of beating do not agree with the computed values; also the formula ceases to hold when the stuff is so far beaten that paper made from it begins to parchmentise. Over a certain range, however, the true corrected degree of beating increases with time according to the compound interest law.

B. P. RIDGE.

Measurement of the transparency to light of printing papers. E. H. RIESENFELD and T. HAMBURGER (Papier-Fabr., 1930, 28, 55–57).—Limitations of some of the general methods of measurement of the transparency to light of papers are discussed, and a method which has proved satisfactory in the works is described. Diffused light from a high-power lamp is passed through the paper to be tested and through a number of layers of tissue paper placed, respectively, in adjacent chambers of the viewing apparatus. These chambers are connected by blackened tubes to a Duboseq prism attachment similar to that of a colorimeter, and the number of layers of tissue paper is altered until equal intensity of transmitted light is obtained in both fields. The number of layers necessary is then a measure of the transparency of the test paper. The tissue paper used is compared with a standard plate of grey glass of known transparency to light. Ordinary newspaper should have transparency numbers (*i.e.*, the number of sheets of tissue paper required for matching) of 16–19, and good newspapers 20–25. The higher the number the more suitable is the paper for printing purposes.

B. P. RIDGE.

PATENTS.

Manufacture of vegetable wool. J. J. LAMBRECHT (B.P. 313,169, 4.6.29. Fr., 9.6.28).—After a mechanical treatment, to impart flexibility by separation of small films and a portion of adhering gum, the fibres are lubricated by means of a hot saponifying solution containing water, oleic acid, mineral oil, sodium hydroxide and carbonate, and subjected to the action of a rapidly circulating cold solution of caustic soda. They are then washed with acidulated water, bleached, rinsed, and softened in a solution of neutral soap and sodium monosulphide.

F. R. ENNOS.

Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,736,829, 26.11.29. Appl., 16.4.27).—The fur is treated with a neutral or acid solution of an inorganic or organic salt of manganese.

F. R. ENNOS.

Rendering animal hair capable of felting. S. MARIAN (Austr.P. 108,990, 20.12.24).—The hair is treated with aqueous solutions of salts (*e.g.*, of thorium, uranium, thallium, copper, zinc) which hydrolyse, and the metals or oxides of which form insoluble compounds with the animal fibre.

L. A. COLES.

Felting of animal hair. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 323,364, 28.12.28).—The hair is treated with a solution of hydrofluoric or hydrofluosilicic acid or a salt of either acid.

F. R. ENNOS.

Manufacture of mineral fibre articles. PLASTIC INC. (B.P. 312,901, 22.11.28. U.S., 2.6.28).—Raw kelp, asbestos, and sodium carbonate are incorporated together, and after addition of a precipitant (calcium

acetate) the whole is passed through a papermaking machine.

F. R. ENNOS.

Spinning of textile materials. Production of artificial textile yarns and threads. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 323,221 and 323,209, 19.9.28).—(A) Filaments of artificial silk, after extrusion from the spinning nozzle, are associated to form a thread, which is stretched, again separated into individual filaments, and subjected to the action of a blast of air in an ejector device, whereby an irregular fibrous mass of filaments is produced. (B) The fibrous mass produced as described in (A) is teased or combed to render the filaments unidirectional, torn or cut to staple lengths, and spun in a manner similar to that used for wool or cotton.

F. R. ENNOS.

Manufacture of viscose. I. FRENKEL (B.P. 323,213, 24.9.28).—To diminish the formation of noxious gases etc. (hydrogen sulphide, sulphur dioxide, carbon disulphide), 0.3–0.4% of sodium nitrite is added to the viscose solution during manufacture.

F. R. ENNOS.

Production of artificial filaments, fibres, bands, sheets, films, etc. of a high lustre from aqueous cellulose solutions. H. KINDERMANN (B.P. 323,173, 4.12.28).—The partially coagulated product obtained by treatment in a preliminary bath of ammonium sulphate is fixed in a sulphuric acid solution (45–50% by wt.) below 10°, and simultaneously subjected to tension. [Stat. ref.]

F. R. ENNOS.

Treating artificial silk cakes made by the centrifuge-spinning process. F. KUTTNER A.-G. (B.P. 300,131, 5.11.28. Ger., 5.11.27).—Flattened and rinsed cakes produced as described in B.P. 287,097 (B., 1928, 854) are also dried while in this form, prior to reeling.

F. R. ENNOS.

Impregnation of celluloid. A. WEBER, H. HÖPPNER, JUN., and H. WEICH (B.P. 318,981, 1.10.28. Ger., 14.9.28. Addn. to B.P. 316,276; B., 1929, 1011).—To the treating solution claimed in the prior patent 3–6% of acetic anhydride is added.

F. R. ENNOS.

Regeneration of sulphite-cellulose waste liquors. SOC. FRANÇ. DES CELLULOSES BARBOU & Co. (F.P. 634,402, 14.9.26).—Sodium sulphite, which may be added as such or produced in the solution by the addition of sodium carbonate or hydroxide followed by treatment with sulphur dioxide, and other reducing agents, *e.g.*, sulphides, polysulphides, hydrosulphides, to retard oxidation of the sulphite, are added to the liquor after cellulosic material has been removed from it.

L. A. COLES.

Saccharification of wood and other cellulosic materials. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 323,693, 3.4.29. Belg., 30.1.29. Addn. to B.P. 311,695; B., 1930, 185).—A modification of the prior patent in which the cellulose is esterified by vapours of anhydrous acid, the ester being subsequently hydrolysed by vapours of dilute formic acid or steam.

F. R. ENNOS.

Manufacture of wood pulp. W. BRYDGES and S. C. FOSTER, ASSRS. to BEDFORD PULP & PAPER CO., INC. (U.S.P. 1,737,542, 26.11.29. Appl., 18.1.29).—Blocks of wood are heated by steam under pressure while immersed in an alkaline solution contained in a closed

tank, the gases and crude oils formed being removed from time to time. The tank is then drained and filled with warm water in which the blocks are soaked before grinding. F. R. ENNOS.

Transparent sheet material. J. CHAMBERLAIN and H. PERIAM (B.P. 323,302, 30.10.28).—Glazed transparent paper ("Glassine") is impregnated with a solution of castor oil in benzene, dried, again impregnated with a solution of shellac or a synthetic resin, and finally dried. F. R. ENNOS.

Protection of wool, fur, rugs, etc. against the attack of moths. E. HARTMANN, M. HARDTMANN, and P. KÜNNEL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,744,633, 21.1.30. Appl., 26.11.26. Ger., 27.11.25).—See B.P. 303,092; B., 1929, 241.

Rotary steam-heated drying cylinders [for yarns etc.]. J. C. EVANS (B.P. 323,621, 11.12.28).

Spinning of artificial silk threads [on to bobbins etc.]. C. TYRER and G. H. MAHOOD (B.P. 323,812, 13.10.28).

Dehydrator (U.S.P. 1,737,533).—See I. **Printing ink** (U.S.P. 1,738,798). **Artificial masses** (B.P. 298,135).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Azoic colours and their application to wool and union goods. J. A. WALLWORK (J. Soc. Dyers and Col., 1930, 46, 10—12; cf. Everest and Wallwork, B., 1929, 774).—The methods and machinery suitable for wool, worsted or union yarn, or cloth, and loose wool and slubbing are described. General rules with regard to the control of the depth of shade on the cotton and wool in a mixture of these fibres are also given. N. CHAPPELL.

Detection of chemical damage of wool. P. KRAIS and V. SCHLEBER (Leipziger Monatsschr. Textil-Ind., 1929, 44, 165—167, 211—215; Chem. Zentr., 1929, ii, 953—954).—Coarser kinds of wool are more resistant than finer towards alkalis. Wool already damaged by alkali is markedly affected by acid dyeing; the action of various dyes is considered. A. A. ELDRIDGE.

Ostwald colour system. SCHOLEFIELD.—See I.

PATENTS.

Preparations suitable as washing, wetting, emulsifying, solvent, and similar agents, particularly in connexion with textiles. H. T. BÖHME A.-G. (B.P. 298,560, 27.9.28. Ger., 11.10.27).—Wetting-out agents highly resistant to saponification and capable of conferring softness on textile materials are prepared by mixing aromatic sulphonic acids (e.g., propylnaphthalenesulphonic acid) with sulphonic acids obtained from fatty acids containing more than 8 carbon atoms, e.g., oleic acid (cf. B.P. 263,117; B., 1928, 845).

A. J. HALL.

Increasing the impermeability to water of articles or products composed of regenerated cellulose, cellulose hydrate, or cellulose ethers which are not resistant to water. WOLFF & Co. KOMM. GES. AUF AKT., and R. WEINGAND (B.P. 298,605, 25.9.28. Ger., 12.10.27).—The articles are treated, in the presence of a plasticiser if desired, with a water-miscible swelling

liquid (formic or acetic acid), the excess of which is expelled by heat, and are afterwards coated with a solution of a cellulose ester with which the swelling liquid is compatible, the solvent being removed by volatilisation.

F. R. ENNOS.

Treatment of films, threads, fabrics, etc. made from or containing cellulose esters and ethers. H. DREYFUS (B.P. 323,521, 24.7.28).—To diminish the water-repelling properties and to facilitate dyeing with direct or mordant dyes, the materials are impregnated with cellulose by treating with (a) viscose, cuprammonium, zinc chloride, etc. solutions of cellulose and precipitating in the usual way, or (b) with a solution of nitrocellulose in a non-solvent for the material treated and de-nitrating by means of a dilute ammoniacal solution of a sulphhydrate.

F. R. ENNOS.

Dyeing animal materials. A. G. BLOXAM. From VER. F. CHEM. U. MET. PROD. (B.P. 315,899, 22.3.28).—Wool, silk, hair, skin, feathers, etc. are dyed by the ice-colour method using as padding (coupling) components aromatic sulphonamides, especially arylsulphonaryl-amides; these have affinity for animal materials, and the latter can be washed after padding. Examples are: *p*-toluenesulphonyl-*m*-phenylenediamine, and its *N'*-2:3-hydroxynaphthoyl- (m.p. 213—214°), *p*-*cresotyl*- (m.p. 194—195°), 1:4-*hydroxynaphthoyl*- (m.p. 120°, decomp.), and 2-*hydroxy*-5:6:7:8-*tetrahydro*-3-*naphthoyl*- (m.p. 210—211°) derivatives; the *N'*-*salicyl*-derivative, m.p. 197—198°, of *p*-toluenesulphonyl-*p*-phenylenediamine; *o*-2:3-*hydroxynaphthamidotoluene*-*p*-sulphonamide and -*anilide*; *ar*-*tetrahydro*- β -*naphthol*-3-*sulphonanilide*; β -*naphthol*-3:6- or -6:8-*disulphonanilide*; *o*-amino-*p*-toluenesulphonamide \rightarrow 1:5-dihydroxynaphthalene; 1-(*p*-aminosulphonyl-*o*-tolyl)-3-methyl-5-pyrazolone.

C. HOLLINS.

Local coloration of materials made of or containing organic esters of ethers of cellulose. BRIT. CELANESE, LTD. (B.P. 299,849, 30.10.28. U.S., 2.11.27).—Acetate silk etc. is treated with basic aluminium acetate or other basic metallic acetate, with or without acetic or formic acid, and preferably dried before printing. Sharp prints are obtained.

C. HOLLINS.

Manufacture of piece-dyed woollen fabrics containing effects of silk. I. G. FARBENIND. A.-G. (B.P. 297,124, 14.9.28. Ger., 16.9.27).—Silk is reserved in wool-silk mixtures by addition of a sulphonic acid capable of precipitating glue and gelatin from acid solution (cf. B.P. 7137/1913 and 288,127; B., 1914, 269; 1928, 613) to the dye-bath, preferably after dyeing for $\frac{1}{2}$ hr. and before chroming.

C. HOLLINS.

Dyeing and printing natural or artificial fibre with sulphur and vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,978, 9.8.28).—Wool in union fabrics is protected from the action of the alkali in sulphide or vat dye-baths by addition of amines or heterocyclic bases, e.g., "triethanolamine," cyclohexylmono- and di-ethanolamines, pyridine, dibutylamine, *m*-xylidine, cyclohexylamine.

C. HOLLINS.

Printing or stencilling of cellulose derivatives [acetate silk]. BRIT. CELANESE, LTD. (B.P. 297,848, 29.9.28. U.S., 30.9.27).—Penetration is improved by

adding to the printing paste ethylene dichloride or other halogen addition or substitution products of ethylene.

C. HOLLINS.

Production of white or coloured discharges on artificial silks. R. CLAVEL (B.P. 308,757, 3.8.28. Ger., 31.3.28).—Artificial silks, especially acetate silk, weighted with tin or zinc salts by the methods described in B.P. 277,602, 280,094, 300,894, or 303,129 (B., 1928, 154, 296; 1929, 811, 774) and dyed with direct dyes are readily discharged by means of pastes containing sodium hyposulphite such that clear white or coloured effects are obtained.

A. J. HALL.

[Sizing of] artificial silk. SONDERMANN & Co. (MECHANISCHE WEBEREI SESSMAR, SONDERMANN & Co. G.M.B.H.) (B.P. 297,778, 27.9.28. Ger., 27.9.27).—Untwisted filaments of artificial silk are unwound from bobbins, treated with sizing agents, dried, and rewound.

F. R. ENNOS.

Mildew-proofing treatment of textile materials, goods, etc. BRIT. COTTON INDUSTRY RES. ASSOC., R. G. FARGHER, L. D. GALLOWAY, and M. E. PROBERT (B.P. 323,579, 18.10.28).—The materials are treated with a suspension or solution of an arylamide of salicylic acid or its sodium salt, *e.g.*, salicyl-anilide or -o-anisidide.

F. R. ENNOS.

Manufacture of colour-printed fabrics. J. MORTON (U.S.P. 1,744,410, 21.1.30. Appl., 3.1.28. U.K., 22.11.27).—See B.P. 292,415; B., 1928, 601.

Treatment [dyeing] of cellulose derivatives. G. H. ELLIS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,723,271, 6.8.29. Appl., 17.5.27. U.K., 21.10.26).—See B.P. 284,376; B., 1928, 229.

Treatment of [dyed materials of] cellulose acetate. G. H. ELLIS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,723,230, 6.8.29. Appl., 11.9.25. U.K., 23.9.24).—See B.P. 243,841; B., 1926, 124.

Obtaining lustre and matt effects on yarns or fabrics composed wholly or in part of viscose in the sulphur state. F. SCHOLEFIELD and N. DENNER, Assrs. to BURGESS, LEDWARD, & Co., LTD. (U.S.P. 1,724,375, 13.8.29. Appl., 1.6.26. Ger., 21.7.25).—See B.P. 261,099; B., 1927, 72.

Dyeing apparatus. ECLIPSE TEXTILE DEVICES, INC., Assces. of L. B. HASBROUCK (B.P. 303,893, 7.1.29. U.S., 12.1.26).

Dyeing apparatus. J. Y. JOHNSON. FROM HUSSONG DYEING MACHINE Co. (B.P. 323,899, 18.12.28).

Process and machine for simultaneous dyeing and finishing using the same treating liquid. H. HELTEWIG (B.P. 324,191, 19.12.28).

Method and apparatus for finishing textile goods. J. HÖFNER (B.P. 324,052, 15.10.28).

Sulphonated aliphatic compounds (G.P. 454,458).—See III. Dyeing with vat dyes (B.P. 320,742).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Chamber process [of sulphuric acid manufacture]. XVIII. Total [heat balance] and differential heat balance [of each section] of a Glover tower.

M. MATSUI and T. SAKAMAKI (J. Soc. Chem. Ind., Japan, 1929, 32, 193—196 B).—The above heat balances were computed from data previously recorded. From the total heat balance the heat-radiation factor for the tower was calculated, the corrected mean temperature of the tower being obtained by multiplying the mean of the gas temperatures at the bottom and top of the tower by the general factor 0.77165. The differential heat balance indicates that maximum formation of acid occurs at the comparatively low section where maximum acid concentration occurs, less acid being formed higher up the tower owing to the condensation of water. This is at variance with the conclusions of Kaltenbach (B., 1920, 484 A).

S. K. TWEEDY.

Determination of available alkalinity in commercial lime. C. M. JOVELLANOS (Philippine J. Sci., 1930, 41, 71—74).—Pure lime shows a sharp end-point on titration with acids; all commercial limes, however, which contain alumina, iron, and particularly silica, which tend to form compounds with the lime during the calcination process, hydrolyse and cause return of the colour of the phenolphthalein indicator. The A.S.T.M. method for the determination of alkalinity is so modified that the completion of the titration is effected on the solution of the entire sample and not on aliquot portions of the diluted solution.

E. LEWKOWITSCH.

Examination of alkali iodides. W. MEYER (Süd-deut. Apoth.-Ztg., 1929, 69, 461—462; Chem. Zentr., 1929, ii, 1566).—Treatment of a freshly prepared 5% solution of alkali iodide with a few drops of starch solution and dilute sulphuric acid should not give an immediate blue colour (absence of iodic acid, copper, and iron); addition of a drop of 0.01*N*-iodine solution should give a blue coloration which does not disappear on shaking (absence of thiosulphate).

A. A. ELDRIDGE.

Preparation of ammonium chloride by reaction between ammonium sulphate and common salt. K. KIEFER (Chem.-Ztg., 1930, 54, 62).—The necessity of recrystallising the ammonium chloride obtained, in order to free it from sodium sulphate, may be avoided by adding sufficient sulphuric acid to convert the normal sulphate into the acid salt.

S. I. LEVY.

Artificially crystallised Karlsbad salt. A. KUHN (Pharm. Zentr., 1930, 71, 5—6; cf. Wallrabe, B., 1929, 895).—The dried mixture of salts prepared from the water itself, or a synthetic mixture of the same composition, gives, on dissolving, a solution which is more alkaline than the natural water, because of the loss of carbon dioxide. To prepare artificially a suitable water, two solid mixtures should be used, one containing sodium bicarbonate and neutral salts, the other sodium bisulphate and neutral salts.

S. I. LEVY.

Stability of hydrogen peroxide solutions. H. H. WILLRATH (Chem.-Ztg., 1930, 54, 51).—Samples of 30% peroxide solutions from various makers showed no appreciable loss in strength after keeping for 3 months at $\pm 7^\circ$. Even at 36° the loss was slight. Exposure to ultra-violet radiation caused no measurable decomposition.

S. I. LEVY.

Sulphur from gas. PETIT; KOERS.—See II. **Permeability of metals to gases.** LOMBARD.—

See X. Manganese dioxide for dry cells. KANEKO and others.—See XI. Mercuric fulminate. WÖHLER and BERTHMANN.—See XXII.

PATENTS.

Reduction of rare refractory-metal oxides. M. N. RICH, ASS. to WESTINGHOUSE LAMP CO. (U.S.P. 1,738,669, 10.12.29. Appl., 9.11.27).—A mixture of the oxide, a calcium-magnesium alloy, and an alkaline-earth halide is heated, *e.g.*, at 800–850°, in a hermetically-sealed container (cf. U.S.P. 1,704,257; B., 1929, 399).

L. A. COLES.

Manufacture of titanium dioxide. F. DOERINCKEL and L. MEHLER, ASSIS. to TITANIUM PIGMENT CO., INC. (U.S.P. 1,738,765, 10.12.29. Appl., 25.1.27. Ger., 1.2.26).—A titanium solution is hydrolysed in the presence of sulphur dioxide, sodium sulphite, or similar reducing agent added in such quantity as to correspond to the tervalent iron and other oxidising agents present in solution; the titania produced remains white at incandescence.

H. ROYAL-DAWSON.

Preparation of ammonium formate from cyanamide. G. TRÜMLER (Swiss P. 119,222, 15.6.26).—A neutral solution of calcium cyanamide containing ammonium sulphate is reduced electrolytically at a nickel or copper gauze cathode previously plated with spongy copper and/or nickel. The anolyte consists of 10% sulphuric acid and the anode of a lead sheet coated with lead dioxide.

A. R. POWELL.

Manufacture of arsenates [calcium arsenate]. C. D. CARPENTER (U.S.P. 1,737,639, 3.12.29. Appl., 30.4.24).—Arsenious oxide is heated above 150° (*e.g.*, at 350°) with sodium nitrate, preferably with excess of sodium hydroxide in addition; after the evolution of nitric oxide has ceased, the product is dissolved in water and calcium arsenate is precipitated by adding milk of lime, collected, washed, and dried.

L. A. COLES.

[Manufacture of] magnesium perchlorate. G. F. SMITH (U.S.P. 1,738,930, 10.12.29. Appl., 13.6.27).—A mixture of anhydrous ammonium perchlorate and precipitated magnesium oxide or carbonate is ground, *e.g.*, in a ball mill, and the product is dried at 180–350°, and, if desired, *in vacuo*.

L. A. COLES.

Oxidation of ammonia. F. G. LILJENROTH (U.S.P. 1,744,652, 21.1.30. Appl., 11.1.26. Swed., 29.1.25).—See F.P. 610,160; B., 1927, 251.

Separation of alkaline-earth phosphates from primary alkali phosphate solutions. F. DRAISBACH, ASS. to J. A. BENCKISER CHEM. FABR. (U.S.P. 1,744,371, 21.1.30. Appl., 29.2.28. Ger., 21.6.26).—See B.P. 298,436; B., 1928, 893.

Preservation of dibasic calcium hypochlorite. H. S. SCHULTZE, ASS. to I. G. FARBENIND. A.-G. (U.S.P. 1,743,242, 14.1.30. Appl., 25.7.27. Ger., 18.8.26).—See B.P. 276,307; B., 1928, 13.

Extraction of iron and titanium compounds from titanium ores. D. GARDNER and L. TAVERNER (U.S.P. 1,743,885, 14.1.30. Appl., 6.8.23).—See B.P. 207,247; B., 1924, 100.

Preparing fire foam (U.S.P. 1,739,094).—See I. Acid-resistant vessels (G.P. 457,732).—See VIII. Silver from films etc. (F.P. 633,685).—See XXI.

VIII.—GLASS; CERAMICS.

Loss of ultra-violet transparency in glasses. S. ENGLISH (Nature, 1930, 125, 85–86).—Experiments supporting Wood and Leathwood's observation (B., 1929, 897) that all glasses are not equally susceptible to loss of ultra-violet transparency are described, together with tests which disagree with the view (cf. *loc. cit.*) that natural solarisation is complete in a few days. Loss of transparency can proceed up to a period of six months.

L. S. THEOBALD.

Crystalline products in a baryta glass. P. NICOLARDOT and P. GAUBERT (Bull. Soc. Franç. Min., 1928, 50, 333–338; Chem. Zentr., 1929, ii, 1196).—The composition and appearance of readily fusible, crystalline spherulites in an optical baryta glass are recorded.

A. A. ELDRIDGE.

Development of an enamel on a eutectic basis. H. E. SIMPSON (J. Amer. Ceram. Soc., 1930, 13, 62–79).—The possibility of combining $\text{RO}-\text{R}_2\text{O}_3-\text{SiO}_2$ eutectics so as to obtain a eutectic solvent which would dissolve the maximum amount of silica was studied, with the object of developing an enamel of high resistance. This involved a study of the $\text{RO}-\text{B}_2\text{O}_3-\text{SiO}_2$ systems, in particular, of the $\text{CaO}-\text{B}_2\text{O}_3-\text{SiO}_2$ system. A member of this system, high in CaO and SiO_2 (CaO 21.5%, B_2O_3 63.1%, SiO_2 15.4%), though not a eutectic, was chosen as being the most suitable for blending with the other eutectics to form a eutectic solvent for enamels. The mixtures were applied to cast iron by the dry process, and the physical properties of the enamels were studied. Silica was also added to the enamels up to 1.5 mol.-equivalents. Greatest brilliance and smoothness were obtained with the high-lead members; the high-soda members showed least tendency to chip and greatest resistance to acid attack.

F. SALT.

Blistering phenomena in the enamelling of cast iron. A. I. KRYNITSKY and W. N. HARRISON (J. Amer. Ceram. Soc., 1930, 13, 16–61).—A continuation of previous work (cf. Harrison, Saeger, and Krynitsky, B., 1929, 96) is reported. Physical defects in the cast iron, particularly "sponginess," give rise to blisters, but the composition, preparation, and method of application of the enamel may also be contributory causes. The gases forming the blisters are carbon monoxide and dioxide. Cast iron normally used for enamelling acquires a very thin surface skin, or "microchill," due to freezing and cooling. Removal of this layer eliminates blistering, except when it is caused by sponginess or similar physical defects. The combined carbon of this skin breaks down during the enamelling process to finely-divided "temper carbon," which is readily oxidised, leading to the formation of these gases. A non-blistering iron is probably one in which the combined carbon is present either in small quantity or in a comparatively stable form. The presence of the "microchill" probably accounts for inconsistencies in results obtained from different sources. To avoid such inconsistencies, a uniform method of sand-blasting to remove the skin would have to be applied.

F. SALT.

Rapid method for estimation of the alumina content of diaspores and high-alumina clay. S. M. PHELPS and A. C. HUGHES (J. Amer. Ceram. Soc.,

1930, 13, 1—4).—The method is based on the relation found between the density and the alumina content of aluminous clays. The apparatus consists simply of a glass bottle, a perforated stopper, and a plunger. Arbitrary values for the bulk density were determined on dry, powdered samples of a number of diaspores, varying in alumina content from 45% to 80%. These values agreed sufficiently well with the alumina contents found by chemical analysis to warrant the use of the method for plant-control purposes. F. SALT.

Process control in continuous production [of ceramic products]. System of the Champion Porcelain Co. F. H. RIDDLE and M. F. ROYAL (Ind. Eng. Chem., 1930, 22, 14—20).—The proportion of rejections in the manufacture of sparking-plug cores has been greatly reduced by the adoption of continuous processes and a rigid system of control tests. The raw material (andalusite) is continuously sampled and its d determined. From each wagon load a small-scale production run is made and storage is so arranged as to give the maximum uniformity to each day's make. After the first grinding any iron is separated and the material is afterwards ground only between sillimanite surfaces. Fineness tests are taken hourly. Batches are similarly tested for fineness, d , and viscosity after grinding. Complete batches are filter-pressed, the pressure being continuously recorded and the cakes sampled. This sample is made into finished cores during the ageing period. The forming of the cores is subject to constant control of the dimensions, which do not vary more than 0.005 in. The Dressler kiln used is fired with fuel oil and possesses a series of thermocouples both in the top and in the sides. The temperature (1455°) in the hot zone can be held constant within 2°, chiefly by means of automatic pressure control at the ends of the kiln. The gas composition is recorded and maintained at the desired figure by the auxiliary oil-feed. The sillimanite products prepared in this way comprise, in addition to sparking plugs, laboratory ware, pyrometer tubes, and refractories.

C. IRWIN.

Stoneware masses. I. Composition. F. SINGER (Ber. deut. keram. Ges., 1929, 10, 269—271; Chem. Zentr., 1929, ii, 1340).—If the system $RO-Al_2O_3-SiO_2$, where $R = Mg, Ca, Sr, Ba, Zn, \text{ or } Fe$, is so heated that at least three phases of a certain consistency are present simultaneously, stoneware having an expansion coefficient of 0.15×10^{-6} can be obtained; this value is one third of that of silica. The composition of the phases is discussed.

A. A. ELDRIDGE.

Recent development of special electromagnetic separators and applications of interest to the general pottery trade. W. E. BOX (Trans. Ceram. Soc., 1929, 28, 447—456).—An account is given of modern apparatus for the removal of both coarse (tramp) and finely-divided iron from pottery materials. F. SALT.

Comparative effects of glass batch, cullet, soda, and sulphate on fireclay bricks. P. P. BUDNIKOV (Feuerfest, 1929, 5, 181—183).—Ten bricks of Russian manufacture of the type used for the bottom and sides of glass tanks were used in the investigation. The chemical composition, the refractoriness under load,

and the porosity were determined, and the chemical resistance was studied on small cubes in which circular cavities had been bored. The cavities were filled with the reagents and the specimens were then heated in an electric furnace and held at 1430° for 2 hrs. The glass batch mixture was slightly more active than the cullet, but in neither case was corrosion very marked. The sulphate showed the most corrosive action, especially in the presence of carbon, when the sodium sulphide formed reacted vigorously with the refractory material. Soda also proved a powerful corrosive. In every case the degree of corrosion was dependent on the porosity of the material. F. SALT.

Absorption [in bricks] in relation to durability. O. J. WHITEMORE (J. Amer. Ceram. Soc., 1930, 13, 80—83).—Criticism is offered of the A.S.T.M. (C34—27) specifications for hollow building bricks on the grounds that the absorption test gives no reliable indication of the ability of the bricks to resist frost action. The data refer only to bricks made from Iowa clays. An alternative classification is suggested. F. SALT.

Apparatus for the determination of adsorbed air [in porcelain slip]. H. M. KRANER and R. A. SNYDER (J. Amer. Ceram. Soc., 1930, 13, 11—15).—A modification of the Toepler pump was used to measure the amount of air in an electrical porcelain slip containing 57% of water and 43% of solid matter. The method of procedure is described. At boiling pressure (25 mm.) 4.65% by vol. of gases had been removed, and this increased to 4.83% at 1.8 mm., when the sample had become dry. Of this volume of removed air, 2.8% was held by the clay particles. F. SALT.

Phase equilibria in the system SiO_2-ZnO . E. N. BUNTING (J. Amer. Ceram. Soc., 1930, 13, 5—10).—Mixtures of the materials were heated in a platinum capsule until equilibrium was established and then quenched in water. The phases present were identified with the petrographic microscope. Only one compound, Zn_2SiO_4 , was formed at high temperatures; its m.p. is $1512^\circ \pm 3^\circ$. It forms a eutectic with tridymite at 1432° and 49.1 mol.-% ZnO , and a eutectic with zinc oxide at 1507° and 77.5 mol.-% ZnO . The optical properties of the zinc orthosilicate obtained agreed closely with those of willemite. No evidence was found of the existence of the metasilicate. The m.p. of zinc oxide was determined in an iridium-platinum vessel contained in a thoria crucible; it was found to be $1975^\circ \pm 25^\circ$. F. SALT.

PATENTS.

Material for window glazing and similar purposes. M. A. COLEFAX (B.P. 323,671, 12.2.29).—Cellulose acetate sheet is interposed between sheets of "Vita" glass, the whole being permeable to ultra-violet rays. S. K. TWEEDY.

Purification of kaolin and clays. Y. M. PIRAUD (F.P. 633,745, 3.5.27).—A suspension of the material in pure water is treated with a protective colloid, such as gum arabic, pectin, soap solution, containing amines or ammonia etc. The sand etc. settles out and the suspension of clay material is decanted off and flocculated by means of acid. S. K. TWEEDY.

Production of raw material for [dental] porcelain casts. KUNSTZAHN. GES.M.B.H. (B.P. 296,765, 7.9.28. Ger., 7.9.27).—Fritted porcelain material is rendered viscous by heating, cooled, and broken up into pieces about the size of a pea; each piece is then heated until sufficiently viscous to assume a spherical form with a smooth exterior. The product, which may be melted with a blowpipe flame, yields blister-free castings.

L. A. COLES.

Manufacture of earthenware vessels resistant to hydrochloric acid. TECHNOCHÉMIE A.-G. (G.P. 457,732, 4.9.27).—The material used, *e.g.*, kaolin and other clays, is ground so that the particles are finer than 8μ . Earthenware made in the usual way from this material does not lose ferric oxide when heated with hydrochloric acid.

A. R. POWELL.

Refractory support for the resistors of [electric] furnaces. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of J. SCHNEFF (G.P. 458,043, 18.7.26).—The usual refractory support is coated with zirconia by dipping it into a suspension of the finely-divided oxide or by covering it with zirconium which is subsequently converted into oxide by burning.

A. R. POWELL.

Refractory material. A. HASSELBACH, Assr. to G. POLYSIUS (U.S.P. 1,744,547, 21.1.30. Appl., 26.2.26. Ger., 5.3.25).—See B.P. 276,185; B., 1927, 816.

Manufacture of refractory, acid-resisting, and other products bonded by means of clay. H. ACKERMANN, W. LANGENHEIM, and H. KNUTH (U.S.P. 1,744,351, 21.1.30. Appl., 17.6.26. Ger., 22.6.25).—See B.P. 253,947; B., 1927, 254.

Manufacture of [curved sheets of] safety glass. E. BURNET (B.P. 323,745 and 323,783, 8.10.28).

Tunnel kilns (U.S.P. 1,737,540).—See I.

IX.—BUILDING MATERIALS.

Mixed Portland cements. I, II. S. NAGAI (J. Soc. Chem. Ind., Japan, 1929, 32, 101—103 B, 190 B).—I. Tensile and compressive-strength tests on 1 : 3 sand mortars of Japanese mixed Portland cements are recorded, which show that whereas the strength of the blast-furnace slag mortars has improved recently, that of "soliditit" mortars is inferior and has not improved. Strength tests on similar mortars of "neo-soliditit" (Jap.P. 80,692) show that this cement has high initial strength, the strength of the mortars increasing remarkably on prolonged ageing and reaching after 13—26 weeks a compressive strength of 800—900 kg./cm.² and a tensile strength of 50—60 kg./cm.²

II. "Neo-soliditit" is made by mixing 70—75% of Portland cement clinker with 10—20% of calcined granite, basalt, etc. and 10—20% of siliceous material rich in soluble silica, alumina, and ferric oxide, and grinding the whole with 3—5% of gypsum. The effect of changing the granite and siliceous constituents on the properties of the finished cement was investigated. Some physical properties of the cement are recorded.

S. K. TWEEDY.

Action of saline waters on hydraulic cements. O. REBUFFAT (Giorn. Chim. Ind. Appl., 1929, 11, 529—531).—In the system $\text{Na}_2\text{SO}_4 + \text{Ca}(\text{OH})_2 \rightleftharpoons \text{CaSO}_4 + \text{NaOH}$, representing the disaggregating action of saline waters

more or less rich in sodium sulphate, the effect is enhanced by moderate dilution of the sulphate solution. The reverse action becomes of importance only with high concentration of the sodium hydroxide.

T. H. POPE.

Composition and properties of so-called "natural cements." HAEGERMANN (Zement, 1929, 18, 861—868; Chem. Zentr., 1929, ii, 1340).—So-called "natural cements" are of poorer quality than ordinary cements. Data are tabulated.

A. A. ELDRIDGE.

Resistance of concrete to chemical action. R. GRÜN (Z. angew. Chem., 1929, 42, 1070—1074).—Whilst the chemical resistance of concrete cannot be greater than that of the aggregate used in its manufacture, the limiting factor is usually the resistance of the binder. The chemical composition of Portland cement, blast-furnace cement, and alumina cement is discussed, together with the reactions involved in setting. The resistance of concrete to various reagents has been examined. It is found to depend both on the physical structure of the concrete and on the nature of the reagents, the results being summarised as follows. Ammonia has been reported as harmful, but experiment does not confirm this, and the injury may have been caused by impurities present. Sulphurous acid probably causes little harm as such, but in practice it is always partly oxidised to the more injurious sulphuric acid. Carbonic acid is harmful only in aqueous solution. Acetic, lactic, and saccharic acids have caused great injury to concrete in certain cases. Ammonium sulphate and magnesium salts are very harmful. Protection is best secured by the maximum density of structure, by the use of a bitumen covering, and especially by extra care during constructional work.

C. IRWIN.

Small-piece testing on strength of cement mortar. I, II. S. NAGAI (J. Soc. Chem. Ind., Japan, 1929, 32, 191—193 B).—The ordinary testing of cement mortars is carried out under conditions which differ from those under which the cement is practically utilised in concrete or mortar. A small-piece testing method is proposed, based in principle on the method of concrete testing. The water-cement ratio is determined by the "slump-testing" or flow-testing methods. If *c* is the compressive strength of a small cylindrical test-piece (15—20 mm. diam., 30 mm. long) and *C* that of a test-piece of normal size, then *C* is a linear function of log *c* and *c* is a hyperbolic function of *C/c*.

S. K. TWEEDY.

Russian asphalt. GRAEFFE.—See II.

PATENTS.

Lime or cement mortar. A. GENAIRON (B.P. 306,038, 13.2.29. Fr., 14.2.28).—Blood-serum is mixed with cement or lime to form a mortar which can be spread with a brush, is absolutely impermeable and resistant to water, heat, and cracking, and adheres perfectly to all materials (including glass).

S. K. TWEEDY.

Manufacture of bricks and the like. CAFFERATA & Co., LTD., and B. J. CAFFERATA (B.P. 323,630, 22.12.28).—The burnt clay or marl brick is soaked in a solution of metallic salt or salts (*e.g.*, sulphate or chloride

of iron, manganese, copper, cobalt, or chromium), and reburned in order to convert the imbibed salt into oxide, which latter imparts colour to the brick. Alternatively, before being reburned, the soaked brick may be immersed in a solution capable of converting the metal salt into hydroxide, silicate, or borate; a deeper and more permanent colour is obtained thereby. Neutralised metal salt solutions may be used with advantage. S. K. TWEEDY.

Preparation of road-construction and the like material. CHEM. FABR. DR. K. ALBERT GES.M.B.H. (B.P. 309,070, 6.2.29. Ger., 4.4.28).—Disintegrated stone or other mineral filler is mixed with a water-insoluble binding medium such as tar, bitumen, pitch, mineral oil, etc., in the presence of an aqueous agent, e.g., sulphite-cellulose waste liquor, which acts as an emulsifier towards the binding medium.

S. K. TWEEDY.

Making of roads, pavings, foundations, etc. BERRY, WIGGINS & Co., LTD., and H. H. HOLMES (B.P. 323,896, 17.12.28).—The viscosity of aqueous bitumen emulsions for use in binding mineral aggregates is increased by the addition of alkali silicate solutions during the mixing operation. L. A. COLES.

Drying of lumber and other moisture-containing materials. H. W. COWAN (B.P. 297,480, 22.9.28. Can., 23.9.27).—Lumber may be heated to as high a temperature as 93° without damage, provided the maximum temperature alternates with periods of reduced temperature. Various cycles of heating and change of air are claimed, based on the above principle.

B. M. VENABLES.

Manufacture of sheets of artificial marble or similar coloured material. J. A. TROUWBORST, Assee. of C. and P. ZWART (B.P. 303,831, 9.1.29. Holl., 10.1.29).

Reproducing surface-markings of marble etc. (B.P. 308,788).—See XXI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Sulphur in the cupola-furnace process. M. NIKLAU (Zentr.-Europ. Giesserei-Ztg., 1929, 2, No. 5, 1—2; Chem. Zentr., 1929, ii, 1581).—The m.p. of the slag is 1300—1400°, and a suitable composition is:—17—20% Al_2O_3 , 22—32% CaO , 50—55% SiO_2 . Desulphurisation is necessary only for contents around 0.05—0.08%. A. A. ELDRIDGE.

Growth of cast iron considered in the light of recent research. P. BARDENHEUER (Stahl u. Eisen, 1930, 50, 71—76).—A comprehensive and critical survey of recent work on the origin and mechanism of the growth of cast iron. A. R. POWELL.

Nature of the segregation in [steel] ingots after solidification. G. D'HUART (Rev. Mét., 1929, 26, 532—537).—A sulphur print taken on the surface of a longitudinal cross-section through the middle of a cylindrical ingot of mild steel shows the presence of sulphide inclusions along the sides of an inverted V at the lower part of the ingot and numerous sulphide segregations of a minor order, each in the shape of a small V and with its point in line with that of the larger

A. The following explanation of this behaviour, which is independent of the shape of the ingot within wide limits, is given. When the molten metal is poured into the mould the first crystals appear as dendrites extending inwards perpendicular to the walls of the mould, the length of the dendrites increasing towards the top of the ingot where the rate of cooling increases. At a certain stage in the cooling contraction is so great that the solid shell shrinks away from the mould and further cooling proceeds more slowly; the ferrite crystals formed in this period appear generally throughout the still liquid mass and fall to the bottom of the central liquid cone, while the globules of sulphide which are thrown out of solution rise through the liquid and become attached to the dendritic crystals first formed, thus forming a conical zone of sulphur-rich metal round an inner core of almost pure ferrite. The sulphide globules form nuclei for the crystallisation of phosphide and carbide, which therefore tend to segregate below and throughout the sulphide zone. The smaller V-shaped segregations at the top of the ingot are due to suction effects on the final very impure liquid metal which is forced towards the top at the end of the solidification. A. R. POWELL.

Hardness and structural changes on heating cold-rolled steel containing 1.15, 0.90, or 0.60% C. R. JONSON (Jernkontorets Annaler, 1929, 207—235; Chem. Zentr., 1929, ii, 1064).—The temperatures at which recrystallisation begins and ends are the higher the higher is the carbon content. The optimum temperature lies between 650° and the A₁ point. Structural changes accompanying recrystallisation were not observed. A. A. ELDRIDGE.

System sulphur-iron-carbon. H. HANEMANN and A. SCHILDKÖTTER (Arch. Eisenhüttenw., 1929—1930, 3, 427—435; Stahl u. Eisen, 1930, 50, 42—43).—The quasi-ternary system iron-iron sulphide-iron carbide contains a ternary eutectic (m.p. 975°) with 87% FeS, 2.5% Fe_3C , and 10.5% Fe; there appears to be no ternary solid solution at the Fe_3C corner of the ternary diagram and only a small range in the iron corner as the solid solubility of sulphur in iron does not exceed about 0.03%. The solubility of iron in ferrous sulphide at the ternary eutectic temperature is about 3%, and that of Fe_3C in iron at the same temperature is 20%. Molten mixtures having a composition indicated by any point on the straight line joining the Fe- Fe_3C eutectic point to the Fe-FeS eutectic point in the triangular diagram separate into two liquid phases, the upper containing 0.22% C and 30.65% S and the lower 4.12% C and 0.98% S. The determination of small quantities of carbon in alloys with a high sulphur content is unsatisfactory by the usual combustion method, as not all the sulphur dioxide is retained by the chromic acid tubes. It is preferable, therefore, to collect both sulphur dioxide and carbon dioxide in a concentrated solution of sodium hydroxide free from carbonate, then to oxidise the sulphite with permanganate, and finally to acidify the solution with sulphuric acid and expel the liberated carbon dioxide into a measured excess of 0.1N-potassium hydroxide; after addition of barium chloride the excess of alkali is titrated with 0.1N-oxalic acid, using phenolphthalein as indicator. A. R. POWELL.

[Resistance to corrosion of] cast iron containing nickel and copper. M. BALLAY (Rev. Mét., 1929, 26, 538—553).—The microstructure and resistance to corrosion of 15 cast irons containing up to 15.6% Ni, 6.5% Cu, and 2% Cr have been investigated. The most resistant alloy of those tested was that containing 2.16% Si, 15.63% Ni, 5.76% Cu, and 1.28% Cr; this withstood cold dilute mineral acids, acetic acid, and sea-water as well as or better than a good bronze, but was more readily corroded by hot sulphuric acid. The alloy has an austenitic structure, is easily worked, and does not scale badly at high temperatures.

A. R. POWELL.

System iron-phosphorus-carbon. R. VOGEL (Arch. Eisenhüttenw., 1929—1930, 3, 369—381; Stahl u. Eisen, 1930, 50, 14—15).—In the binary system iron-phosphorus, alloys with up to 21% P contain the constituent Fe_3P when cooled rapidly and Fe_2P when cooled slowly, the former being in stable and the latter in unstable equilibrium. The α - γ transformation region in phosphorus-iron alloys is a closed field, as is the γ -region; the maximum solid solubility of phosphorus in α -iron is 0.6% and in γ -iron 0.25%. Addition of phosphorus to iron-carbon alloys reduces the solid solubility of carbon at all temperatures. In the system $\text{Fe}-\text{Fe}_3\text{C}-\text{Fe}_3\text{P}$ there are (i) five primary liquidus surfaces, at the temperatures of which there separate from the liquid phase on cooling ternary α - or δ -solid solution, ternary γ -solid solution, cementite, Fe_2P , and Fe_3P ; (ii) corresponding secondary surfaces of equilibrium between two of the above crystal phases and liquid; and (iii) two planes of equilibrium between three crystal phases and liquid, namely (A) a transition plane at 1005°, the phases of which are: α -solid solution with 0.3% C and 2.2% P, γ -solid solution with 0.5% C and 2% P, liquid with 0.8% C and 9.2% P, and the phosphide Fe_3P ; and (B) the plane of the ternary eutectic at 950°, with the phases γ -iron with 1.2% C and 1.1% P, Fe_3P , Fe_3C , and liquid with 2.4% C and 6.89% P. Owing to the transformations which iron undergoes in the solid state and to the irregular contour of the fields of existence of the various phases, the study of the equilibria in solid ternary alloys is highly complicated; thus, on cooling some of the alloys, a new phase may separate at a certain temperature and redissolve again at a lower temperature. In alloys in which at 745° two three-phase equilibria occur, viz., (a) between α -iron containing 0.1% C and 1.5% P, γ -iron with 0.8% C and 1% P, and the phosphide Fe_3P , and (b) between γ -iron, Fe_3P , and Fe_3C , a reaction takes place between the four phases which results in the slow disappearance of the α -iron and Fe_3P . Thus in alloys with a low phosphorus content the constituent Fe_3P disappears at 745°; during cooling to 720° a further reaction takes place, the γ -iron solid solution decomposing into pearlite in which the ferrite constituent contains at the most 0.1% C and 1.5% P. The temperature of the pearlite transformation point in steels is therefore raised slightly by the presence of phosphorus, the maximum temperature being 745°.

A. R. POWELL.

Determination of tungsten, chromium, and vanadium in high-speed tool steels. W. BRÜGGE-

MANN (Chem.-Ztg., 1929, 53, 927—928, 947—950).—For the determination of tungsten a sample of the steel (1—5 g. according to the tungsten content) is dissolved in 50—100 c.c. of 1:2 hydrochloric acid together with a drop or two of hydrofluoric acid if much silicon is present. The solution is slowly evaporated to about 20 c.c. and the iron and tungsten are oxidised by the cautious addition of just sufficient dilute nitric acid; after boiling for 3 min., 100 c.c. of boiling water are added and the tungstic acid is collected on a pulp filter, washed first with dilute hydrochloric acid and then with 1% ammonium nitrate, ignited, and weighed. No silica is contained in the precipitate, but in the case of poorly heat-treated steel some chromium carbide may be undecomposed by the above treatment. In this case the ignited precipitate is fused with sodium hydroxide at a low temperature and the tungsten precipitated as mercurous tungstate from the filtered solution of the fusion. An aliquot part of the acid filtrate from the tungsten is evaporated with 10 c.c. of sulphuric acid and 10 c.c. of phosphoric acid until fumes just begin to appear, 500 c.c. of tap water are added, and the chromium is oxidised to chromic acid by boiling with 5 c.c. of 0.5% silver nitrate solution and 10 g. of ammonium persulphate until bubbles of oxygen cease to appear. The silver is precipitated and any permanganic acid destroyed by further boiling with 5 c.c. of 1:1 hydrochloric acid until the silver chloride coagulates and settles readily. After cooling, a slight excess of ferrous sulphate solution over that required to reduce the chromic and vanadic acids is added and the solution is titrated with permanganate. The difference between the amount of permanganate required for the volume of ferrous sulphate solution alone and that required after addition of the assay is calculated to chromium, a small allowance of 0.1—0.3 c.c. being made for errors in obtaining the end-point. An excess of ferrous sulphate is added to the solution after titration of the chromium to reduce vanadic acid to vanadyl sulphate, the excess of ferrous sulphate is oxidised by addition of ammonium persulphate to the cold solution, and the vanadyl sulphate is titrated with permanganate. The persulphate excess is without action either on vanadyl sulphate or on permanganate in cold solutions.

A. R. POWELL.

Permeability of metals to gases. Classification of the various gas-metal systems. V. LOMBARD (Rev. Mét., 1929, 26, 519—531).—Recent work on the diffusion of gases through metals is critically reviewed and an attempt is made to deduce an equation which will express the rate of diffusion of any gas through any metal. The closest approximation to the experimental results is afforded by the expression $d = K(\sqrt{p/h}) \times 10^4$, where K and d are constants which are different for every system, p is the pressure of the gas on one side of the metal (the other being in a vacuum), h the thickness of the metal, and t the absolute temperature. The various systems so far investigated are arranged in order of decreasing permeability of the metal at 500° as follows: hydrogen-palladium, hydrogen-mild steel, hydrogen-electrolytic iron, hydrogen-nickel (1% Co, 0.5% Fe, and 0.15% Si), hydrogen-electrolytic copper,

hydrogen-electrolytic nickel, hydrogen-zinc, carbon monoxide-mild steel, helium-nickel, hydrogen-platinum, argon-nickel, nitrogen-nickel, oxygen-silver, hydrogen-aluminium. A. R. POWELL.

Corrosion of metals and alloys. A. PORTEVIN (Rev. Mét., 1929, 26, 606—631, 635—654).—A review of recent work on corrosion, detailing the nature and cause of the various types of corrosion of steel, copper alloys, and light alloys, and the numerous methods of studying the rate of corrosion and the value of protective coatings. Research directed towards the production of incorrodible alloys such as stainless steels is discussed with especial reference to the theoretical aspect of the subject. A. R. POWELL.

Action of water on aluminium vessels, and the effect of aluminium compounds on the organism. C. MASSATSCH (Hausz. V.A.W. Erftw. Alum., 1929, 1, 75—88; Chem. Zentr., 1929, ii, 1212).—Experiments with pure aluminium and silumin and different waters at the ordinary temperature and at 80° show that the extent of attack is of the same order as with other metals. Acid foods cause greater corrosion than fatty foods. Alumina is physiologically harmless. A. A. ELDRIDGE.

Copper-aluminium [alloys] containing manganese, tin, or cobalt. E. MORLET (Rev. Mét., 1929, 26, 464—487, 554—569, 593—605).—Full experimental details are given of work on the effect of manganese, tin, and cobalt on aluminium bronzes containing 80—90% Cu, the chief results of which have already been published (A., 1929, 995). Quenching of alloys with tin or manganese from 850—900° generally produces a martensitic structure. The solubility of cobalt in alloys with a structure consisting of $\alpha + (\alpha + \gamma)$ increases with rise of temperature. Excess of cobalt over the limit of solid solubility results in the appearance of needles of a new constituent along the grain boundaries; this constituent is insoluble in α but dissolves in γ to a limited extent. A. R. POWELL.

Reduction of zinc oxide by means of gaseous carbon monoxide at atmospheric pressure and at high pressures. O. DONX (Bull. Acad. Roy. Belg., 1929, [v], 15, 254—264).—The reduction of zinc oxide by gaseous carbon monoxide at 1100° proceeds more rapidly than in the usual commercial method by means of solid carbon. Increase of pressure favours the rate of production of the metal. O. J. WALKER.

Zinciferous slags and the rotary [furnace] process. W. STAHL (Chem.-Ztg., 1930, 54, 79).—The zinc in the slags of the zinc- and lead-smelting industries is mainly present as ferrite, ZnFe_2O_4 , although a little is in the form of silicate and of zinc oxide mechanically retained in the slag. The method of recovering the zinc by roasting the slag with coke in rotary furnaces is described; during this process deposits containing a considerable proportion of metallic iron are formed in them. H. F. HARWOOD.

Treatment of residues from [the working-up of lead ores by] the Harris process. F. VOGEL (Chem.-Ztg., 1930, 54, 49—50).—A discussion of the economics of treatment of the residues for recovery of antimony and tin. S. I. LEVY.

Rapid determination of iron in nickel[-plating] baths. O. GRUBE (Chem.-Ztg., 1929, 53, 935).—Into a 10-c.c. centrifuge tube are placed 5 c.c. of the solution to be analysed, 4 c.c. of 35% sodium acetate solution, a few drops of acetic acid, and 1 c.c. of 1% potassium chlorate solution, the mixture is heated for 10 min. in a boiling water-bath, and the tube placed in the centrifuge and rotated for 3 min. at maximum speed. The volume occupied by the precipitate multiplied by 20 gives the weight in g. of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) per litre of electrolyte sufficiently accurately for technical purposes. A. R. POWELL.

Enamelling of cast iron. KRYNITSKY and HARRISON.—See VIII. **Aluminium vessels for culinary purposes.** THIEME.—See XIX.

PATENTS.

Blast-furnace operation. W. A. HAVEN (U.S.P. 1,738,577, 10.12.29. Appl., 9.4.27).—The pressure of the blast supplied to a furnace is caused to fluctuate by means of a rotary pump and pulsator. C. A. KING.

Blast-furnace tuyère. E. H. HOLZWORTH (U.S.P. 1,738,901, 10.12.29. Appl., 11.7.27).—The nose of a tuyère is jacketed with an air chamber from which a jet of air is directed downwardly into the molten metal adjacent to the mouth of the tuyère. C. A. KING.

Hot-blast cupola. C. D. BARR, Assr. to AMER. CAST IRON PIPE Co. (U.S.P. 1,738,277, 3.12.29. Appl., 15.11.27).—To form a recuperator for a cupola, rectangular blocks of iron or other material are set round the refractory lining and are connected together by means of tapered nipples. As many rings are installed as may be necessary. Air from a compressor enters an upper chamber and travels helically down the recuperator blocks to a bottom collecting chamber, out of which the tuyères are supplied. C. A. KING.

Melting and refining furnace. T. F. BAILY (U.S.P. 1,739,343, 10.12.29. Appl., 28.1.28).—A melting furnace heated by electric arc is superposed on a refining furnace of crucible type provided with a bottom electrode and a ring electrode near the top. Molten metal runs from the upper furnace into the refining furnace through a layer of slag above the refined metal, which is tapped from the lower part of the furnace. C. A. KING.

Melting furnace for metals. REBOURG & DUPONT (F.P. 634,113, 10.5.27).—The furnace comprises an upper melting chamber, in which the charge is placed on a mushroom-shaped support and melted by the flames of an oil burner which play around the support, and a lower collecting chamber in which the molten metal is further heated by induction to the desired casting temperature. Oxidation of the molten metal is thus almost completely prevented. A. R. POWELL.

Continuous-heat annealing furnace. F. J. WINDER, Assr. to ALLEGHENY STEEL Co. (U.S.P. 1,738,130, 3.12.29. Appl., 30.3.28).—The length of a sheet-annealing furnace is sufficient to allow of the preheating, annealing, and cooling of the sheets which are suspended edgewise from the top and carried by travelling means on the side wall of the furnace. The furnace may be

closed to allow the internal atmosphere to be controlled.

C. A. KING.

Coating of metal articles with metal. J. VON BOSSE (B.P. 323,847, 1.11.28).—The metal article is given a preliminary coating with, *e.g.*, nickel, then treated as is described in B.P. 286,632 (B., 1928, 677) to remove occluded gases, again coated with a second metal (*e.g.*, chromium), and finally degassed. The process is of use in the production of sliding surfaces for light-metal cylinders for internal-combustion motors.

H. ROYAL-DAWSON.

Tubes or flexible hose-pipes with an internal non-rusting coating. O. MEYER-KELLER & CO. (Swiss P. 123,773, 22.7.26).—Metals bands from which the tube or hose is to be made by means of a longitudinal weld or by spiral winding and welding are previously coated on one side by the spraying process with a metal which is resistant to corrosion and which has a higher m.p. than the metal of which the bands are made, so that it remains unaffected during the subsequent welding.

A. R. POWELL.

Recovery of silver and lead from their pure oxides. SOC. METALLURG. CHILENA "CUPRUM" (G.P. 450,229, 5.6.23. Addn. to G.P. 447,686; B., 1930, 10.).—Calcium chloride is used instead of sodium chloride in the leaching solution.

A. R. POWELL.

Oxalate bath for electrolytic tinning. STUDIEN-GES. F. WIRTSCHAFT U. INDUSTRIE M.B.H. (G.P. 457,874, 13.5.26).—The bath comprises a saturated solution of ammonium oxalate, oxalic acid, and ammonium chlorostannate, to which is added dithionic acid.

A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 309,071, 5.3.29. U.S., 4.4.28).—A sugar, such as barley sugar, cane-sugar caramel, malt sugar or syrup, is added to the acid cadmium bath to secure a bright, homogeneous, and coherent deposit.

H. ROYAL-DAWSON.

Hardening metal articles by nitrogenisation. A. FRY, ASSR. to NITRALLOY CORP. (U.S.P. 1,737,711, 3.12.29. Appl., 31.10.28. Ger., 17.11.27).—See B.P. 300,633; B., 1930, 63.

Aluminium alloy. H. C. HALL and T. F. BRADBURY, ASSRS. to ROLLS ROYCE, LTD. (U.S.P. 1,744,545, 21.1.30. Appl., 27.3.29. U.K., 3.4.28).—See B.P. 300,078; B., 1929, 24.

Tunnel kilns (U.S.P. 1,737,540). Separation of ores (B.P. 303,810).—See I. **Electric furnace (U.S.P. 1,739,344).**—See XI.

XI.—ELECTROTECHNICS.

Electric heating [for rectifying stills] and furnaces. O. DONY (Bull. Acad. Roy. Belg., 1929, [v], 15, 243—253).—Soft iron, in the form of rods at least 5 mm. in diam., is recommended as the heating resistance for electric furnaces. Temperatures up to 1200° can be maintained for long periods, and the internal volume of the graphite furnace described can be as high as 15 litres without using more than 200 amp. A lead rectifying still, in which the heating source consists of a soft-iron filament immersed directly in the liquid, is described. By adjusting the filament current the speed of the

distillation can be regulated very exactly, and it is possible, for example, to purify toluene completely from thiophen by distillation alone. O. J. WALKER.

Manganese dioxide for dry cells. I, II. S. KANEKO, C. NEMOTO, and S. MAKINO (J. Soc. Chem. Ind., Japan, 1929, 32, 205 B, 206 B).—I. The *E.M.F.* and capacity of a dry cell are independent of the manganese dioxide content of the oxide used, but are larger in the case of artificial oxide than in the case of the natural substance; the coarser-grained oxide gives the higher *E.M.F.* The relations between the capacity and the grain size and the rate of discharge are irregular.

II. X-Ray examination showed that various kinds of natural manganese dioxide, such as pyrolusite and psilomelane, have the same crystal structure, and that natural and artificial dioxide have different crystal structures.

S. K. TWEEDY.

Russian asphalt. GRAEFE.—See II. **Electromagnetic separators.** BOX.—See VIII. **Nickel-plating baths.** GRUBE.—See X. **Acidity of oils.** SELTZ and SILVERMAN.—See XII. **Electrodialysis cell.** CROWTHER and BASU.—See XVI. **Electrodialysis of molasses.** KAMEYAMA and MAYEDA.—See XVII.

PATENTS.

Electric furnace. T. F. BAILY (U.S.P. 1,739,314, 10.12.29. Appl., 28.1.28).—In a furnace of inverted-cone shape, adapted more especially for the production of synthetic pig iron from scrap steel, an electrode is arranged at the lower smaller end and movable electrodes at the upper end are adapted to be moved downwards in paths substantially parallel to the side walls of the furnace and in alinement with the upper end of the lower electrode. The upper part of the furnace side walls may be lined with carbon for conveying part of the current. The furnace is provided with a central charging opening at the top and a removable bottom carrying the bottom electrode.

J. S. G. THOMAS.

Electric furnace [for producing carbon disulphide]. P. BROWN, ASSR. to BROWN CO. (U.S.P. 1,737,566, 3.12.29. Appl., 17.6.25).—Sulphur and charcoal are introduced into the furnace at a temperature near or within the optimum temperature range (800—1000°) of the reaction $C + 2S = CS_2$. The furnace comprises a jacketing or heating chamber surrounding a reaction chamber throughout which the desired temperature may be maintained. The jacketing chamber wall is constructed of heat-conducting refractory material, and a pair of electrodes is arranged near the bottom of the reaction chamber.

J. S. G. THOMAS.

Inductor furnace. E. F. NORTHRUP (B.P. 316,659, 14.5.29. U.S., 2.8.28).—The outer part of the furnace is protected against stray magnetic fields by surrounding the inductor coil by an auxiliary coil, the current in which produces a magnetic field assisting the return magnetic flux in the space between the two coils, but opposes and approximately balances the return flux in the space outside the auxiliary coil. J. S. G. THOMAS.

[High-power] electric induction furnace. ALLMÄNNA SVENSKA ELEKTRISKA AKTIEBOLAGET (B.P. 310,031, 12.4.29. Swed., 21.4.28).—Parts of the winding of the

generator or transformer feeding the furnace are connected between the several series-connected portions into which the primary winding of the furnace is divided. The power factor is corrected by condensers connected in series or in parallel with each of the furnace-winding portions.

J. S. G. THOMAS.

[Mounting of resistors in] electric furnaces. A. N. OTIS and G. W. HEGEL, ASSRS. to GEN. ELECTRIC CO. (U.S.P. 1,738,446, 3.12.29. Appl., 24.10.28).—An electric heating resistor is removably mounted on a refractory framework and secures the latter to supports in the furnace side walls.

J. S. G. THOMAS.

Electrical resistance and its formation. H. PENDER and J. H. MUELLER (U.S.P. 1,739,256, 10.12.29. Appl., 22.8.24).—A film of germanium is formed on the inner walls of a glass tube by passing a stream of germanium hydride through the tube heated at about its softening temperature. Electrical contact is made between the film and heated metal caps containing rosin and fusible alloy thrust over the ends of the tube.

J. S. G. THOMAS.

Electrical resistance wire. OESTERR. SIEMENS-SCHUCKERT-WERKE, ASSECS. of F. BERGER (Aust.P. 108,851, 15.9.26).—The part of the wire connecting the resistance, *e.g.*, of a wire-wound furnace, to the source of current is wound round a wire of a highly-conducting metal such as copper. In this way it is unnecessary to make a welded or soldered joint, which is a source of weakness in most furnaces of the wire-wound type.

A. R. POWELL.

Carbon disc resistance. N. V. MACHINERIEEN EN APPARATEN FABR. (F.P. 633,579, 29.4.27. Ger., 5.5.26).—In the type of resistance consisting of a column of loose discs of carbon with metal plates between; the latter consist of nickel, silver, non-rusting steel, or other difficultly oxidisable metal.

A. R. POWELL.

Incandescence cathode. SIEMENS & HALSKE A.-G. (Aust.P. 109,083, 14.10.25. Ger., 17.12.24).—Refractory metal filaments are coated with hafnium, produced by reduction of hafnium compounds, *e.g.*, the oxide, by an alkali metal. Hafnium so produced has a greater emissive power than hafnium produced otherwise, *e.g.*, by reduction with carbon.

J. S. G. THOMAS.

Incandescence cathode. C. H. F. MÜLLER (Aust.P. 109,080, 19.3.25. Ger., 12.3.25).—Uranium or its compounds are incorporated in filaments or wires of tungsten, molybdenum, etc. Such filaments or wires are more easily mechanically worked, are not sensible to traces of gases, and can be overheated without detriment to their emissive power.

J. S. G. THOMAS.

Oxidised incandescence electrodes. V. H. WOHL (Aust.P. 108,259, 13.4.26).—Platinum or platinum-iridium wire coated (preferably electrolytically) with a metal (silver) having a great affinity for mercury is immersed in, *e.g.*, barium amalgam, and then heated in a non-reacting atmosphere, so that the mercury and silver volatilise, leaving a coating of barium on the wire.

L. A. COLES.

Cutting electrode. R. W. HOLT, ASSR. to FUSION WELDING CORP. (U.S.P. 1,738,246, 3.12.29. Appl., 11.10.26).—A core of alumina is coated with silica.

J. S. G. THOMAS.

Treatment of electron-emission elements. S. RUBEN, ASSR. to RUBEN TUBE CO. (U.S.P. 1,739,044, 10.12.29. Appl., 12.1.27).—A coating consisting of a mixture of an alkaline-earth oxide, *e.g.*, strontium oxide, and a binder, applied to a support, *e.g.*, a tungsten filament, is heated and compressed at a temperature near its m.p. Suitable apparatus is described.

J. S. G. THOMAS.

[Gas-filled] electric-discharge tubes. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 323,766, 3.8.28).—The tubes contain excess of one or more of the metals hafnium, zirconium, and titanium in contact with one or more compounds of the alkali or alkaline-earth metals, which are reduced by the former on heating and produce non-volatile or only slightly volatile substances.

J. S. G. THOMAS.

[Operation of] electric [arc] discharge tubes [emitting ultra-violet radiation]. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 299,704, 22.10.28. Holl., 29.10.27).—The operating current density is maintained so low, either by use of a suitable voltage and a resistance in series with the discharge, or by the use of a suitable gas filling, *e.g.*, argon, that the temperature of the tube is below 100°, and the vapour of the mercury contained in the tube is less than 0.3 mm.

J. S. G. THOMAS.

Screen inside X-ray tubes. PHÖNIX RÖNTGEN-RÖHRENFABR. A.-G. (Aust.P. 108,849, 23.7.26. Ger., 5.8.25).—The cathode of the tube is surrounded by a lead screen backed with copper, iron, or other metal, which can readily be degassed. A small aperture is provided in the screen to allow the rays to pass through in a single beam.

A. R. POWELL.

Electrical gas-purification process. ELEKTR. GASREINIGUNGS GES.M.B.H., and H. ROHMANN (G.P. 457,888, 17.5.22).—To protect the insulation of the winding of the transformer of the plant, the formation of powerful electrical oscillations during spark-discharges in the purification chamber is prevented by interposing resistances in all possible oscillatory circuits in the apparatus so that only well-damped or aperiodic discharges can occur.

A. R. POWELL.

Regulation of chemical reactions. [Automatic titration apparatus.] K. C. D. HICKMAN, ASSR. to EASTMAN KODAK CO. (U.S.P. 1,739,230, 10.12.29. Appl., 1.8.27).—The apparatus comprises a chamber in which reagents are mixed, an electrolytic cell and means for renewing the supply of electrolyte therein, a suction device connected with the cell for drawing gas evolved in the chamber into contact with electrolyte in the cell and a flow regulator for at least one of the streams of reagents flowing to the mixing chamber, this regulator being actuated by the value of the electrolytic conductivity of the electrolyte in the cell.

J. S. G. THOMAS.

Detection and segregation of bacteria in liquids. R. B. SHEMITZ and H. F. WECHSLER (U.S.P. 1,738,801, 10.12.29. Appl., 2.12.26).—A current is passed between electrodes contained within capillary tubes immersed in a U-tube holding electrolytic solution containing the bacteria, which are thus caused to segregate upon the electrodes.

J. S. G. THOMAS.

[Magnetic-core] induction furnace. P. E. BUNET, Assr. to Soc. ACIÉRIES DE GENNEVILLIERS (U.S.P. 1,743,956, 14.1.30. Appl., 25.5.28. Fr., 2.6.27).—See B.P. 291,456; B., 1929, 823.

Treatment of organic substances in liquid state electrochemically. C. LONGHI (U.S.P. 1,744,173, 21.1.30. Appl., 27.2.26. It., 7.3.25).—See F.P. 612,036; B., 1927, 436.

Construction [from lead or lead alloy] of grids, plates, frames, etc. for electric accumulators. E. T. WILLIAMS (B.P. 324,022, 13.10.28).

Prevention of boiler-scale deposits (B.P. 306,145).—See I. Ammonium formate (Swiss P. 119,222).—See VII. Support for resistors of furnaces (G.P. 458,043).—See VIII. Melting furnace (U.S.P. 1,739,343 and F.P. 643,113). Coating of metal articles (B.P. 323,847). Tinning (G.P. 457,874). Cadmium plating (B.P. 309,071).—See X. Crystals from sugar solutions (G.P. 444,575).—See XVII.

XII.—FATS; OILS; WAXES.

Cacao butter. II. The partial iodine value [P.I.V.] of fats, especially of cacao butter. H. P. KAUFMANN (Z. angew. Chem., 1929, 42, 1154—1157).—Unsaturated fats and acids are only partially brominated by a 0.1*N*-solution of bromine in methyl alcohol, which has been saturated with sodium bromide, and to which has been added iodine equivalent to the free bromine. The degree of saturation is determined by addition of potassium iodide after 2—3 hrs., and titration of the total free iodine by means of thiosulphate. With many fats, e.g., palm-kernel oil, milk fat, lard, and maize, sesamé, liver, wood, and linseed oils, the new constant (P.I.V.) agrees closely with the thiocyanogen value; this is true also for cacao butter, for which the P.I.V. is about 5 units below the ordinary iodine value, and generally slightly below the thiocyanogen value. With other fats, e.g., beef, mutton, and hardened fats, and olive, castor, and soya-bean oils, the P.I.V. is different from the thiocyanogen value, being sometimes greater and sometimes less. Examination of the free acids and of their glycerides showed that the rates and extents of bromination varied considerably, possibly by reason of different residual valencies and stereochemical configurations. S. I. LEVY.

So-called "exact iodine value." M. SETTIMI (Giorn. Chim. Ind. Appl., 1929, 11, 537—539).—Results published in the literature and those of the author's experiments show that, in the reaction between unsaturated fatty substances and halogens, processes of substitution and re-formation of a less active double linking are not excluded. Hence the use of the expression "exact iodine value" is criticised. T. H. POPE.

The thiocyanogen value and its application. New method for determining mixtures of fats. W. KIMURA (J. Soc. Chem. Ind., Japan, 1929, 32, 187 B).—The amounts of oleic, linoleic, and saturated acids in palm, olive, rice, and camelia oils (after hydrolysis) have been determined by Kaufmann's method. The validity of the method is demonstrated. A. A. GOLDBERG.

Quantitative analysis of hemp-seed oil. H. P. KAUFMANN and S. JUSCHKEWITSCH (Z. angew. Chem., 1930, 43, 90—91).—The oil (dark green) had d_4^{20} 0.9285, n_D^{20} 1.4789, iodine value (Hanus, also Kaufmann) 167, thiocyanogen value 101.6, and on analysis by the method previously described for linseed oil (cf. Kaufmann and Keller, B., 1929, 135, 401) was found to contain saturated acids 9.5, oleic acid 11.8, linoleic acid 49.8, linolenic acid 22.8 (7.5% of this is α -linolenic acid), unsaponifiable matter 0.97, and glyceryl residue 5.13%. The high content of linolenic acid serves to explain its use in the preparation of varnish. B. P. RIDGE.

Pine kernels, pistachio nuts, and their oils. W. PEYER (Apoth.-Ztg., 1929, 44, 699—700; Chem. Zentr., 1929, ii, 897).—Pine kernels give the following values: moisture 5.5, ash 4.4, light petroleum extract 45.6, ether extract 48.1, alcohol extract 56.3, nitrogen 6.5, crude protein 40.6%. Corresponding values for pistachio nuts are: 4.4, 2.6, 50.6, 52.2, 55.5, 2.0, and 12.5%. Values for the oils are, respectively, d_4^{15} 0.920, 0.9179; saponif. value 193.1, 192.0; iodine value 118.0, 88.2; acid value 4.4, 4.0; f.p. —20°, —9°; unsaponif. matter 1.98, 1.02. A. A. ELDRIDGE.

Water-white coconut oil and coconut flour. A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1930, 41, 51—58).—By cold-pressing the shredded (Philippine) coconut meats, which had been freed from the brown seed coats, a water-white oil of exceptionally high quality was obtained. This oil, and the yellow oil expressed from the commercial parings (seed coats), had, respectively, d_4^{20} 0.9150, 0.9124; saponif. value 261.4, 243.7; iodine value 5.4, 19.7; free fatty acids (as oleic) 0.05%, 0.77%; unsaponifiable matter 0.15%, 0.29%; n_D^{20} 1.4522, 1.4550; moisture and volatile matter 0.03%, —; colour (Wesson colorimeter) red 0.05, 0.9; yellow 0.30, 6.0. The sp. gr. of the water-white oil decreases uniformly with rise of temperature, being 0.9150, 0.9114, 0.9079, 0.9043 at 30°, 35°, 40°, and 45°, respectively, and is suggested as a standard for the evaluation of commercial oils. The white oil-cake, obtained after expressing the oil from shredded coconut, after drying, grinding, and extraction with a suitable solvent, forms a flour containing 20% of proteins and suitable for human consumption. E. LEWKOWITSCH.

Fatty acids from the oil of *Lycopodium clavatum*. K. H. BAUER and W. PINERS (Pharm. Zentr., 1930, 71, 33—38).—The fatty acids of lycopodium oil contain 72% of unsaturated and 28% of saturated fatty acids, including 4% of 9:10-dihydroxystearic acid (cf. "lycopodium-oleic acid" of Bukowsky, Chem.-Ztg., 1889, 21, 174), m.p. 98°, methyl ester m.p. 72°, crystallising from the esters of the "liquid" acids from a lead salt-alcohol separation. The unsaturated fatty acids consist of oleic, linoleic, and 12:13-hexadecenoic acid (cf. "lycopodium-oleic acid" of Langer, Arch. Pharm., 1889, 227 *et seq.*), the latter being identified by the occurrence of butyric and dodecanedicarboxylic acids in the products of ozonolysis. E. LEWKOWITSCH.

Japanese fish oils, including rock-fish and sand-eel oils. M. HIROSE and T. SHIMOMURA (J. Soc.

Chem. Ind., Japan, 1929, 32, 176—178 B).—The iodine value, saponif. value, acid value, refractive index, and sp. gr. of the oils expressed from a number of Japanese fish have been determined. The fatty acids from rock-fish oil contain highly unsaturated acids and cholesterol, but not vitamin-A. K. V. THIMANN.

Determination of acidity of oils and fats by the quinhydrone electrode in non-aqueous solutions. H. SELTZ and L. SILVERMAN (Ind. Eng. Chem. [Anal.], 1930, 2, 1—2).—The method previously described (cf. A., 1928, 723) has been modified. Amyl alcohol was replaced by butyl alcohol, and the empirical reference electrode and bridge replaced by a simple silver-silver chloride electrode, which can be used for long periods without deterioration. J. O. CUTTER.

The sulphur monochloride reaction of fatty oils. E. H. HARVEY and H. A. SCHUETTE (Ind. Eng. Chem. [Anal.], 1930, 2, 42—44).—The thermal interaction of purified sulphur monochloride and fatty oils with a calorimeter of low radiation loss has been investigated. It is concluded that sulphur monochloride does not specifically interact with the unsaturated linkings present in the fatty oils, but that other reactions also occur. Certain organic substances such as pyridine, quinoline, aliphatic halides, and the so-called "rubber accelerators" increase the velocity of the interaction, the increase being specific for any one type of oil. This effect is not catalytic, as some thermal interaction occurs between the added substances and the sulphur monochloride. J. O. CUTTER.

Photochemical method for measuring susceptibility of fats and oils to oxidation. G. R. GREENBANK and G. E. HOLM (Ind. Eng. Chem. [Anal.], 1930, 2, 9—10).—It has been found that the first stages in the oxidation of oils and fats which are practically undetectable by ordinary chemical means could be determined by the changes in oxidation-reduction indicators, especially when the change was accelerated by light. A glass-sided cell filled with a mixture of fat and methylene-blue (the most suitable indicator) was exposed to a constant light source. The rate of decrease, determined empirically, in optical density, which occurred consequent on the bleaching of the methylene-blue (measured by a potassium hydride photoelectric cell), was taken as a measure of the velocity of the initial oxidation processes. The results obtained are comparable with those given by the direct oxygen-absorption method (induction period). J. O. CUTTER.

PATENTS.

Deodorisation of animal and vegetable oils [fish oils]. M. ROGORIN, Assr. to ROGSTONE CHEM. RES. INC. (U.S.P. 1,737,731, 3.12.29. Appl., 14.2.25).—The oils or fats (100 gals.) are mixed, with constant agitation, with water (20 gals.) and kept at the b.p. until the water has evaporated, the temperature being raised to 115—120° for a short time to remove the last traces. The process may be accelerated by passing a current of inert gas over the surface of the liquid. E. LEWKOWITSCH.

Conversion of triglycerides into other esters. C. VAN LOON, Assr. to NAAML. VENN. A. JURGENS MAR-

GARINEFABR. (U.S.P. 1,744,596, 21.1.30. Appl., 24.2.27).—See B.P. 249,916; B., 1926, 553.

Making emulsions (B.P. 323,534).—See I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Composition of American turpentine. G. DU-Font and M. BARRAUD (Bull. Inst. Pin, 1929, 155—156; Chem. Zentr., 1929, ii, 1598).—Commercial turpentine had d_{25}^{25} 0.8601, $n_{D,25}^{25}$ 1.4675, $[\alpha]_{D,25}^{25}$ —10.13°, and contained approx. 61.4% of pinene (feebly laevorotatory) and 33.5% of nopinene. Turpentine from *Pinus heterophylla* had d_{25}^{25} 0.8553, $n_{D,25}^{25}$ 1.4631, $[\alpha]_{D,25}^{25}$ —30.78°, and contained 75.6% of pinene (strongly laevorotatory) and 21.2% of nopinene. That from *Pinus palustris* had d_{25}^{25} 0.8618, $n_{D,25}^{25}$ 1.4657, $[\alpha]_{D,25}^{25}$ +7.89°, and contained 64.3% of pinene (fairly strongly dextrorotatory) and 31.8% of nopinene. A. A. ELDRIDGE.

Quantitative hydrogenation of the principal unsaturated components of turpentine, pine oil, and rosin. W. E. SHAEFER (Ind. Eng. Chem. [Anal.], 1930, 2, 115—117).—A simple hydrogenation apparatus, limited in use to room temperatures and atmospheric pressure, is described. The hydrogen reservoir, an inverted 500-c.c. measuring cylinder, was connected directly to the reaction flask, and the whole apparatus mounted upon a board which could be shaken vigorously. Maleic acid, α -pinene, and diterpene were quantitatively hydrogenated in glacial acetic acid solution with moist palladium hydroxide as a catalyst, but only one unsaturated linking in dipentene and abietic acid, respectively, was saturated under these conditions. J. O. CUTTER.

Physical tests of pigments. F. SCHMID (Z. angew. Chem., 1929, 42, 1101—1107).—The basic principles underlying the methods used in the determination of the covering power, brightness, and colouring power of pigments used in paints are discussed, and the construction and methods of using modern instruments for determining these properties are described. The brightness of white pigments is measured by the Ostwald half-shadow photometer, the Pulfrich step-photometer, or the Bloch unimeter, using *magnesia usta* or normal white as the standard, the relative brightnesses of which are 104:100; on this basis blanc fixe has a brightness of 101—103, zinc sulphide, titania, lithopone, zinc white, and white lead 98—101, and purified barytes 96—98. The colouring power of a pigment is measured by determining the brightness of a linseed oil suspension containing 2% of the pigment and various proportions of bone-black, the standard being a titania suspension with 10% of the weight of pigment in black which has a brightness factor of 30. This standard is used to standardise the brightness of a normal white disc which is reduced to 30 by the interposition of a ground-glass plate in the incident light, and this disc is then used as the standard in actual determinations. The relative quantities of black required to give the same brightness with a constant suspension of the pigment in oil are: pure titania 673, mixed titania pigment 348, lithopone and zinc white 200, and white lead 100. The covering power is best determined by the Duboscq

immersion colorimeter, in which is determined the thickness of the pigment-in-oil emulsion which is necessary to cut the incident light down to 0.1—0.2% of its original value. If the value for the weight of pure titania to cover a given area is taken as 100, that of mixed titania pigment is 170, of lithopone and zinc white 330, and of white lead 545; i.e., the areas which will be completely covered by 1 kg. of the pigment in oil or varnish are, respectively, 17.5, 10, 5.5, and 3.3 m.² If an average thickness of paint film of each coat is taken as 55—60 μ , the number of coats required to cover completely a black background are 1.12, 1.82, 2.78, 4.00, and 2.66, respectively, for pure titania, mixed titania pigment, lithopone, zinc white, and white lead. The high number of coats required for zinc white is due to the much greater proportion of oil required in making the paint. A. R. POWELL.

[German] specification for zinc white and zinc oxide for use in paints. ANON. (Farben-Ztg., 1930, 35, 846—847).—Specification RAL 844C issued by the Reichsausschuss für Lieferbedingungen is summarised. "Metal zinc white" indicates pure zinc oxide containing not less than 99% ZnO, and not more than 0.4% PbO, the three grades "White, Green, and Red Seal" corresponding to decrease in fineness. "Zinc oxide" covers materials containing as a minimum 90% ZnO, whilst materials containing more than 10% PbO and other impurities are classed as "lead-containing zinc oxides." Zinc white pastes must contain 15—25% of refined linseed oil (to Spec. RAL 848A). "Zinc oxides" are not specified in paste form. Details of packing, sampling, testing, etc. are given. S. S. WOOLF.

Resins. IV. E. Stock (Farben-Ztg., 1930, 35, 847—848; cf. B., 1930, 204).—Acid values, saponification values, and ash content are quoted for various samples of fused amber and one of fused Congo copal. A sample of "East African copal" was examined. In appearance and analytical constants it does not agree with any known grade and is considered to be an unfamiliar type or possibly a mixture of various (West African?) copals. It shows no advantage over Congo copal for trade purposes. S. S. WOOLF.

Viscosimeter. MATTHIJSEN. Ostwald colour system. SCHOLEFIELD.—See I.

PATENTS.

Printing ink composition and its manufacture. G. A. RICHTER and E. W. LOVERING, Assrs. to BROWN Co. (U.S.P. 1,738,798, 10.12.29. Appl., 30.8.27).—Waste sulphite liquor from the digestion of wood (conditioned by boiling off the free sulphur dioxide and concentrating as required, e.g., to 32.6% of solids) is dispersed by agitation in petroleum oil (*d* 0.94—0.95) with the addition of pigment; small amounts of soap, sulphonated oils, etc. may advantageously be added to stabilise the emulsion and reduce the viscosity. E. LEWKOWITZSCH.

Refining of wood rosin. I. W. HUMPHREY and L. N. BENT, Assrs. to HERCULES POWDER Co. (U.S.P. 1,737,763, 3.12.29. Appl., 15.5.23).—The "drop liquor" obtained in the extraction of rosin from wood by low-boiling gasoline etc. is agitated with an equal weight

of a dilute (0.2—1%) ammonia or alkali solution, separated, washed with water, and distilled in the usual manner to separate the rosin, which is (preferably) distilled under vacuum at 150—205° (cf. U.S.P. 1,719,431; B., 1929, 863). The alkali having removed colouring matter, rosin so prepared can be used for soap manufacture, limed varnishes, etc. E. LEWKOWITZSCH.

Production of alkyl esters of resin acids. HERCULES POWDER Co., Asses. of A. C. JOHNSTON (B.P. 298,972, 16.6.28. U.S., 18.10.27).—Resin acids are esterified by heating the alkali salt at 130—140° with an alkyl halide (ethyl chloride, butyl bromide, amyl chloride, etc.) and alkali, preferably in presence of the alcohol corresponding with the alkyl halide. C. HOLLINS.

Decoration of artificial masses. II. OEXMANN (B.P. 298,135, 3.10.28. Ger., 3.10.27).—Colouring substances in the dry, viscous, or liquid state are introduced in a suitable manner into the powdered material (casein, cellulose derivatives, horn, ivory, etc.) which is afterwards pressed and hardened. F. R. ENNOS.

Liquid coating composition. C. DREYFUS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,742,587, 7.1.30. Appl., 5.4.24. U.K., 18.5.23).—See B.P. 222,168; B., 1924, 977.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spiral model for rubber. II. FIKENTSCHER and H. MARK (Kautschuk, 1930, 6, 2—6).—The atomic configuration of rubber suggested by Meyer and Mark (A., 1928, 1252), by a slight modification yields a spiral arrangement the capacity of which to be stretched in the manner of a spring is regarded as the cause of the elasticity of rubber. This conception is believed to explain resilient energy, the thermal changes on stretching and on crystallisation, the variation in density, the double refraction and Röntgen interferences in stretched rubber, and permanent set. Swelling of rubber by solvents is attributed to the effect of solvent molecules which force themselves between the turns of the spiral and, becoming attached to residual valencies, cause a straightening of the main valency chain. Vulcanisation effects a union between the caoutchouc spiral chains by sulphur bridges; the difference between vulcanite and soft vulcanised rubber arises from the much more complete anchorage of the caoutchouc chains with one another in the former case, whereby relative movement is prevented. D. F. TWISS.

Conditions affecting the vulcanisation of rubber.

I. Heating curves of raw rubber. Y. TOYABE and K. FUKUNAGA. II. Special properties of vulcanisation accelerators. Y. TOYABE, K. FUKUNAGA, and D. FUKUDA (J. Soc. Chem. Ind., Japan, 1929, 32, 341—342 B, 343—344 B).—I. When sheeted masticated rubber is heated in air, carbon dioxide, or hydrogen considerable evolution of heat occurs; in nitrogen little, if any, such heat generation is induced.

II. In testing mixtures of rubber with zinc oxide, sulphur, and a vulcanisation accelerator as to the degree of tendency to premature vulcanisation ("scorching"), a softening effect is found to precede incipient vulcanisation. On progressive vulcanisation the chloroform

extract increases more rapidly for accelerated mixings (containing diphenylguanidine or hexamethylenetetramine) than for the corresponding unaccelerated mixing.

D. F. TWISS.

Rate of cure [vulcanisation] of reclaimed rubber.

II. F. L. KILBOURN, JUN., and G. W. MILLER (Ind. Eng. Chem., 1930, 22, 69—73; cf. Shepard, Palmer, and Miller, B., 1928, 238).—Extraction of thinly sheeted reclaimed rubber with boiling water for 4 hrs. failed to remove all the soluble alkali from alkali reclaim. Extraction for 60 hrs. yielded three times as much alkali, and incorporation of this proportion of alkali into reclaimed rubber produced by a water process increased the rate of vulcanisation up to that of the alkali reclaim. The proportion of combined sulphur and the presence of residual accelerator (di-*o*-tolylguanidine) or of oxidation products had little influence on the vulcanisation of reclaimed rubber, the factor mainly responsible for its high speed of vulcanisation being residual alkali.

D. F. TWISS.

Vulcanisation [of rubber] without sulphur. F.

KIRCHHOFF (Kautschuk, 1930, 6, 6—12).—A review of earlier work and an account of vulcanisation with trinitrobenzene and picric acid, already recorded (cf. B., 1929, 1023).

D. F. TWISS.

Ostromislensky's method of vulcanisation [of rubber] without sulphur. W. ESCH (Kautschuk, 1930, 6, 12—14).—Commercial grades of aniline have marked anti-oxidant power, and it is to the presence of aniline that Ostromislensky's products vulcanised with trinitrobenzene owe their good ageing properties. The use of aniline or naphthylamine for this purpose in Ostromislensky's work was not novel. Ostromislensky's more recent results (India-rubber World, 1929, June, p. 54) are critically reviewed. The effect of trinitrobenzene on rubber is probably a partial conversion into *cyclocaoutchouc* products. It is doubtful whether trinitrobenzene will ever displace sulphur as a vulcanising agent; if the price permits, technical use may possibly be made later of trinitrobenzene or similar agents, alone or together with sulphur, for the manufacture of special products such as steam-packing.

D. F. TWISS.

Ageing of vulcanised rubber. VIII. Action of sunlight filtered through coloured glasses on the mechanical properties of vulcanised rubber. T. YAMAZAKI. XI. Oxidation of acetone-extracted vulcanised rubber, and the behaviour of the combined sulphur therein. T. YAMAZAKI and K. OKU; YAMA (J. Soc. Chem. Ind., Japan, 1929, 32, 367—368 B, 368—370 B).—VIII. Exposure of a vulcanised mixture of fine Para rubber (92½%) and sulphur (7½%) to the filtered sunlight showed that the effect of wave-lengths greater than 3300 Å. was serious, but that those greater than 4360 Å. were less effective.

IX. A mixture of rubber and sulphur was successively vulcanised, powdered, extracted with acetone, and then heated for prolonged periods at 70°. The oxidised samples were found to yield soluble sulphur compounds, derived from the combined sulphur, on extraction with acetone; these soluble sulphur compounds contained some sulphuric acid, and the proportion of both was

higher the greater the degree of vulcanisation of the original sample. Although the behaviour of a mixture containing also zinc oxide and diphenylguanidine was similar, the degree of the change was considerably less.

D. F. TWISS.

Thermodynamics of stressed vulcanised rubber.

R. H. GERKE (Ind. Eng. Chem., 1930, 22, 73—77).—Contrary to expectation from the Joule effect, the stress-strain curves produced at the customary rate show vulcanised rubber to be less stiff at higher temperatures. This discrepancy arises from hysteresis effects. By oscillating a strip of rubber loaded with a weight in the neighbourhood of its equilibrium length, it is found that the equilibrium length finally attained is independent of the direction in which it is approached; stress-strain curves so produced are reversible and show the rubber to be stiffer at higher temperatures. The absorption of shocks or vibrations by rubber depends on hysteresis, and the second law of thermodynamics is not applicable. Up to 600% elongation the "equilibrium stress-strain curves" obtained as above, unlike the ordinary stress-strain curves, are identical for different degrees of vulcanisation; this is evidence that vulcanisation does not greatly affect the elastic properties, its most important result being greater resistance to plastic flow or permanent set.

D. F. TWISS.

Sp. gr. and thermal [volume] expansion of the rubber-sulphur system. S. KIMURA and N. NAMIKAWA (J. Soc. Chem. Ind., Japan, 1929, 32, 196—197 B).

—Smoked rubber sheets press-vulcanised with flowers of sulphur at 153° for 30 hrs. were used. The sp. gr.-combined sulphur curve shows a deflection at a point of definite sulphur content which moves in the direction of higher sulphur content as the temperature is increased. The expansion curve shows a similar irregularity in that temperature range where the transition between hard and soft rubber occurs. The deflection is at —85° for crude rubber and shifts towards higher temperatures as the sulphur content is increased, ceasing to shift at about 32% of sulphur. Below the temperature corresponding to the deflection the coefficient of expansion is small; above it is large. The dependence on temperature of the position of the deflection is regarded as evidence against the deflection indicating the existence of a definite compound.

S. K. TWEEDY.

Flexing test for [rubber] tyre carcass stocks.

W. A. GIBBONS (Ind. Eng. Chem. [Anal.], 1930, 2, 99—102).—A strip composed of several plies of fabric and rubber is bent around a hub until its two faces are nearly parallel. A weight is applied to the hub so that the strip is pulled taut. The ends of the strip are then moved reciprocally so that the strip moves to and fro around the hub. The movement is continued until separation occurs between the plies of the strip. Working details are given.

D. F. TWISS.

Estimation of the degree of penetration of rubber

into fabrics. E. O. DIETERICH (Ind. Eng. Chem. [Anal.], 1930, 2, 102—103).—The samples, preferably rubber-coated on both sides, are vulcanised to a hard condition, e.g., by heating in an aqueous solution of sodium or calcium polysulphide containing a water-soluble accelerator. Sections, e.g., 0.05—0.1 mm.

in thickness, are then cut and placed in concentrated sulphuric acid until the cotton fibres have disappeared; after being washed in clean acid they are mounted in the same medium and photomicrographed, *e.g.*, at 35–50 diam. Typical photographs are reproduced.

D. F. TWISS.

PATENTS.

Vulcanisation of rubber. GOODYEAR TIRE & RUBBER Co., Assecs. of J. TEPPEMA (B.P. 300,949, 18.7.28. U.S. 21.11.27).—The products (salts) obtained by mixing (alcoholic) solutions of substituted 2-thiolbenzthiazoles and aliphatic or aromatic amines are used as accelerators. Examples are diethylamine and diphenylguanidine compounds of 5-chloro- and 6-amino-2-thiolbenzthiazoles.

C. HOLLINS.

Manufacture of vulcanised rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 320,699, 26.7.28).—As accelerators are used amines containing an unsaturated alkyl group, *e.g.*, products obtained by action of ammonia or amines on vinyl acetate, butadienes, etc.; the 2-thiolbenzthiazole salts of such amines are particularly suitable. Examples are butenylpiperidine and dibutenylmethylaniline.

C. HOLLINS.

Production of accelerators for the vulcanisation of rubber. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,739,486, 10.12.29. Appl., 1.8.24).—Substituted guanidines, triphenylguanidine in particular, are obtained by heating a thiocarbamide with a primary amine and a mixture of lead carbonate and litharge. The use of lead carbonate instead of litharge alone expedites the reaction and permits a reduction in the proportion of amine necessary. By heating 85 lb. of thiocarbamide with 15 lb. of litharge, 100 lb. of basic lead carbonate, and 36 lb. of aniline, with agitation at 100° for 45 min., and removing the resulting water towards the end of the period, it is possible to obtain a yield of thiocarbamide 90% of the theoretical and of 95% purity.

D. F. TWISS.

Manufacture of organic materials [such as of rubber]. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and A. NIVEN (B.P. 323,755, 6.10.28).—To a quantity of aqueous dispersion, *e.g.*, of rubber, of high viscosity and of natural or artificial colour, is added a smaller quantity of a dispersion or dispersions of comparable viscosity but different in colour. The dispersions may be caused to intermingle by gentle irregular movement of the surface. A mould or former dipped into such a prepared bath acquires a coating with irregular coloured ornamentation; the coating may be stripped subsequently from the mould so as to yield a varicoloured hollow article.

D. F. TWISS.

Manufacture of artificial rubber. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 323,721, 3.10.28).—The products obtained by polymerisation of an aqueous emulsion of a diolefine such as butadiene or isoprene are treated, while still in the emulsified condition or during subsequent mechanical working, with agents having a reducing action or a decomposing effect on oxidising agents. Such treatment yields a paler product and also ensures the decomposition of any residual oxidising agent such as hydrogen peroxide which may have been used to promote the polymerisation process.

Sodium hyposulphite or bisulphite, naphthols, pyrogallol, aniline, and colloidal solutions of metals are amongst those mentioned.

D. F. TWISS.

Buoyant and oil-proof india-rubber. E. YOSHIOKA (U.S.P. 1,744,602, 21.1.30. Appl., 9.5.27).—See B.P. 284,912; B., 1928, 277.

Testing of elasticity (B.P. 306,555).—See I.

XV.—LEATHER; GLUE.

Tanning materials of Central Japan. G. GRASSER (Cuir tech., 1929, 22, 225–230; Chem. Zentr., 1929, ii, 1120).—The fruit of *Quercus castanopsisifolia* contains a pyrogallol tanning substance 25.5%, non-tans 10.1%, and water 10.5%; that of *Q. glandulifera* contains a pyrocatechol tan 5.8%, non-tans 3.1%, water 9.5%, whilst the galls contain 18.4% of a readily extractable pyrogallol tan. The fruit (and leaves) of *Areca catechu*, L., contain, respectively, 11.4 (3.4)% of a tanning material, 10.5 (17.1)% of non-tans, and 6.9 (6.5)% of water. The wood (and bark) of *Castanopsis taiwaniana*, Hayata, contain, respectively, a pyrogallol tan 2.4 (8.5)%, non-tans 1.8 (2.5)%, water 6.7 (7.7)%; those of *C. kawakamii*, Hayata, contain 3.1 (9.5), 1.5 (2.4), 6.9 (8.5)%, respectively. *Acacia confusa*, Merr., contains a pyrocatechol tan 11.0%, non-tans 7.3%, and water 8.7%. The fruit of *Dioscorea rhipogonoides*, Oliv., contains 8.0% of tans and 9.5% of non-tans. (Cf. B., 1929, 950.)

A. A. ELDRIDGE.

“Red heat” in salted hides. (Miss) D. J. LLOYD [with R. H. MARRIOTT and (Miss) M. E. ROBERTSON] (J. Soc. Leather Trades' Chem., 1929, 13, 538–569).—“Red heat” is caused by halophilic bacteria—red or yellow sarcina—brought on to the hide by the salt used in curing. They are particularly common in salts of marine origin occurring in samples of brine from Cheshire and Worcester, a Cheshire rock salt, and in large quantities in a salt from an Argentine hide-curing establishment. Gelatin was not liquefied by the red sarcinae, but was liquefied by yellow sarcinae, and there was evidence that greater damage was done to the hide by mixed cultures than by the yellow organisms alone. The yellow organism grew more rapidly than the red, growth being stimulated by higher temperatures up to 37° and by more humid atmospheres. Their growth was checked by additions of sodium bisulphate or bisulphite to the curing salt. The effect of these additions on the structure of the hide and the weave of the fibres has been investigated, and a number of photomicrographs are given to show that the limed hide obtained from salting with brine liquor containing sodium bisulphate was equal to that obtained by using saturated brine liquor only. The fibres were too much separated in the limed pelt obtained from a hide, which had been cured with a brine liquor containing sodium bisulphite, although the latter was most effective in destroying bacteria. Disorganised fibre weaving was apparent in a limed hide which had been obtained from a hide cured with a brine containing more than 2% of soda ash, although a brine containing 1% of soda ash was free from this objection. These amounts of soda ash are insufficient to destroy the infection. The best results were obtained with a hide cured with common salt mixed with 0.25% of sodium bisulphate. D. WOODROFFE.

Raw hide. G. GRASSER (Cuir tech., 1929, 22, 202—203; Chem. Zentr., 1929, ii, 1117).—The ash (1.7—37%; the average natural mineral content of the hide is 2%) contained CaSO_4 0.2—3.9, MgSO_4 0—6.6, Na_2SO_4 0—16.6, NaCl 1.2—20.5, SiO_2 0.3—7.7, ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) 0.2—4.1. The natural fat content is 0.5—2%; 13—20% has been found in Chinese hides. The water content of the dry hide varies between 13 and 20%. A. A. ELDRIDGE.

Biochemistry of soaking and liming [of animal skins]. IV. Influence of gaseous environment on liming. E. R. THEIS and J. M. MILLER (J. Amer. Leather Chem. Assoc., 1930, 25, 2—15; cf. B., 1929, 613).—Pieces of hide were limed in atmospheres of air, nitrogen, oxygen, and hydrogen, and *in vacuo*; the nature of the gaseous products and composition of the lime liquors were determined each day. Most ammonia was produced in an atmosphere of nitrogen, and least *in vacuo*. Hair-loosening was retarded by passing air into the lime liquors for 1 hr. daily, but was accelerated by passing hydrogen. The total dissolved nitrogenous matter was decreased by passing the gases through the lime liquors, oxygen, hydrogen, and air causing the largest decreases. Decomposition of the dissolved protein matter occurred in increasing amounts as the current of oxygen was continued through the lime liquors. The nitrogen and sulphur decompositions were equal. Very little volatile acid was found in the lime liquors, indicating that the liming process is not a deaminising one. The greatest hydration of the hide occurred when the lime liquor was continuously agitated. The effect of the gases used was chemical, causing 10—15% increase in the ammonia produced. The evolved sulphur was in a reduced form in the case of hydrogen and nitrogen, indicating a strongly reducing medium.

D. WOODROFFE.

PATENTS.

Tanning of animal hides. H. RHEINBOLDT and H. BREUER (G.P. 453,534, 13.2.26).—Hide pelts are pretreated with a protein precipitant, *e.g.*, copper salts, chrome alum, formaldehyde, phenols, tanning materials, and then treated with a dilute alkaline or neutral solution of natural or synthetic humins, which are fixed in the leather by subsequent treatment of the latter with acids and/or salts; alternatively, the hide pelts may be treated with alkaline or neutral solutions of humic acid, to which such protein precipitants have been added.

D. WOODROFFE.

Manufacture of leather. C. HÖPER (G.P. 457,818, 30.5.26).—Pelts are treated first with a solution of sodium thiosulphate, then with a liquor containing potassium dichromate, ferrous and zinc sulphates, and hydrochloric acid, and finally with a solution of sodium carbonate.

D. WOODROFFE.

Manufacture of black or coloured upper leather. H. WELTI (Swiss P. 121,819, 13.2.26).—Wet mineral-tanned or partly mineral-tanned hides or skins are completely impregnated with an emulsion of a high-polishing wax or substance containing it, *e.g.*, montan wax, beeswax, carnauba wax, or walrus oil. Leather so treated can be tanned, fat-liquored, and dyed in the usual way. A high glaze is produced on the surface by brushing.

D. WOODROFFE.

Treatment of leather and production of leather articles. D. M. STRAUCHEN, Assr. to RITTER DENTAL MANUF. CO., INC. (U.S.P. 1,738,934, 10.12.29. Appl., 12.11.26).—Leather, preferably rough-tanned, is impregnated with a suitable softening liquid, *e.g.*, acetone, after which it may or may not be flexed and/or moulded, the liquid allowed to evaporate, and the product subsequently impregnated with a liquid or wax composition, *e.g.*, beeswax, carnauba, montan, and paraffin wax, which will harden it and maintain the shape.

D. WOODROFFE.

Making emulsions (B.P. 323,534).—See I. **Sulphonated aliphatic compounds** (G.P. 454,458).—See III. **Artificial masses** (B.P. 298,135).—See XIII.

XVI.—AGRICULTURE.

Hydrogen and hydroxyl ions in the ionic layer of suspended particles and dispersed ultramicros. G. WIEGNER and H. PALLMANN (Trans. 2nd Comm. and Alkali Sub-Comm. Internat. Soc. Soil Sci., 1929, B, 92—144).—Suspended clay and permutit particles retain hydrogen ions in the outer ionic layer. These are active towards an immersed electrode. Sedimentation of the particles removes hydrogen ions from the effective sphere of the electrode and higher p_H values are thereby recorded. The p_H of the centrifuged dispersion medium has a constant value. In dilute suspensions changes in recorded p_H values are directly proportional to the number of particles suspended. Alkaline solutions added to clay suspensions cause an initial increase in hydrogen-ion concentration due to increased dispersion, followed by normal neutralisation. Addition of neutral salt solutions to acid clay suspensions causes replacement of hydrogen in the outer layer by sodium. The hydrogen-ion concentration of the dispersing medium increases and the potential acidity of the particle decreases. Electrometric measurements of the apparent p_H of the suspension are confirmed by sugar inversion methods. The effect of the ionic layer surrounding the colloidal particle is independent of the density of the layer and of its size or form. Following the formation of porous aggregates during the coagulation of clay, sugar inversion methods indicate a higher hydrogen-ion concentration than does the electrometric process. The latter depends on external surface ions only, whereas sugar molecules penetrate within the aggregate. Differences in results of the two methods give information as to total exposed surface of the particles and therefore of soil texture.

A. G. POLLARD.

Buffering of soil silicates. W. U. BEHRENS (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 95).—Buffer curves of soil silicates show two maxima and differ from those of artificial permutits. A. G. POLLARD.

Capillary phenomena and the heterogeneity of the soil. J. H. ENGELHARDT (Proc. Internat. Soc. Soil Sci. [Soil Res. Suppl.], 1929, 4, 239—301).—The regulation of water movements in soils by capillary forces is discussed. Numerous records of capillary pressure measurements by means of capillarmeters show the relationship between capillary pressure, size of particles, and depth of water table.

A. G. POLLARD.

Chemical nature of soil organic matter, methods of analysis, and the rôle of micro-organisms in its

formation and decomposition. S. A. WAKSMAN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 172—197).—A comprehensive and critical survey of the literature on this subject. A. G. POLLARD.

Methods of determining humus [in soils]. L. KOTZMANN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 198—203).—Experimental comparison of existing methods for humus determination shows considerable lack of agreement in results. A. G. POLLARD.

Rôle of sulphur in the formation of arable land. L. RIGOTARD (Compt. rend., 1930, 190, 199—201).—Not only does the presence of sulphur lead to the disintegration of the rocks, but an analysis of 19 earths reveals the fact that a high sulphur content is complementary to a high nitrogen content (humus formation). C. C. N. VASS.

Effect of frost on arable soils. A. VON NOSTITZ (% Pflanz. Düng., 1930, 15A, 273—279).—Prolonged and severe frost did not greatly alter the aggregate structure of soils, though the degree of flocculation changed perceptibly. Soils poor in lime tended to become slightly acid. The absorbing capacity for acid increased in some soils, but the base-absorbing capacity increased in nearly all cases. The catalytic power of soils was not greatly affected. The lighter colour of some soils after freezing was probably due to increased flocculation. Improved fertility in frosted soils is in part the result of mineral disintegration, but is principally caused by changes in physical conditions, leading to improved aeration, more rapid warming, ease of root penetration, and accelerated microbiological activity, together with abnormal precipitation of dust containing nitrogenous compounds. A. G. POLLARD.

Alkali soils and soil reclamation. A. A. J. VON SIGMOND (Mezőg.-Kutat., 1929, 2, 272—292; Chem. Zentr., 1929, ii, 1578).—The causes of the formation of Hungarian alkali soils are discussed. The first phase consists in an accumulation of sodium chloride, sulphate, carbonate, and hydrogen carbonate; in the second phase base exchange leads to transference of sodium to the absorption complex; in the third hydrolysis occurs; in the fourth sodium is replaced by hydrogen. Alkali soils of the last two phases can be improved by liming. A. A. ELDRIDGE.

The C:N ratio in various Hungarian soils. K. PÁTER (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 204—206).—In the soils examined the C:N ratios varied from 8.6 to 13.7, with the exception of highly acid samples. High humus contents were associated with high C:N values. In soils of similar physical and chemical composition variations in C:N ratios were within restricted limits. This is probably the outcome of similar biological activities. A. G. POLLARD.

Microflora of leached alkali soils. II. Leached sodium chloride soil. J. D. GREAVES (Soil Sci., 1930, 29, 79—83; cf. B., 1930, 72).—Physiological and morphological characteristics of the microflora are recorded. A. G. POLLARD.

Amelioration of lime- and soda-containing Szik soils. A. HERKE (Trans. 2nd Comm. and Alkali Sub-Comm. Internat. Soc. Soil Sci., 1929, B, 184—195).—Alkali soils of the solonetz and solontschak types are

described. Treatment of the latter with gypsum, bauxite, iron and aluminium sulphates, calcium chloride, or sulphur decreases the p_H values and improves the physical condition, but in the first year of treatment plant injury may be increased. This is ascribed to the liberation of adsorbed sodium from the clay complex, with a consequent temporary increase in the soluble sodium content of the soil. After leaching by one season's rain, crop yields increase beyond those of untreated soils. Accumulations of sodium salts at depths of 40 cm., even when the top soil is normal, affect plant growth in dry warm seasons. Addition of farmyard manure increases the ameliorative value of the above materials. A. G. POLLARD.

Effect of absorbed ions on soil reaction. B. AARNIO (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 98—100).—The p_H values of clays treated with various electrolytes are determined. Hydrogen- and aluminium-clays have acid characteristics, but all others are neutral or slightly alkaline. Leaching with neutral salt solutions decreases the acidity of acid soils. Salts of univalent elements are more effective in this respect than those of bivalent elements. A. G. POLLARD.

Soil acidity and soil adsorption. D. J. HISSINK (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 111—124).—Comparison is made of the lime requirement (Kappen), the "lime factor" (lime required to give p_H 7.0), and the value S (Hissink). Soils treated with lime according to Kappen's values have approximately the same V values, but lower than the V value reached in soils limed to p_H 7.0. The buffer capacity (lime required to change p_H by 0.1) per unit humus content was independent of the actual humus content of the soils examined. In humus soils the buffer capacity increased as the neutral area was approached. In Kappen's method for determining hydrolytic acidity the soil sample is preferably reduced to 25 g. and the reaction period increased to 3 hrs. In di Gléria's method for determining exchangeable bases the treatment with barium chloride displaces hydrogen and aluminium ions from the absorbing complex, in addition to the usual cations examined. This leads to higher S values than those obtained by Hissink's method. In neutral and weakly alkaline soils, some barium ions may be removed from the clay complex during washing with water (di Gléria and Bobko-Askinasi methods). Tentative methods for determining S values in saline soils are described. Lime requirements to produce p_H 5.0 in soils, as calculated from hydrolytic acidity values (Kappen and Kutchinsky), are of doubtful value for weakly acid and alkaline soils. A. G. POLLARD.

Soil acidity and absorption. R. GANSEN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 46—47).—Among the exchangeable bases of soils the proportion of calcium varies considerably. Determinations of the degree of saturation of soils must include separate and direct determinations of each of the bases present. In Hissink's method (V value), titration with lime or baryta solutions does not necessarily indicate the saturation point. The additional base absorbed is not independent of the nature and distribution of bases

already present. The methods of Kappen (calcium acetate) and of Hutchinson and McLennan (calcium bicarbonate) are most suitable for determining the saturation condition of soils. A. G. POLLARD.

Soil acidity and absorption. O. LEMMERMANN and L. FRESSENIUS (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 36—43).—For determinations of p_H values soil suspensions should be used, and in the case of sandy soils and light loams a soil : water ratio of 1 : 1 or 1 : 0.5 is most satisfactory. Measurements should be made 15—20 min. after mixing fresh soil and water, and only for exceedingly dry soils should this period be extended. With the quinhydrone electrode equilibrium is not always instantaneous. Excess of quinhydrone and a narrow soil : water ratio lead to more rapid equilibrium. Values obtained in aqueous suspensions are dependent on electrolytes present at the time of sampling, but those of potassium chloride suspensions are characteristic of more permanent soil conditions. Both determinations should be made. Daikuhara's method for determining lime requirement yields uncertain results. Calculated values from soil-titration curves are preferable to those calculated from the hydrolytic acidity (Kappen). Pot experiments show the lime requirement to be less than 3 times the titration value (Christensen). Hissink's method is too lengthy for general application, and is restricted to carbonate- and gypsum-free soils. The view that optimum growth conditions in soils corresponds to 70% saturation with bases (Gehring) is not confirmed. A. G. POLLARD.

Dehydration and soil acidity. H. G. COLES and C. G. T. MORISON (Soil Sci., 1930, 29, 59—70).—Heating of soils to 98° decreased their p_H values. In peat soils little change occurs until nearly all the water has been removed, but in mineral soils the effects were more gradual. The changes were reversible in mineral soils, but not in organic ones. The p_H of soils from which exchangeable bases were removed were not appreciably altered by heating. Drying decreased the amount of exchangeable bases in soils, and increased the water-soluble calcium, potassium, and phosphate. The extent of the p_H changes on drying were more closely related to the original p_H than to the clay contents of the soils. A. G. POLLARD.

Fixation and mobilisation of phosphoric oxide in clays. A. DEMOLON and G. BARBIER (Compt. rend., 1929, 189, 1310—1312).—When decalcified clay is treated with aqueous ammonium dihydrogen phosphate solution an equilibrium is reached between the solid and liquid phases; the amount of phosphate fixed by the solid rises with increasing initial concentration of the phosphate solution, and is a maximum at about p_H 3.5. The ferric oxide present in brick-clay is responsible for the fixation; the phosphate can be subsequently removed by shaking with 0.5% sodium hydroxide solution. Adsorbed calcium in clay favours the adsorption of phosphoric oxide, but if the phosphate solution contains an excess of calcium ions, the adsorption is not favoured. If the acids commonly used (i.e., 1% acetic, 2% citric, and dilute nitric) for the determination of the total phosphoric oxide in soils

contain above a certain amount of phosphate, the soil may actually adsorb phosphate from them.

H. BURTON.

Correlation between the fineness and carbon solubility of calcareous grindings and their neutralising action on acid soils. C. BRIOUX and E. JOUIS (Compt. rend., 1930, 190, 277—280).—The rate of neutralisation of acid soils by these products when they were added at the rate of 1.666 g. CaCO_3 per kg. was measured from the change in p_H after intervals of from 15 hrs. to 15 days. It bears a close relationship to the fineness as determined by means of standard sieves, and to the solubility in water saturated with carbon dioxide, and these two properties provide a rational means of evaluation. J. GRANT.

Regularity of the absorption process in the determination of hydrolytic acidity [in soils]. A. VON RÄTH (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 77—83).—Hydrolytic acidity values as determined by treatment of soils with calcium acetate solution vary with the proportion of soil to water used. The process of absorption of calcium by soils is examined by successive leachings with calcium acetate solution. The nature of the absorption curves may serve as a basis for the grouping of soils according to type. The Kappen factor (3) used in calculating the lime requirement of soils from their hydrolytic acidity is not universally applicable. For each group of soils a characteristic factor is necessary. A. G. POLLARD.

Electrodialysis and mineral soil acidity. M. TRÉNEL (Trans. 2nd Comm. and Alkali Sub-Comm. Internat. Soc. Soil Sci., 1929, B, 144—153).—Electrodialysis of permutit indicates the gel to be a mixture of variable composition without the characteristics of the supposed "permutit-acid" and without exchangeable hydrogen ions. Treatment of electrodialysed permutit with neutral salt solution results in the liberation of iron and aluminium from the complex, according to the equation: $\text{Fe}[\text{Al}](\text{OH})_3 + 3\text{KCl} \rightleftharpoons 3\text{KOH} + \text{Fe}[\text{Al}]\text{Cl}_3$. The presence of silica with which the alkali can combine favours the direct equation, and the formation and hydrolysis of the ferric salt produces an acid reaction. Ferric ions appear in water in which electrodialysed "iron silicate gel" is suspended, and the effect is increased by the addition of neutral salts. Exchange acidity in soils is not the result of direct displacement of hydrogen ions. Permutit from which bases are completely removed cannot be regenerated at ordinary temperatures. Purified silica gel cannot effect neutral salt decomposition. A. G. POLLARD.

Indicators used in determining the exchange acids (easily soluble acids in soil) by Daikuhara's method. S. GOY, P. MÜLLER, and O. ROOS (Z. Pflanz. Düng., 1930, 15A, 233—236; cf. B., 1929, 787).—Comparison is made of the use of methyl-red and phenolphthalein (both cold and boiling) in titrating the soil extract in Daikuhara's process. No soil examined which showed p_H (potassium chloride extract) less than 5.3 exhibited exchange acidity when methyl-red was used, but with phenolphthalein exchange acidity was indicated with p_H values up to 5.5. In these cases

heating the solution for titration affected the soda consumption to a small but irregular extent. Generally, hot solutions required more alkali than cold, but both series of figures were consistently higher than when methyl-red was used. The presence of phosphates and aluminium salts explains these differences. Titration values with methyl-red are in close agreement with electrometric measurements. A. G. POLLARD.

Liming as a factor in the mobilisation of phosphoric acid in podsols. D. L. ASKINASI and S. S. JARVASOV (Z. Pflanz. Düng., 1930, 15A, 218—233).—Field trials are described showing the effect of liming on the assimilable phosphate of soils and on crop yields. Lime brings into solution the easily decomposable humus which is rapidly mineralised, producing available phosphate and nitrogen. The effect is most marked in the first year of application, and slowly decreases as time proceeds. Besides increasing the availability of soil nutrients and reducing soil acidity, lime disturbs the podsolisation process, and hinders the natural decrease in soil fertility. A. G. POLLARD.

Effect of phosphate and lime on the rate curve of solubility of phosphorus from a Wooster silt loam soil. R. H. SMON (Soil Sci., 1930, 29, 71—78).—The solubility of soil phosphates is examined by means of "rate curves" (cf. Vanstone, B., 1925, 1002). The utilisation of these curves for ascertaining the probable response of soils to fertilisers is suggested. A. G. POLLARD.

Fixation of phosphate by a peat soil. J. L. DOUGHTY (Soil Sci., 1930, 29, 23—35).—Both physical adsorption and chemical precipitation are factors in the fixation of phosphates by peat, although at $p_H > 2$ the formation of phosphates of iron, aluminium, and calcium offer adequate explanation of observed facts. Removal of these bases from peat by acid treatment decreases the phosphate-fixing power at $p_H > 5$; the artificial addition of bases has the reverse effect. Precipitation of calcium phosphate in the presence of peat is possible only from water-soluble calcium salts, adsorbed calcium ions being inoperative. Solubility- p_H curves of ferric phosphate were similar to those obtained when soluble phosphates were added to peat pretreated with ferric chloride. Cations present in peat react with soluble phosphates in the same manner as do the pure salts in the absence of peat. A. G. POLLARD.

Decomposition of crude phosphates by peat and the influence of neutral salts on this process. S. N. ROSONOV (J. Landw.-Wiss., Moscow, 1926, 3, 751; Bied. Zentr., 1930, 59, 12).—The percentage decomposition of rock phosphates by peat increases with the proportion of peat to phosphate used. Gypsum and calcium and potassium chlorides have no influence on this decomposition. A. G. POLLARD.

Influence of silicic acid on the resorption of soil phosphoric acid. A. NĚMEC (Z. Pflanz. Düng., 1930, 15A, 258—262).—The assimilation of phosphate by seedlings in Neubauer's process was greatest in soils having the highest proportions of soluble silica. Further Neubauer tests with soils treated with sodium silicate showed this compound to have caused a larger intake of phosphorus by the plants and to have increased

their ash content. These effects were greatest in soils having originally the lowest soluble silica contents.

A. G. POLLARD.

Determination of (A) the degree of unsaturation, (B) the exchangeable bases, of soils. J. DI GLÉRIA (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 58—64, 64—69).—(A) The absorption of bases by soil is both chemical and physical. The absorption of baryta by carboraffin, active silica, and blood-charcoal agrees closely with the van Bemmelen adsorption equation $x = Kc^{1/q}$. The experimental values for K and $1/q$ for different soils examined varied from 15 to 45 and from 0.142 to 0.254, respectively. The fixation of ammonia from aqueous solution is the basis of the method described for determining chemical absorption by soils. The unsaturation of soils so determined differs considerably from that obtained by the methods of Hissink and of Kappen. (B) In the Bobko-Askinasi method for determining the total exchangeable bases in soils the removal of barium chloride is more complete than in Kelly's method (using ammonium chloride) since peptisation of the soil is greater in the latter case. An improved process consists of repeated treatment of the soil with hot N -barium chloride and filtration until a litre of filtrate is obtained. In the filtrate the exchangeable bases (except calcium) are determined, calcium being calculated by difference. The residual soil is washed on the filter till free from barium, then with chlorine-free nitric acid to remove all adsorbed barium, which is determined in the filtrate. In chalky soils the nitric acid is replaced by chlorine-free sodium nitrate solution. Soils containing chalk or soluble salts yield falsely high values which must be corrected. Bases present as carbonates are determined by distillation with ammonium chloride, the ammonia liberated being collected in standard acid. In soils containing soluble salts displacement is carried out with a neutral salt the anion of which differs from those of the soil salts. Then exchangeable bases = total leached bases — base equivalent of anions of soluble soil salts.

A. G. POLLARD.

Simple two-compartment electro dialysis cell for the determination of exchangeable bases [in soils]. E. M. CROWTHER and J. K. BASU (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 100—102).—An alundum thimble containing the soil rests on a nickel or copper cathode, an anode of platinum or gold being arranged just above the soil surface. The whole is placed in a glass vessel having a constant water-level adjustment. Exchangeable bases determined by means of the apparatus agree well with those obtained by displacement with ammonium chloride solution.

A. G. POLLARD.

The quinhydrone and indicator methods for determining p_H values of soils: their comparison, and tests of their utility. S. KÜHN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 125—132).—Apparent increases in the p_H values of soils during the period of examination by the quinhydrone electrode are not proportional for all soils. Comparison is made in the values obtained with indicators. True soil solutions are much more weakly buffered than the corresponding suspensions. Use of small amounts of quinhydrone

leads to best results by the electrode method, but time variations are still irregular. The indicator method is preferred.

A. G. POLLARD.

Error in soil reaction determinations by the quinhydrone method. S. G. HEINTZE and E. M. CROWTHER (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 102—111).—With certain soils the quinhydrone electrode indicated much higher p_H values than the hydrogen electrode. This may be due to the interaction with quinhydrone of an active form of manganese dioxide present in these soils. The resulting production of manganous oxide leads to partial neutralisation of the soil acids.

A. G. POLLARD.

Determination of the unsaturation and lime requirement of soils from their hydrolytic acidity. J. VON CSIKY (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 84—94).—The lime required to neutralise hydrolytic acidity is determined by direct treatment of the soil sample with lime water and subsequent titration of the filtered liquid. Values so obtained are compared with those by Kappen's method and with field trials. The author's method gives higher values than Kappen's, but no direct relationship between them is apparent. By increasing the proportion of calcium acetate solution to soil in Kappen's method, the hydrolytic acidity values increase steadily to a stationary figure. The relationships between these maximum values and other observed data are discussed with a view to their utilisation in lime-requirement determinations.

A. G. POLLARD.

Determination of the amount of lime which a soil can absorb under prescribed conditions, particularly in Kappen's method. J. VAN DER SPEK (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 69—77).—The effect of varying the proportion of soil to calcium acetate solution, and the period of contact, on the observed absorption of calcium in Kappen's method is considerable, and increases with the proportion and degree of unsaturation of the clay and humus fractions present. The use of 25 g. of soil with 250 c.c. of *N*-calcium acetate is recommended. Using these proportions, a leaching process is developed for determining the lime required to bring the p_H value of an acid soil to 6.6—7.0. No arbitrary factor is necessary.

A. G. POLLARD.

Reaction, lime content, and lime requirement of soils. J. LENDE-NJAA (Nord. Jordbrugsforsk., 1926, 412; Bied. Zentr., 1930, 59, 3—4).—In uncultivated moor soils lime content and p_H value are closely related, but in cultivated areas the p_H values are relatively lower. Moor soils with high ash content have higher p_H values than those with less ash. In mineral soils these relationships are less marked. The lime content corresponding to a definite p_H value is less in mineral than in moor soils. Plant growth (especially of wild plants) is closely dependent on soil reaction. Double maxima in growth- p_H curves (Arrhenius) were not observed. For the calculation of lime requirements from soil titration curves, the latter should be determined with lime water, since soda-titration curves are not identical. Chalk and quicklime yield similar titration curves up to p_H 7.0. Beyond this point lime values rise

rapidly to p_H 12.0, whereas with chalk a maximum of p_H 8.3 is reached slowly, the rate varying with the carbon dioxide content of the soil. In partially dried or aerated soils the p_H values are generally lower than those of fresh samples.

A. G. POLLARD.

Reaction and lime requirement of Hungarian soils. A. A. J. VON SIGMOND (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 43—46).—Methods for determining soil reaction and lime requirement are compared. Calculation of lime requirement from buffer capacities of soils is unsatisfactory. The azotobacter test records some soils with p_H 7.3 to be deficient in lime.

A. G. POLLARD.

Determination of the lime requirement of soils and the lime absorption in acid humus soils. S. TOVBORG-JENSEN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 47—58).—The Christensen-Jensen method (B., 1927, 309) is applicable to humus soils if the "limiting factor" of 2.9 is used. In field trials, in which varying amounts of lime had been added to the soil, the p_H values of the samples taken six months after the application were of the order anticipated by the titration curves. The increase in lime requirement after 2½ years was approximately the same for all plots, irrespective of the amount of lime originally added. At this period the ratio of the exchangeable calcium present to the amount of lime originally added was the same throughout the series, viz., 1:3.

A. G. POLLARD.

Determination of the reaction condition and lime requirement of soils. A. GEHRING (Z. Pflanz. Düng., 1930, 15A, 196—212).—Existing methods for determining the lime requirement of soils are discussed together with their application to different soil types. In general, insufficient attention is given to the complete amelioration of the physical properties of soils by lime. On sandy soils the Gehring-Wehrmann method yields higher values than do others based on measurements of hydrolytic acidity. On humus soils values are erratic. In field trials soils limed to the full Gehring-Wehrmann value yielded heavier crops than when only one half of this amount was used, and these in turn exceeded those in which the lime application was based on hydrolytic acidity. In sandy soils no relationship existed between the p_H of the potassium chloride extract and the degree of saturation with bases, and p_H 7.0 does not correspond with 70% saturation (Hissink). In clay soils p_H and saturation agreed more closely, but at p_H 7.0 the degree of saturation frequently exceeded 70%. Hydrolytic acidity and p_H values were closely parallel. Details of new and amended methods for determining the degree of saturation of soils with calcium, and their hydrolytic acidity, are described.

A. G. POLLARD.

Acidity, degree of saturation, and lime requirement of various soils, based on pot experiments. D. MEYER, P. OBST, and F. WILCZEWSKI (Z. Pflanz. Düng., 1930, 15A, 279—296).—Pot experiments with soils receiving varying proportions of lime are described. The maximum saturation of soil with exchangeable bases corresponded with an alkaline reaction. The amount of lime required to neutralise hydrolytic acidity was in some soils greater, in others less, than that necessary to produce 70% saturation (Hissink). The

amount of lime necessary to produce maximum crops, whether calculated from the exchange acidity or degree of saturation with bases, varied with the individual crop and soil type.

A. G. POLLARD.

Determination of the lime requirement of acid soils on the basis of laboratory and vegetation experiments. D. J. HISSINK (*Z. Pflanz. Düng.*, 1930, 15A, 296—300).—Kaufmann's results (B., 1928, 937) are criticised.

A. G. POLLARD.

Methods for determining the available potassium of soils. L. C. WHEETING (*Soil Sci.*, 1930, 29, 1—21).—Existing methods for extracting the available potash from soils were examined. Neubauer tests accurately determine potash deficiencies in soils, and, when fertilised soils are used, give reliable indications of the available potash contents. Among chemical solvents, water and carbon dioxide were not satisfactory in removing available potash from soils. Citric acid solution (1%) used in the normal manner or in conjunction with steam and pressure did not sufficiently differentiate between rich and poor soils. The closest approximation to plant activity is shown by *N*-ammonium chloride, which gives better results by continuous leaching than by single-extraction methods. The rate of solubility of soil potash in leaching experiments with ammonium chloride is at first uniform and subsequently decreases gradually. The total available potash in a soil may be calculated from the amount removed in the first 200 c.c. of leachate. The solubility constants of potash under these conditions are similar for all soils examined. The easily available potash is considered to be the replaceable potash of soils.

A. G. POLLARD.

Significance of the degree of saturation of soils with potash in evaluating their potash requirement. A. GEHRING and O. WEHRMANN (*Z. Pflanz. Düng.*, 1930, 15A, 213—218; cf. B., 1929, 335).—Field trials confirm the value of the authors' laboratory method (potash required = % saturation with potash \times % saturation with lime) and also of the "relative Neubauer value." In the application of the Gehring method to soils containing more than 3% of humus, the leaching of potassium salts with sodium chloride solution is slow and inaccuracies may occur. In black-earth soils the potash saturation value recorded increases with the proportion of soil used for analysis. In rich humus soils of other types the effect of the humus content on the values obtained is less marked.

A. G. POLLARD.

Significance of ionic density in the regular and quantitative relationships between degree of acidity, adsorptive capacity, and buffer power of soils, and a comprehensive method for determining the latter factor. S. Goy (*Z. Pflanz. Düng.*, 1930, 15A, 237—248).—Acidity, adsorptive capacity, and buffer power of soils are closely interrelated and may be measured on a basis of the "ionic density," i.e., the ratio of the amount of base required to neutralise a definite degree of acidity, to that which a soil adsorbs in attaining a neutral reaction. The application of the author's electrometric method (B., 1929, 787) to the measurement of the above values is described.

A. G. POLLARD.

Determination of the fertiliser requirement of

soils. J. KÖNIG and J. HASENBÄUMER (*Trans. 2nd Comm. Internat. Soc. Soil Sci.*, 1929, A, 142—146).—Working details of the authors' method are given and results compared with other well-known processes. The method is not applicable to highly acid soils nor to those having more than 2% of chalk. With soils of high manganese content the citric acid extract must be evaporated with hydrochloric and nitric acids.

A. G. POLLARD.

Laboratory methods for determining the fertiliser requirement of soils. A. A. J. VON SIGMOND (*Trans. 2nd Comm. Internat. Soc. Soil Sci.*, 1929, A, 147—159).—Comparison of the results of the author's method with the Azotobacter method (Niklas) and Neubauer's method shows a general similarity, but a divergence in detail. The three methods supplement one another in a general characterisation of a soil. In view of variations in the assimilability of soil phosphates with soil reaction, the selected limiting values for calculating fertiliser requirements must be flexible. The use of nitric acid for the extraction of assimilable potash is preferable to citric acid, since the latter is too weak to remove all exchangeable bases from the clay complex.

A. G. POLLARD.

Comparative tests of Swedish soil for available phosphoric acid. C. DREYSPRING and C. KRÜGEL (*Superphosphate*, 1929, 2, 121—133, 141—151, 161—168).—Mitscherlich's and Neubauer's methods gave parallel results. Barley is not a suitable plant for use in determining the phosphate requirement of soils.

CHEMICAL ABSTRACTS.

Determination of the phosphate requirement of soils by the Lemmermann-Fresenius citric acid method. O. LEMMERMANN (*Trans. 2nd Comm. Internat. Soc. Soil Sci.*, 1929, A, 135—142).—The importance of a consideration of the total content of phosphorus in soil and its relative solubility is emphasised, and the impossibility of assigning definite limiting values for these figures explained. Among recognised methods for determining the phosphate requirement of soils the author's method agrees most closely with that of Neubauer. The citric acid method is more rapid, more certain, and of more general application.

A. G. POLLARD.

Determination of the potash and phosphate requirement of soils by the seedling method. H. NEUBAUER (*Trans. 2nd Comm. Internat. Soc. Soil Sci.*, 1929, A, 159—170).—Working details and "limiting values" for various crops are recorded. Seeds are preferably dipped in a 0.1% solution of mercury chlorophenoxide.

A. G. POLLARD.

Mechanism of assimilation of nutrients by plants. V. V. BUTKEVITSCH (*Landw. Jahrb.*, 1929, 69, 521—540; *Chem. Zentr.*, 1929, ii, 1547).—Experiments with a collodion membrane and with oats and buckwheat show that with increasing hydrogen-ion concentration the anions, and with diminishing hydrogen-ion concentration the cations, diffuse more readily. Acidification of a flowing nutrient solution increases nitrogen hunger with ammonium as source of nitrogen. If phosphorus is absent an alkaline reaction is injurious. With lack of potassium the yield of oats is higher for alkaline than for acid reactions.

A. A. ELDRIDGE.

Vegetative trials of various forms of nitrogen [fertiliser]. F. KELLER (Landw. Jahrb. Schweiz, 1928, 42, 240; Bied. Zentr., 1930, 59, 18).—Swiss fertiliser trials are described. Calcium nitrate is practically as efficient as sodium nitrate and ammonium sulphate. Synthetic nitrates should not contain more than 1% of nitrite. Plant injury by granulated cyanamide is probably due to the presence of dicyanodiamide, and ill-effects on germination are traced to cyanamide and free ammonia. A. C. POLLARD.

Fertiliser trials with sugar cane in Natal. JERMITSZ (Ernährung der Pflanze, 1929, 3; Bied. Zentr., 1930, 59, 15–18).—Numerous fertiliser trials on various soil types are recorded. Phosphate manuring is of principal importance in the sugar-cane culture in this area. A. G. POLLARD.

Production of nitrate in soil. S. LIMBACH (Zentr. Bakt. Par., 1929, II, 78, 354–375; Chem. Zentr., 1929, ii, 1578).—Other measurable factors being constant, nitrification is subject to periodical variations, increasing in spring and autumn and diminishing in summer and winter. Air-dried soil behaves otherwise. The cause of the variation is discussed. A. A. EDLIDGE.

Can nodule bacteria of leguminous plants fix atmospheric nitrogen in the absence of the host? F. E. ALLISON (J. Agric. Res., 1929, 39, 893–924).—There is little justification for the generally accepted view that legume-nodule bacteria can fix small amounts of nitrogen when grown independently of the host plant. Investigations over a period of 5 years, using 31 strains of legume-nodule bacteria, grown in a very large number of media under a variety of culture conditions, in no case showed that *Rhizobia* could fix atmospheric nitrogen when grown apart from the host. E. HOLMES.

Can *Bacterium radiculicola* assimilate nitrogen in the absence of the host plant? M. P. LÖHNIS (Soil Sci., 1930, 29, 37–57).—Examination of the growth of various strains of *B. radiculicola* in a number of media failed to show any fixation of nitrogen in the absence of a host plant. Positive results reported are due to losses of nitrogen from the nutrient media. A. G. POLLARD.

Effect of various fumigants on the germination of seeds. H. D. YOUNG (J. Agric. Res., 1929, 39, 925–927).—Seeds of 13 crop plants enumerated may be fumigated with *tert.*-butyl chloride, isopropyl formate, ethylene dichloride, and trichloroethylene, in concentrations up to twice the minimum lethal concentration required to kill rice weevils (*Sitophilus oryza*), without seriously impairing the germination of the seeds. Ethylene oxide and methyl chloroacetate seriously impair germination of the same seeds. E. HOLMES.

Biological and chemical examination of stall manures. G. RUSCHMANN (Zentr. Bakt. Par., 1929, II, 77, 216–239; Bied. Zentr., 1930, 59, 21–24. Cf. B., 1929, 410).—The decomposition of various forms of stall manure in soil is examined by means of the carbon dioxide produced. Fresh cold-fermented manure and deep stall manure were the most rapidly decomposed, “edelmist” irrespective of its age was less active, whilst

warm-fermented manure and yard manure were the least rapidly affected. Differences in the rate of carbon dioxide production were most pronounced during the first week, and tended to become more uniform later. In spite of thorough aeration in cylinders, decomposition was not in any case completed in 3 months, and in the case of “edelmist” continued for nearly a year at a rate which was characteristically uniform. The number and nature of the micro-organisms present in the manure were without influence on the rate of decomposition. The steady transition of organic matter into humus in “edelmist” is an important point in its evaluation. On a dry-matter basis the proportion of humic acids increased by 10% in 1½ yrs. Temperature of fermentation is the chief factor in determining the conservation of easily decomposable matter in manure. Hot fermentation for even a short period causes very considerable losses in such material. Loss of easily fermentable substances is paralleled by increased numbers of thermophilic organisms. There is a significant decrease in bacterial numbers at temperatures near 30°, although the rate of decomposition continues to increase.

A. G. POLLARD.

Fertilising effect of manures containing magnesium. A. GEHRING (Z. Pflanz. Düng., 1930, 15A, 300–310).—Crop increases resulting from treatment of soils with magnesium salts is usually most marked under acid conditions. Treatment of soil with increasing amounts of lime water leads to a steady increase in the easily soluble magnesium content to a maximum point corresponding to 70% saturation of the soil with bases. Further addition of lime decreases the solubility of magnesium. The magnesium required for maximum crop production is more closely connected with the degree of saturation of the soil with bases than with the percentage of lime saturation. The discoloration of certain sensitive plants in acid soils may be directly due to magnesium deficiency.

A. G. POLLARD.

Influence of manuring on the baking quality of bread cereals. M. P. NEUMANN and O. NOLTE (Mitt. deut. landw. Ges., 1929, 44, 447–449; Bied. Zentr., 1930, 59, 20–21).—Wheat grown with complete manures produces the same loaf-volume as that from unmanured soil, but this figure decreases when unbalanced manures are used.

A. G. POLLARD.

Influence of phosphoric acid on the yield and quality of hops. E. G. DOERELL (Superphosphat, 1929, 5, 82–85; Bied. Zentr., 1930, 59, 19).—The importance of balanced manuring on the crop yield, quality, and incidence of diseases of hops is discussed. The nutrient requirement of the bines increases as the hops mature. Adequate phosphate manuring is important.

A. G. POLLARD.

Effect of phosphoric acid on the firmness of stalks. E. G. DOERELL (Superphosphate, 1929, 2, 168–170).—Stalks of barley grown on soils deficient in phosphoric acid have thinner membranes, more spongy tissue, and much larger cells than those from soils well supplied with phosphoric acid.

CHEMICAL ABSTRACTS.

Carbon dioxide supply to plants. W. SCHMIDT (Forts. Landw., 1929, 4, 360; Bied. Zentr., 1930, 59, 25—29).—The effect of temperature changes, wind velocity, and simple diffusion on the carbon dioxide content of the air within and near the soil surface is discussed.

A. G. POLLARD.

Relation between reaction condition of soils and plant growth. O. LEMMERMANN and L. FRESSENIUS (Trans. 2nd. Comm. Internat. Soc. Soil Sci., 1929, A, 96—97).—Records of optimum p_H values and of acidity condition for the growth of numerous farm crops are recorded.

A. G. POLLARD.

Proof of the constancy of "effect" factors [in growth curves]. H. NIKLAS and M. MILLER (Z. Pflanz. Düng., 1930, 15A, 193—196).—The mathematical basis of Mitscherlich's experimental proof of the constancy of "effect factors" is adversely criticised (cf. B., 1926, 1024).

A. G. POLLARD.

The yield law [of crops] and the "effect law." A. RIPPPEL and R. MEYER (Z. Pflanz. Düng., 1929, 14A, 1—13).—Mathematical examination of Mitscherlich's newer formulæ of growth curves shows an insufficiently rigid mathematical basis. The constancy of the "effect factors" is not consistently upheld, and the nature of the assumed curves representing maximum yield and the incidence of toxic factors is insufficiently supported. Important physiological factors in plant growth are ignored in Mitscherlich formulæ.

A. G. POLLARD.

Formulation of the yield law [of crops]. E. A. MITSCHERLICH and W. U. BEHRENS (Z. Pflanz. Düng., 1929, 15A, 94—101).—The mathematical representations of Ripppe and Meyer (cf. preceding abstract) are concluded to be untenable.

A. G. POLLARD.

Preparation of hydrochloric acid extracts of soils. A. A. J. VON SIGMOND (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1929, A, 13—18).—The methods of van Bemmelen and of the Prussian Geological Institute for examining soil bases by extraction with hydrochloric acid both lead to decomposition of the original minerals and kaolin-like silicates and cannot give information as to the amount of nutrients present or the degree of weathering of the soil. This is contrary to the results obtained by Ganssen (Internat. Congress Soil Sci., 1927). The ratio $Al_2O_3 : SiO_2$: bases obtained by these methods bears no definite relationship to soil acidity.

A. G. POLLARD.

Percolating cylinder and some of its uses [in soil investigations]. A. N. PURI (Agric. J. India, 1929, 24, 408—412).—The lower end of a glass percolating cylinder is flanged and ground to face a perforated disc, the underside of which is in turn ground to face the flanged edge of a glass funnel. The joint cylinder-disc-funnel is firmly held by a screw clamp. To gauge the texture of a soil the sample (100 g.) is stirred (once only) into 250 c.c. of water, and after being kept overnight the suspension is diluted to a total volume of 500 c.c. The disc of the percolation tube is covered with filter paper and the suspension poured into the tube. The percolate is returned to the tube periodically. After a few hours percolation proceeds uniformly at a

rate determined by the texture of the soil. The values so obtained are reproducible. The effect of lime on the physical condition of an acid soil and the results of treatment of alkali soils with ameliorants may be examined by observations of the changes in percolation rates.

A. G. POLLARD.

Sedimentation apparatus [for soils]. E. RAUTERBERG (Z. Pflanz. Düng., 1930, 15A, 263—269).—The soil suspension is placed in a cylindrical separating funnel, the lower end of which is connected with a constant-level water supply and the upper end is closed with a rubber stopper containing a bent delivery tube. The flow of water through the tube is regulated by a screw clip and rubber connector. The apparatus is used as an ordinary elutriator, the rate of flow being measured directly.

A. G. POLLARD.

Destruction of weeds etc. HERZOG.—See XXIII.

PATENTS.

Manufacture of water-soluble fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,985, 8.4.29).—A fertiliser in which $N : P_2O_5 : K_2O :: 1 : 0.75 : 1.25$ is prepared by treating with ammonia the product obtained by heating phosphoric acid with potassium and/or ammonium chloride until the whole or the greater part of the chlorine is expelled as hydrogen chloride, mixed with ammonium nitrate and, if desired, potassium nitrate or nitric acid, and urea.

L. A. COLES.

Immunisation of seed grain. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 323,813, 13.10.28).—A dusting powder for grain comprises a mixture of about 2—2.5% of an alkyl mercuric halide, acetate, sulphate, nitrate, cyanide, thiocyanate, or hydroxide with non-reacting material, e.g., talc, charcoal, silica gel, "sodium naphthol-pitch sulphonate," lignite, iron hydroxide, together with wetting agents and other fungicides, if desired.

L. A. COLES.

Disinfectant (U.S.P. 1,738,740).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Influence of sodium sulphite on the decomposition of sugar when heated in alkaline solutions. O. SPENGLER, F. TÖDT, and W. WINKLER (Z. Ver. deut. Zucker-Ind., 1929, 79, 668—679).—Sucrose solutions of 15 and 50% concentration, containing 1% of sodium carbonate, were heated for 4—27 hrs., with and without 1% of sodium sulphite, in glass flasks of about 300 c.c. capacity under reflux condensers, in a bath at temperatures of 80—112°. It was first ascertained that the rate of decomposition of the sugar, as measured by the fall in polarisation, was practically independent of the alkalinity of the solutions, at least in the range p_H 9—12. The p_H value diminished in course of heating, but no inversion occurred on the alkaline side of p_H 8—9. It was found that the presence of sulphite had no influence on the rate of decomposition of sugar, although it greatly lessened the discoloration. At 100° in absence of sulphite the results were rather irregular, and in some cases the polarisation actually increased. This was probably due to the formation of labile dextrorotatory

products. At other temperatures, and in presence of sulphite, the results were much more regular. For 15% solutions the rate of decomposition per hr. was at 90° about 0.02%, at 100° about 0.06%, and at 112° about 0.2% of the total sugar. These values are higher than those obtained under different conditions by Herzfeld. The decomposition which occurs during evaporation in sugar factories is usually about 0.05% of the total sugar.

J. H. LANE.

Behaviour of silicic acid in the purification of [beet] juice. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zucker-Ind., 1929, 79, 821—830).—Evaporator incrustations in beet-sugar factories often contain large proportions of silica, and as much as 70% has been reported. According to Claassen, siliceous deposits retard heat transmission much more than does calcium sulphate or carbonate. Possible sources of the silica are the beets, the water, and the lime, but it is now shown that only the last is of practical importance. The first carbonatation eliminates any quantity of silica likely to be present, leaving only about 1 mg. per 100 c.c. in the filtered juice, and if the second carbonatation is carried out without further liming or if the lime used is free from silica the amount in the final juice is reduced to about 0.5 mg. per 100 c.c., which is not a serious amount from the point of view of incrustation. If, however, lime containing silica is added before the second carbonatation the final juice may contain several mg. of silica per 100 c.c. Minute quantities of dissolved silica in juice may be conveniently determined as silicomolybdate by Winkler's colorimetric method.

J. H. LANE.

Calcium oxalate deposits in evaporators [in beet-sugar factories]. O. SPENGLER and S. BÖTTGER (Z. Ver. deut. Zucker-Ind., 1929, 79, 651—667).—In view of the almost complete precipitation of oxalates from sugar solutions by liming and carbonatation (B., 1929, 223), it is probable that the large amounts of calcium oxalate sometimes found in evaporator incrustations are produced during evaporation. The most likely source is glyoxylic acid, which occurs in beets especially when these are unripe. If added to a sugar solution it forms considerable amounts of oxalate under the usual conditions of liming and carbonatation, and also during subsequent boiling of the filtered liquid. Glycollic acid produces no appreciable amounts of oxalate under these conditions. Sucrose itself yields traces of oxalate on prolonged boiling of solutions of alkalinity corresponding to 0.05% CaO, but it is probably much less important than glyoxylic acid as a factor in incrustation. The presence of carbon monoxide in carbonatation gas does not give rise to the formation of oxalate under practical conditions, but Sigmund's observation that it retards carbonatation (B., 1929, 572) is confirmed by the authors.

J. H. LANE.

Viscosity of beet-house syrups. A. N. BENNETT and A. R. NEES (Ind. Eng. Chem., 1930, 22, 91—96).—The viscosities of the following types of solution have been determined over a wide range of temperature and concentration: pure sucrose, sucrose + raffinose, non-stiffened and stiffened syrups, and

syrups resulting from the barium process for recovering sugar from molasses. The effect of the p_H of molasses on the viscosity has been investigated. H. INGLESON.

Electrical determination of ash in raw sugar. O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1930, 80, 1—10).—Of 108 comparisons of the ash contents as calculated from the conductivity of a solution of 5 g. of sugar made up with 25 c.c. of 0.025*N*-hydrochloric acid to 100 c.c. with the results arrived at by direct ashing it was found that in 70% the means of duplicates agreed within 0.02%. Of the pairs, the nearest determinations fell within this limit in 84% of the cases. Use of a more concentrated sugar solution gave similar agreement. Only a part of the discrepancies could be referred to insoluble ash. Since the effect of ash on molasses formation is variable and not the sole factor, the differences between the electrical and chemical methods are too slight to have any significance in assessing quality and yield.

F. E. DAY.

Vapour pressures of saturated equilibrated solutions of lactose, sucrose, dextrose, and galactose. E. O. WHITTIER and S. P. GOULD (Ind. Eng. Chem., 1930, 22, 77—78).—The vapour pressures of saturated solutions of sucrose, dextrose, galactose, and lactose were determined at 25° to obtain a comparison of the tendency of these sugars to absorb moisture. The values found show that the tendency decreases in the order above. The importance of the observations in the manufacture of confectionery and sweetmeats is discussed.

H. INGLESON.

Determination of the moisture content in potato starch by dielectric measurements. SPROCKHOFF (Z. Spiritusind., 1930, 53, 21—22).—Determinations of the water content in potato starch and potato flour by measuring their dielectric constants yielded results which were not concordant. The results were governed by the amount of free water in the starch and flour, and were irregularly affected by the varying amounts of adsorbed and partly combined water which might be present.

C. RANKEN.

Determination of the adhesive power of starch by Saare's method. W. EKHard (Chem.-Ztg., 1929, 53, 975—976).—See B., 1929, 298.

Sugar-cane culture. JERWITZ.—See XVI.
Molasses air-yeast. MEZZADROLI and VEREMEENCO.—See XVIII.

PATENTS.

Electrodes for causing the separation of crystals from sugar solutions. A. GRÄNTZDÖRFFER (G.P. 444,575, 6.5.25).—The positive electrode is wedge-shaped or pointed with the apex towards the cathode, and is provided with perforations so that the gas evolved in electrolysis stirs the solution thoroughly and the individual gas bubbles form nuclei for the crystals. Alternatively, the electrodes may be hollow to allow steam to be blown into the solution. A small current only is passed between the electrodes during evaporation of the liquor.

A. R. POWELL.

Concentration and evaporation of sugar syrups. G. R. BAKER and W. E. PRESCOTT, Assrs. to BAKER

PERKINS Co., INC. (U.S.P. 1,744,096, 21.1.30. Appl., 21.12.26. U.K., 1.1.26).—See B.P. 268,437; B., 1927, 538.

Making emulsions (B.P. 323,534).—See I. **Saccharification of cellulosic materials** (B.P. 323,693).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Causes influencing the yield of molasses air-yeast. G. MEZZADROLI and P. VEREMEENCO (Giorn. Chim. Ind. Appl., 1929, 11, 531—536).—The yield of yeast obtained from molasses wort increases considerably as the aeration of the wort is increased, and continuation of the aeration after the end of the fermentation causes a further slight increase in yield. The addition to the wort of even moderate proportions of alcohol has a harmful influence on the yield and fermenting power of the yeast. Other conditions which increase the yield are large seeding, slight acidity of the wort, addition of phosphate, use of suitable nitrogenous nutriment, and high dilution of the wort. Decolorisation of the wort with animal charcoal or activated vegetable carbons lowers the yield, although it results in pale yeast resembling in colour that obtained from grain worts. Yeast of good baking strength, keeping qualities, and consistency, and free from infection, may be readily obtained in 100% yield calculated on the sugar used. Beet molasses gives better results than cane molasses. Tests on a semi-industrial scale confirm these laboratory results.

T. H. POPE.

Detection of fruit wines in grape wine by means of the sorbitol method. A. RÖHLING and J. RICHARZ (Chem.-Ztg., 1930, 54, 61—62).—The tribenzylidene-mannitol obtained from genuine grape products may be distinguished from the corresponding sorbitol derivative obtained from fruit wines not only by the m.p., but by the marked difference in fluorescence under the quartz lamp. The fluorescence will distinguish the sorbitol derivative even when the adulterant is present in small proportions.

S. I. LEVY.

Modern methods of preparation of absolute alcohol. K. R. DIETRICH (Z. angew. Chem., 1930, 43, 40—44).—A review. The methods of dehydration of alcohol are divided into two classes: (a) that in which a solid or liquid dehydrator is used; (b) that employing the constant-boiling mixtures which ethyl alcohol forms with hydrocarbons such as benzene, chloroform, trichloroethylene and cyclohexane. The earlier methods made use of lime as the dehydrating agent, 30% of the alcohol remaining absorbed in the lime, and of which 25% was recoverable. Adaptations of this are the processes of Lorette (B., 1924, 308) and of Merck. In the latter process 98% of the charge is recovered as absolute alcohol by treatment of the saturated lime in autoclaves. More important is the cyclic process first introduced by Young (cf. B., 1902, 721) and which is now used in France, Germany, and the United States; modifications are introduced when impurities such as aldehyde, acetone, isopropyl alcohol, furfuraldehyde, etc. are present. G. E. WENTWORTH.

Hop culture. DOERELL.—See XVI.

PATENTS.

Manufacture of enzymes by means of bacteria.

KALLE & Co. A.-G. (B.P. 297,684, 24.9.28. Ger., 24.9.27).—Amyolytic and proteolytic enzymes are prepared by the addition of a mixture of lactose and maltose, or a mixture of substances containing these sugars, to a mash obtained by the process described in B.P. 16,198 of 1914 (B., 1915, 846). If milk products are used, 1% of formalin is added to prevent coagulation during the subsequent sterilisation at 133°.

C. RANKEN.

Manufacture of rapidly maturing beers.

W. GREINER (G.P. 457,379, 8.7.25).—Carbon dioxide produced by fermentation is led over the surface of fermenting beer, and as the gas passes from one tank to another it is freed by filtration from the volatile substances which give rise to the immature flavour of the beer. The floors of the tanks are raised in the middle, and at their highest point have the wort valve: the yeast collects in and is run from the trough at the outside edge of the tank bottom.

C. RANKEN.

Manufacture of wine, sparkling wine, etc.

L. BESSETTE (F.P. 632,491, 8.4.27. Mex., 20.4.26).—A mixture of water and desiccated residue from fresh fruit juices is sterilised with ozone and seeded with yeast. The sparkling wines are obtained by carrying out the fermentation in closed vessels.

C. RANKEN.

Stabilisation of fermentation liquids. A. KOEHLER (F.P. 633,878, 4.5.27).—The greater proportion of the fermentation organisms is removed from fruit juice etc. by centrifuging and the remainder is destroyed by the addition of sulphur dioxide.

C. RANKEN.

Manufacture of cereal extract.

L. WALLERSTEIN, ASSR. to WALLERSTEIN Co., INC. (U.S.P. 1,737,279, 26.11.29. Appl., 3.4.23).—A cereal extract having a high content of vitamin-B is produced by making an aqueous mash of malt with material consisting chiefly of wheat germs, and filtering off the fluid extract after saccharification. If desired, proteolytic enzymes may be added to the mash.

C. RANKEN.

Purification of sulphite alcohol. UDDEHOLMS AKTIEBOLAG, ASSCES. of N. WEGNER (Swed.P. 59,818, 21.3.24).—Methyl and ethyl alcohol and acetaldehyde are obtained by the fractional distillation of sulphite spirit mixed with a 1% solution of acid or of acid salt.

C. RANKEN.

Distillation and rectification column. A. M. J. SOREL (F.P. 632,450, 8.4.27).—An inclined column, which can be used for the rectification of wine, contains a series of perforated, horizontal plates arranged so that the perforated portion of one plate covers the imperforate portion of the plate immediately beneath.

C. RANKEN.

Manufacture of absolute alcohol.

E. RICARD, ASSR. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,744,503—4, 21.1.30. Appl., [A] 23.1.24, [B] 3.7.24. Belg., [A] 15.2.23, [B] 6.7.23).—See B.P. 211,454 and 218,662; B., 1925, 646, 563.

Heating elements for brewing-vats. G. GASSBERGER (B.P. 307,884, 14.3.29. Anstr., 15.3.28).

XIX.—FOODS.

Chemical and physico-chemical changes induced in wheat and wheat products by elevated temperatures. I. W. F. GEDDES (Canad. J. Res., 1929, 1, 528—558).—Western Canadian, hard red, spring wheat and the flour milled from it was subjected to heat-treatment in a revolving steel drum, mounted eccentrically and heated by a constant-temperature oil-bath. Samples of freshly milled, unbleached, straight-grade flour (1200 g.) containing 13.90% of moisture were heated for definite periods at temperatures between 54° and 88°, and then, after ageing for 35 days at 18° and 70% R.H., standard baking tests were carried out. Marked improvement in baking quality, notably crust character and crumb texture, was exhibited by flours subjected to low heat-treatment for short periods, but with more severe treatment rapid degradation of quality ensued. No significant change in loaf-volume was observed, and no sharp line of demarcation between improving and damaging temperatures was observed. Baking tests on the same flours with addition of 0.001% of potassium bromate revealed a marked improvement in the control flour, but deterioration in all the heat-treated flours, which exhibited characteristics of over-fermentation. To investigate the effect on fermentation tolerance, baking tests were carried out on the heat-treated flours both by the standard procedure and with addition of 0.001% of potassium bromate, after fermentation periods of 1.75, 3.0, and 4.25 hrs. The baking scores computed from loaf volume, colour, grain, and texture showed that with the basic formula heating for periods up to 24 hrs. at 54.4—60°, or up to 10 hrs. at 62.8—65.5°, caused no decrease in baking quality, but by the bromate test all heat-treated samples showed deterioration. Evidence was afforded that heat-treatment and bromate action are similar in character, and the hypothesis that the former initiates two opposing effects, one of which is oxidation, is supported by the observation that, after the initial derangement, the damage to baking quality is a linear function of temperature for constant time. The scores for baking quality disagree with Kent-Jones' assertion (cf. Cereal Chem., 1928, 5, 235), since all heat-treated flours showed marked decrease in fermentation tolerance, which was not due to change in diastatic activity, since samples baked with addition of 1% of diastatic malt gave parallel results. To investigate the influence of moisture content, some of the flour was dried in a vacuum oven at 35° to 4.9% of moisture, whilst another portion was stored in a humidity chamber till a moisture content of 15.11% was reached. Dampened wheat also was milled to give flour of high moisture content. Baking tests, with and without bromate, on these flours heated for 3 hrs. at temperatures ranging from 54.4° to 100° showed that the temperature at which alteration in baking quality occurs is lower the higher is the moisture content. Thus a sample (moisture content 7.25%) heated at 93.3° was but slightly damaged, whereas a sample containing 15.11% of moisture, when similarly heated, had a baking quality of 25% of that of the control. The safe temperature limit for various moisture contents has been fairly well defined. Heat-treatment of wheat produces less

damage to the flour milled from it than heat-treatment of the flour under similar conditions.

H. J. DOWDEN.

Maturation of flour: supposed maturation by the action of peroxides. D. MAROTTA and F. DI STEFANO (Annali Chim. Appl., 1929, 19, 524—528).—Determinations at intervals after grinding of the proportions of nitrogenous constituents soluble in 70% alcohol in untreated flour and in the same flour bleached with Novadelox (about 25% of benzoyl peroxide+75% of calcium phosphate) give identical results for the two flours. Hence the peroxide has no maturing effect.

T. H. POPE.

Determination of sand in rice flour. EDELHÄUSER (Chem.-Ztg., 1930, 54, 44).—The following modified procedure is suggested, which is applicable to rice flour and to similar foodstuffs which contain silica. The material (5 g.) is carefully incinerated over a micro-burner, and the ash boiled for 10 min. with 10% hydrochloric acid; the liquid is filtered, and the filter paper and residue are gently boiled for $\frac{1}{2}$ hr. with 100 c.c. of 1.25% potassium hydroxide solution. The residual sand is collected, washed with very dilute hydrochloric acid, ignited, and weighed.

H. F. HARWOOD.

Detection of hard wheat grits in dough. H. KÜHL (Z. ges. Getreidew., 1929, 16, 125—128; Chem. Zentr., 1929, ii, 945).—The aqueous extract of the fat-free material is heated at 100° with sodium hydroxide and copper sulphate solution, when various qualities of wheat are distinguished by the intensity of the yellow colour. A greenish-yellow coloration in Nessler's test indicates hard wheat, whilst differentiation is also possible by means of Liebermann's reaction.

A. A. ELDRIDGE.

Composition of cow's milk in nymphomania and piroplasmosis. F. A. WINZER (Z. Fleisch-Milch-hyg., 1929, 39, 361; Chem. Zentr., 1929, ii, 1236).—Considerable fluctuations of fat content, density, dry substance, and f.p. were observed. The enzyme activity is also affected.

A. A. ELDRIDGE.

Number and type of bacteria in commercially prepared infant foods. G. J. and A. M. HUCKER (New York State Agric. Exp. Sta. Tech. Bull. 153, 1929, 28 pp.).—Numbers of bacteria varying widely about an approximate average of 20,000 per g. of dried material are present in various makes of infant food examined. The bulk of the flora are milk-peptonising spore-formers resembling *B. subtilis*, together with a considerable percentage of thermophiles. Seasonal variation in the raw milk does not greatly affect the bacterial content of the prepared product. A tryptophan-broth-peptone-agar containing yeast extract and dextrose is recommended as superior to the standard nutrient agar medium for this work, and a growth of less than 10,000 colonies per g. on plates incubated for 2 days at 37° and 56° is recommended as a maximum standard.

K. V. THIMANN.

Sanitary control of commercially prepared infant foods. G. J. and A. M. HUCKER (New York State Agric. Exp. Sta. Tech. Bull. 154, 1929, 16 pp.).—The introduction of sanitary control, periodical sterilisa-

tion of piping by steam under pressure, and similar precautionary measures in an infant-food factory reduced the average bacterial content from 14,200 to 4700 colonies per g. developing at 37°, and from 34,500 to 3600 colonies per g. developing at 56°. Less than 10,000 colonies per g. were attained in 43 out of 46 samples, and this number is recommended as a standard for infant foods.

K. V. THIMANN.

The Grindrod impact steriliser. G. J. and A. M. HUCKER (New York State Agric. Exp. Sta. Tech. Bull. 155, 1929, 31 pp.).—In this steriliser steam under 30–40 lb. pressure is forced through a fine nozzle into milk kept under a vacuum of 26 in. The milk is thus very rapidly superheated and after 1–3 min. is drawn off into a condensing pan where the excess water is removed. Reduction of bacterial content to 10 per g. was achieved in the case of spore-forming and thermophilic bacteria by heating at 115° for 2 min., and in the case of hæmolytic streptococci at 110° for 1 min. A slight precipitate of albumin which is produced was at first considered to be bacterial debris. The method is suitable for the preparation of dried or evaporated milk products or for use with tropical milk supplies.

K. V. THIMANN.

Control of bacteria that grow during pasteurisation [of milk]. M. W. YALE (New York State Agric. Exp. Sta. Tech. Bull. 156, 1929, 25 pp.).—Thermophilic bacteria increase in numbers considerably during pasteurisation of milk. Their presence in the raw supply is due in some cases to failure to sterilise milk-cans; their growth in the pasteurising plant is encouraged by pauses in the pasteurising run and by other defective treatments.

K. V. THIMANN.

Energy and protein content of foods. F. G. BENEDICT and A. G. FARR (N.H. Agric. Exp. Sta. Bull., 1929, No. 242, 6–60).—Combustion of the food of mixed meals in oxygen shows that the energy value of the total food may be estimated with fair accuracy by multiplying the weight of the air-dried mixture by the factor 5.

CHEMICAL ABSTRACTS.

Microscopical characterisation of different mixtures of tea and its substitutes. A. NIETHAMMER (Pharm. Zentr., 1930, 71, 17–18).—*Tea sinensis* shows no special microscopical characteristics when treated with hydrogen chloride on the slide; many plants used as adulterants and substitutes may, however, be distinguished.

S. I. LEVY.

Suitability of pure aluminium vessels for culinary purposes. A. THIEME (Chem.-Ztg., 1929, 53, 973–974).—From tests on dogs and rats extending over several months it is concluded that even very large quantities of aluminium oxide or aluminium salts are entirely without deleterious physiological action, hence the minute quantities of aluminium which dissolve in food cooked in aluminium vessels cannot have any effect on the human system. The amounts dissolved are on the average much less than is already present in many foods.

A. R. POWELL.

Action of water on aluminium vessels. MASATSCH.—See X. Partial iodine value of fats.

KAUFMANN. Coconut flour. CRUZ and WEST.—See XII. **Manuring and baking quality of bread cereals.** NEUMANN and NOLTE.—See XVI. **Vapour pressure of sugar solutions.** WHITTIER and GOULD.—See XVII.

PATENTS.

Egg product and its production. A. K. EPSTEIN (U.S.P. 1,737,365, 26.11.29. Appl., 13.10.28).—The viscosity and emulsifying power of whole egg and/or yolk are increased by the admixture under suitable conditions of a hydrophilic colloid, such as gum tragacanth, gum arabic, pectin, capable of being dispersed uniformly in water at room temperature.

E. B. HUGHES.

Protective treatment of fruit and the like. E. M. BROGDEN, ASSR. to BROGDEN CO. (U.S.P. 1,738,864, 10.12.29. Appl., 15.2.22).—Citrus fruits, apples, peaches, cucumbers, tomatoes, etc. retain their freshness if thinly coated with paraffin wax and/or paraffin oil in a volatile hydrocarbon, with or without the addition of starch and harmless colour.

E. B. HUGHES.

Dehydrator (U.S.P. 1,737,068). **Making emulsions** (B.P. 323,534). **Blending of liquids** (B.P. 323,242).—See I.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Petrolatum liquidum. S. A. SCHOU and I. B. NIELSEN (Dansk Tidskr. Farm., 1930, 4, 1–11).—The temperature variation of the viscosity of 11 samples of American and European liquid paraffins was not characteristic of their origin. All the samples had an absorption band with a maximum at the same wave-length, $\lambda =$ about 2730 Å., and a minimum at about 2500 Å. The heights of the bands varied and were in good accordance with the sulphuric acid test for organic impurities, and thus the degree of purity may be expressed numerically.

E. H. SHARPLES.

Stabilisation and fermentation of belladonna leaves. J. P. TODD (Pharm. J., 1930, 124, 94–98).—Leaves of belladonna (*Atropa belladonna*), stabilised by treatment with the vapour of 96% alcohol at various pressures above atmospheric, are in appearance and many physical properties inferior to carefully dried leaves. No loss of alkaloid occurs during drying unless this is unduly prolonged, when up to one fifth of the alkaloid may disappear. It is concluded that the enzymes are responsible for the loss of alkaloid. During the extraction of carefully dried and enzyme-free preparations, emulsification of the solvent is much more troublesome than with badly dried and fermented specimens, and an assay process is described which offers many advantages, if slightly modified, in the assay of solanaceous drugs.

E. H. SHARPLES.

Morphine content of opium from hybrids of *Papaver somniferum*. L. F. CAVARA and A. CHISTONI (Rend. Accad. Sci. Fis. Mat. Napoli, 1929, [iv], 35, 31–34).—By cultivation of hybrids of *Papaver somniferum album* and *nigrum* (Rend. R. Accad. Lincei, 1926, [vi], 3), an intermediate type has been obtained which yields opium containing about 13.3% of morphine, whilst

the selected *album* variety gives 12.4%, and imported Persian poppy 14.2%. The effect of rain in reducing the yield of morphine probably accounts for the results recorded in the literature of reduced yields from opium poppies grown in Europe.
R. K. CALLOW.

Microchemical behaviour of the official alkaloids. L. ROSENTHALER (Amer. J. Pharm., 1929, 101, 821—829).—The use of microchemical reactions for identification of the pharmaceutically important alkaloids is advocated, and tests claimed to be suitable are described.
S. I. LEVY.

Use of Kalium sulfoguaiacolicum D.A.B. VI as reagent for the detection of methyl alcohol in spirituous preparations. H. MATTHES (Apoth.-Ztg., 1929, 44, 730; Chem. Zentr., 1929, ii, 920).—The reagent is purified by evaporation of a 2% solution to dryness on the water-bath; it then gives a violet colour with methyl alcohol or formaldehyde, but no colour with a mixture of sulphuric acid and acetaldehyde.
A. A. ELDRIDGE.

Detection of gold in homœopathic preparations. H. NEUGEBAUER (Apoth.-Ztg., 1929, 44, 667—668; Chem.-Zentr., 1929, ii, 920—921).—The gold in solution is reduced with lactose.
A. A. ELDRIDGE.

Composition of the essential oil of *Nepeta Cataria*, var. *Citriodora*, Beck. B. RUTOVSKI and I. VINOGRADOVA (Riechstoffind., 1929, 4, 43, 63—64; Chem. Zentr., 1929, ii, 941).—Oil from the leaves had d_{20}^{20} 0.8902, $\alpha_D +1.8^\circ$, n_D^{20} 1.4735, acid value 6.29, ester value 13.04, aldehyde content 30%; oil from withered, seedless heads had d_{20}^{20} 0.8930—0.8960, $\alpha_D -2^\circ$ to 0° , n_D 1.4756—1.4762, ester value 6.37—10.97, ester value after acetylation 227.6—250.7; aldehyde content 10—20%; this oil contained tiglic acid (?), α - and β -citral, limonene and dipentene, geraniol, citronellol, nerol, a levorotatory sesquiterpene, acetic, butyric, and valeric acids.
A. A. ELDRIDGE.

Japanese peppermint oils. III. Wild mint oil of Hokkaido. IV. Constituents of Japanese peppermint oils. Y. SHINOSAKI and T. NAGASAWA (J. Soc. Chem. Ind., Japan, 1929, 32, 180—182 B).—III. The wild mint oil consists largely of ketones, containing about 20% of pulegone, 40% of *l*-menthone, 30% of *l*-limonene, and 5.8% of fatty acids, in addition to an unknown phenolic substance. The analytical values are given.
IV. In addition to the substances already generally recognised as constituents of the peppermint oils, α -pinene, camphene, *l*- and *d*-isomenthones have been isolated. Δ^8 -Hexenol is present as an ester (yield 1.4%), but its refractive index differs from that given by Walbaum (A., 1918, i, 302).
K. V. THIMANN.

Karlsbad salt. KUHN.—See VII. **Impact steriliser.** HUCKER and HUCKER.—See XIX. **Sagrotan and Thissiol disinfectants.** SABALITSCHKA and BÖHM.—See XXIII.

PATENTS.

Manufacture of a product of therapeutic value from dihydrocodeinone. A. BOEHRINGER (C. H. BOEHRINGER SOHN) and C. SCHOPF (B.P. 320,749, 6.10.28).

—Dihydrocodeinone is treated with acetic anhydride to give an *O*-acetyl derivative, m.p. 154—155° (*hydrochloride*, m.p. 132—135° [decomp.]), having cough-restraining, analgesic, and hypnotic properties.

C. HOLLINS.

Production of a barbituric acid compound. F. HEFTI (B.P. 307,484, 19.9.28. Switz., 9.3.28).—An analgesic and sedative is obtained by melting together 5-phenyl-5-allylbarbituric acid and pyramidone. [Stat. ref.]
C. HOLLINS.

Manufacture of perfumes. I. G. FARBENIND. A.-G. (B.P. 293,703, 9.7.28. Ger., 7.7.27).—Aromatic carbonyl compounds carrying two nuclear *isopropyl* groups are valuable as perfumes. *Däisopropylbenzaldehyde*, b.p. 147—153°/12—13 mm., is obtained from cumene or diisopropylbenzene, carbon monoxide, and aluminium chloride. The aldehyde may be converted by the usual reactions into acids and esters, diisopropylbenzyl alcohol, etc., which are also perfumes.
C. HOLLINS.

Production of a mercury compound of 4-nitro-*o*-cresol. G. W. RAIZISS, ASSR. to ABBOTT LABORATORIES (Re-issue 17,563, 14.1.30, of U.S.P. 1,554,293, 22.9.25).—See B., 1925, 901.

Dental porcelain (B.P. 296,765).—See VIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Photographic films. P. E. DYER and J. McK. HANNAH (B.P. 323,030, 20.6.28).—The emulsion is coated upon a transparent cellulose paper (the product "Sidac" of the Société Industrielle de la Cellulose, Belgium, is recommended); distortion and stripping of the emulsion which usually accompany the use of such supports are avoided by rapidly cooling the emulsion layer immediately after coating. Two suitable emulsion formulæ are given.
J. W. GLASSETT.

Production of photographic images [by the diazotype process]. H. D. MURRAY, and NORTON & GREGORY, LTD. (B.P. 320,603, 14.6. and 14.7.28).—The discoloration of unwashed diazotype prints on keeping is avoided by reducing the alkalinity of the coupling solution by means of a buffer salt. With suitable coupling components (phloroglucinol, resorcinol, *m*-phenylenediamine) the alkali may be supplied in the form of sodium carbonate or phosphate, borax, etc. together with sodium or ammonium acetate, citrate, tartrate, or the like.
C. HOLLINS.

Recovery of silver from old photographic films etc. C. PERROT (F.P. 633,685, 6.9.26).—The films are treated with a warm solution of sodium hydroxide which removes the silver-gelatin layer, the solution is acidified with hydrochloric acid, and the precipitate collected and fused with sodium carbonate to recover its silver content.
A. R. POWELL.

[Photographically] reproducing the surface-markings of marble, wood, and other natural materials. OXFORD VARNISH CORP., Assees. of L. V.

CASTO (B.P. 308,788, 6.3.29. U.S., 28.3.28).—After application of the transfer a coating of a transparent lacquer containing a suitable dye is applied.

J. W. GLASSETT.

Photographic reproduction of images [of three-dimensional objects]. I. MORIOKA (B.P. 321,243, 13.8.28).

XXII.—EXPLOSIVES; MATCHES.

Purification and some properties of technical mercuric fulminate. L. WÖHLER and A. BERTHMANN (Z. angew. Chem., 1930, 43, 59—63).—The preparation of mercuric fulminate free from colour and of crystal size suitable for use as a detonating charge for trinitrotoluene has been investigated. Both the white and the brown technical products, even when the former is prepared from paraldehyde, contain only 98—99% of fulminate. The impurity here is due to mechanically enclosed mother-liquor, and not to water of crystallisation as suggested by Kast and Selle (A., 1926, 1129); mercuric fulminate recrystallised from water is identical with the technical product. The brown colour is due to the presence of polymerised fulminic acid, the grey being due to formation of metallic mercury from a basic mercuric nitrate present as impurity; the fact that a product of 99.8% purity is obtained by recrystallisation from nitric acid, ammonia, or water is considered noteworthy. The minimum detonation charge increases with decrease in crystal size independently of the method of preparation and purification of the sample, the values when plotted lying on a hyperbolic curve. This difference of charge for large and small crystals diminishes with increasing pressure; addition of lead azide reduces the detonation charge required.

G. E. WENTWORTH.

PATENTS.

Manufacture of trinitrotoluene. DYNAMIT A.-G. VORM. A. NOBEL & Co., and R. O. REUTER (G.P. 452,907, 22.10.20).—A mixture of nitric and sulphuric acids is run into molten dinitrotoluene, the temperature being maintained at 85° by means of stirring and suitable cooling. Risk of oxidation, or of fire and explosion, is avoided, and the time of nitration is reduced. A product solidifying at 76.5—77.5° may be obtained.

W. J. WRIGHT.

Blasting cartridges. WESTFÄLISCH-ANHALTISCHE SPRENGSTOFF A.-G. CHEM. FABR. (G.P. 457,151, 2.5.25).—The cartridges are coated with a chlorinated hydrocarbon, or a mixture of such hydrocarbons, having m.p. below 15°. The cartridge wrappers may first be impregnated with zinc ammonium chloride solution to render them unflammable.

W. J. WRIGHT.

Safety sheaths for blasting cartridges. DEUTS. SPRENGSTOFF A.-G., and H. METTEGANG (G.P. 457,602, 24.2.26).—The sheaths consist of magnesia cement, in which the magnesium chloride may be wholly or partly replaced by calcium chloride, with addition of potassium or sodium chloride. The composition is made sufficiently fluid to be cast into moulds. W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Carbon monoxide; danger and determination. F. WIRTH and O. KÜSTER (Zentr. Gewerbehyg. Unfallverhüt., 1929, 16, 149—153; Chem. Zentr., 1929, ii, 1057).—Determinations of carbon monoxide in the air of a garage containing a running motor-car engine are recorded.

A. A. ELDRIDGE.

Determination of small amounts of benzene vapours in air. H. F. SMYTH (J. Ind. Hygiene., 1929, 11, 338—348).—A measured volume of air is aspirated through a special absorber containing 10 c.c. of a mixture of equal volumes of concentrated sulphuric acid and fuming nitric acid. The mixture is diluted with water and steam-distilled in presence of 16.7% of anhydrous sodium sulphate. An excess of titanous chloride is then added, in the absence of oxygen, to the distillate acidified with sulphuric acid and the mixture is heated. The excess of titanous chloride is titrated with ferric alum solution, potassium thiocyanate being used as indicator. Benzene in air containing 200—1800 pts. per million may be determined with an error of less than 1%. No interference is caused by small quantities of ethyl, butyl, and amyl alcohols or acetates, nor by the paraffins which boil near the b.p. of benzene. A modification of the method is given to eliminate the interference caused by toluene when present.

W. O. KERMACK.

Bactericidal action of "Sagrotan" and "Thi-sirol"; disinfectants containing chloroxylenols. T. SABALITSCHKA and E. BÖHM (Pharm. Ztg., 1930, 75, 74—75).—"Thi-sirol" (aqueous solution of about 57% of castor-oil soap and 29% of chloroxylenol mixture) is superior to "Sagrotan" (molecular quantities of *s*-monochloroxylenol and *p*-chloro-*m*-cresol in soap solution) and about 10 times as powerful as Lysol in bactericidal efficiency. The mixture of chloroxylenols is apparently more effective than the *s*-monochloro-derivative alone. Both the above disinfectants protect surgical instruments against rust.

E. H. SHARPLES.

Evaluation of cresol-rosin soap solutions. G. KOGAN (Pharm. Zentr., 1929, 70, 488—490).—The stability of the emulsion of raw cresols in rosin soap solution and the composition and analysis of the materials separating from such emulsions are discussed. The disturbing effect of the rosin acids on the determination of cresol by distillation with petroleum is emphasised, and a more reliable method based on the separation of cresols from the soaps by the addition of barium chloride followed by steam-distillation is described.

E. H. SHARPLES.

Destruction of vermin and weeds by means of the by-products of saccharin manufacture. W. HERZOG (Chem.-Ztg., 1930, 54, 50—51).—A résumé of proposals for the utilisation of the by-products, from the recent technical and patent literature. S. I. LEVY.

Decomposition of pure substances in sewage. W. RUDOLFS (N.J. Agric. Exp. Sta. Bull., 1929, No. 486, 36—47).—Addition of peptone, albumin, casein, asparagine, glycine, or leucine to settled sewage lowered the p_H from 7.0 to 4.0 after incubation (40—125 days), probably owing to production of nitrate; with lactose,

dextrose, starch, or cellulose the initial fall was recovered in 20 days.

CHEMICAL ABSTRACTS.

Changes in composition of material suspended in [sewage sludge] supernatant liquid. J. T. PEDLOW (N.J. Agric. Exp. Sta. Bull., 1929, No. 486, 59–70).—Determinations of total, ammonia-, nitrate-, and amino-nitrogen, p_H , acidity, carbonate, sulphate, sulphur, and carbon in the (a) decanted or (b) filtered liquid above digesting sewage sludge were made.

CHEMICAL ABSTRACTS.

Changes in composition of gas during digestion of fresh [sewage] solids. W. RUDOLFS (N.J. Agric. Exp. Sta. Bull., 1929, No. 486, 54–59).—The carbon dioxide varied from 37.7% (first week) to 25.3% (end), and the methane from 47.8 to 69.8%. When 80–85% of the gas has been evolved the sludge is stable; for complete evolution of gas the digestion time must be doubled.

CHEMICAL ABSTRACTS.

Floc produced [in water] by chlorinated copperas. H. S. HOPKINS and E. R. WHITMORE (Ind. Eng. Chem., 1930, 22, 79–81).—Laboratory experiments under simulated plant conditions indicate that chlorinated copperas is an efficient coagulant producing a compact, easily settling floc at any p_H above 3.5. The amount of coagulant required to produce a satisfactory floc is approximately equal to that of alum, but larger than that of ferric chloride. Manganese is not completely removed by chlorinated copperas below p_H 9.4, but a highly coloured swamp-water was considerably improved by its use in conjunction with lime. The floc is a basic sulphate and does not adsorb sulphate except between p_H 3.6 and 6.6 when the concentration of chlorinated copperas employed was 1710 p.p.m. (cf. B., 1929, 190).

C. JEPSON.

PATENTS.

Septic sewage-disposal plant. L. J. BOMHOFF, Assr. to M. G. SLAWSON (U.S.P. 1,738,521, 10.12.29. Appl., 20.3.28).—Suspended matter and grease may be removed from a septic tank effluent by passing the liquor successively through a series of straining units consisting of a coarse upward-flow filter followed by a still pool. The accumulated sludge and grease may be removed periodically by subjecting the filter to a jet of steam and/or water which may be projected upwards through the grating which forms the base of the filter-bed.

C. JEPSON.

Purification of sewage. J. R. DOWNES, Assr. to PACIFIC FLUSH-TANK Co. (U.S.P. 1,738,362, 3.12.29. Appl., 22.9.28).—Sewage is aerated and purified in an activated-sludge tank in which the contents are circulated on the spiral-flow system by means of a number of air-lifts placed longitudinally along one side of the tank. The maintenance of the sludge in suspension is assisted by scrapers oscillating near the floor of the tank and which bring any deposited sludge into proximity with the air-lift feed. The exit end of the tank is utilised as a settlement basin, and in this case the air-lifts return the deposited sludge either to the inlet channel or to the plant provided for its subsequent disposal.

C. JEPSON.

Incineration of refuse and similar waste materials. O. UHDE (B.P. 323,841, 29.10.28).—By distributing the refuse around the walls of a shaft-type furnace, a more rapid combustion is claimed than is possible when the material is evenly distributed. The increased height of the material at the sides causes the coarser portions thereof to roll towards the centre and, being usually of a more combustible nature, the thinner layer is readily burned. At the same time the roasting effect on the material at the sides liberates combustible gases, and the slagging produced tends to bring more material into the hotter centre zone. The introduction of an air blast at higher pressures than is usual is utilised to carry away fine ashes and similar refractory material for subsequent collection and disposal, while the more easily fusible materials are melted at the temperatures attained and give an improved type of slag. This slag may be removed at frequent intervals, without unduly affecting the operation of the furnace, by means of a movable, central grate portion actuated by a hydraulic ram. Sufficient glowing material is retained on subsidiary grates to ignite the fresh charge.

C. JEPSON.

Paris-green insecticide. B. F. WALLACE (U.S.P. 1,738,089 and 1,738,090, 3.12.29. Appl., 9.2.25).—A powder which floats on water and is suitable for the destruction of mosquito larvæ comprises a mixture of about 4% of Paris-green with light, flocculent calcium carbonate which is waterproofed by heating at, e.g., 220–240° with about $\frac{1}{2}$ –1% of a digestible fatty acid, preferably stearic acid. In (A) the mixture, and in (B) the calcium carbonate alone, is subjected to the waterproofing process.

L. A. COLES.

Manufacture and utilisation of disinfectant and antiseptic embodying furan derivatives. J. P. TRICKEY and C. S. MINER, Assrs. to QUAKER OATS Co. (U.S.P. 1,738,740, 10.12.29. Appl., 23.8.24).—A dusting powder which gradually liberates furfuraldehyde by the action of atmospheric moisture comprises, e.g., a 1:1 mixture (by wt.) of furfuramide and kieselguhr, which may be rendered somewhat more stable by heating at 100–120°.

L. A. COLES.

Marble tube-filter for use in water-supply systems, for fixing free active carbon dioxide. O. RITSCHEL (G.P. 458,510, 29.5.26).—Water is passed through a number of narrow, vertical, sinuous tubes filled with marble, solid sodium silicate, &c.

J. S. G. THOMAS.

Removal of iron from water by aeration. F. BAMBERG (G.P. 458,874, 14.8.25).—Water is aerated under pressure in a closed vessel, and filtered through an open filter.

J. S. G. THOMAS.

Apparatus for regeneration of filtering material used in water-softening filters. RUHLANDWERK A.-G. (G.P. 458,017, 27.10.26).—The filter is connected with a vessel, preferably funnel-shaped and containing the regenerating fluid, by a tube having a three-way tap which, on completion of the regenerating process and flushing with water, is turned to deliver water to be softened in the filter.

J. S. G. THOMAS.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAR. 28 and APRIL 4, 1930.*

L.—GENERAL; PLANT; MACHINERY.

Reliability of fusible tin boiler plugs in service.

J. R. FREEMAN, JUN., J. A. SCHERRER, and S. J. ROSENBERG (Bur. Stand. J. Res., 1930, 4, 1—22).—Examination of 184 fusible plugs from ships' boilers showed that 10% would not operate owing to the formation of an infusible crust consisting mainly of tin oxide. The incrustation is the result of leakage past the plug. The specification of the U.S. Steamboat Inspection Service limits the copper content of the tin to below 0.3%, which necessitates pouring the tin into the bronze cup at a low temperature and leads to imperfect filling. It is recommended that a maximum copper content of 0.5%, with total impurities not exceeding 0.7%, should be permitted and higher pouring temperatures employed.

C. J. SMITHELLS.

Differential thermostat for use in calorimetry.

S. WITCOMBE (Gas J., 1930, 189, 378—379).—For water-flow calorimeters, used with gas, where the water temperature must not be more than 5° below that of the room, a differential thermostat is available. Its action depends on the movement of two rods, made of a special brass with a high coefficient of linear expansion, one of which is immersed in the water supply and the other is surrounded by air. This movement, due to expansion or contraction, is transmitted, with suitable magnification, to a gas governor which controls the supply of heat to a small boiler, or to a valve which regulates the amount of steam passing through a heating coil.

R. H. GRIFFITH.

Simple control stopcock for gas-analysis apparatus.

M. SHEPHERD (Bur. Stand. J. Res., 1930, 4, 23—26).—An improved stopcock is described in which the plug has two bores, one of which has a fine constriction. It is used for controlling the flow of mercury and reagents, a rough balance being obtained through the wide bore, and the cock then turned so that the final adjustment is made through the capillary bore.

C. J. SMITHELLS.

Mixture formulæ. K. K. JÄRVINEN (Suomen Kem., 1929, 2, 80—86).—Variations of the simple mixture formula are discussed, together with their uses and applicability.

S. K. TWEEDY.

See also A., Feb., 185, **Extraction apparatus with fritted glass filter-plates** (SCHÖBEL). 186, **Apparatus for determination of moisture in solids** (KUENTZEL). **Cochius viscosimeter** (SCHULZ). **Claisen fractionating apparatus** (FRIEDRICHS).

PATENTS.

Furnaces with revolving firegrates. K. PETERSEN (B.P. 299,313, 20.10.28. Denm., 22.10.27).—A revolving grate for boiler and other furnaces is divided into seg-

ments, each with its own chamber, below, for forced draught, which is regulated according to the depth of fuel on each segment; dampers transverse to the radius of a segment may also be provided. The furnace is conveniently stoked by an underfeed device which pushes the fuel and ash inwards so that the decrease in bulk as combustion proceeds is compensated for.

B. M. VENABLES.

Continuous [heat-treatment] furnace.

F. T. COPE and A. H. VAUGHAN, Assis. to ELECTRIC FURNACE Co. (U.S.P. 1,738,039, 3.12.29. Appl., 29.3.28).—In a furnace of rectangular shape, trays of the material under treatment are conveyed along one side, across one end, and back along the other side. Discharge of the trays is effected by tilting them at a point which is near the point of charging. The trays may remain continuously in the furnace or be brought outside for the purpose of filling and emptying.

B. M. VENABLES.

[Boiler] furnaces. H. S. HORSMAN (B.P. 323,836, 26.10.28).—A combustion chamber separate from the boiler proper is provided with walls composed of water-cooled tubes connected to headers which are separate from the boiler drums, but are connected to them by tubes so that the main separation of steam and water is in the latter. The tubes forming one wall are brought over the top of the combustion chamber to a header which is common to the other wall. Part of the roof thus formed is left unobstructed by the brickwork baffles so that the gases can emerge to the boiler proper.

B. M. VENABLES.

[Self-regulating] apparatus for supplying steam or other vapours.

P. M. CABELL (U.S.P. 1,737,826, 3.12.29. Appl., 1.5.25. Renewed 1.3.29).—The apparatus is intended to supply a small quantity of steam at a constant pressure irrespective of the demand. Water is supplied from a closed high-level tank under air pressure (maintained constant by a hand inflator) through a long vertical pipe which turns back on itself twice at the lower end and is there provided with a number of restrictions. The water enters a flash boiler which is supplied with an excess of heat, so that water can only enter the boiler (and steam be made) when the pressure is allowed to drop very slightly below the air plus hydrostatic pressure. Steam is prevented from blowing back into the feed tank by the restrictions and bends.

B. M. VENABLES.

Apparatus for heat exchanging. G. T. MOTT (U.S.P. 1,738,914, 10.12.29. Appl., 4.8.26).—A form of tubular heat exchanger with provision for differential expansion between the tubes and the casing is described. Longitudinal baffles are also provided to divide the bundle of tubes into groups.

B. M. VENABLES.

* The remainder of this set of Abstracts will appear in next week's issue.

Heat-transfer apparatus. J. N. JACOBSEN, ASSR. to PFAUDLER CO. (U.S.P. 1,737,849, 3.12.29. Appl., 6.6.27).—In a heat exchanger of the bundle-of-tubes type, the inner fluid passes through the tubes in series, and the outer fluid is given a whirling motion by causing it to enter through suitably inclined orifices.

B. M. VENABLES.

[Fibrous, heat-insulating material [suitable also for acid-resistant filters]. ISOLA-GES.M.R.H. (F.P. 634,542, 17.5.27).—Materials containing magnesia, e.g., dolomite, magnesite, hornblende, are melted and drawn out into fibres. The "wool" so obtained will withstand temperatures up to 1600°. A. R. POWELL.

Conducting thermal reactions between pulverised substances and a gas. G. S. LOY (F.P. 569,342, 17.10.22).—The powdered material is fed into a flue where it meets a stream of hot gas which carries the finer particles into the reaction chamber; the coarser particles collect on the sloping floor of the flue and are returned to the grinding machine. The process is suitable for the distillation of fuel, for the manufacture of calcium cyanamide, and for the roasting of ores.

A. R. POWELL.

Carrying out of exothermic gas reactions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 324,158, 19.11.28).—The apparatus is suitable for reactions between materials of which at least one is liquid at the ordinary temperature, and provides for effective heat-interchange between the initial and final materials, and also in the catalyst tubes, all in one casing. Examples of its application are the catalytic conversion of acetylene into acetone, and of methane into hydrogen and oxides of carbon, by means of a large excess of water vapour. Thus, in the latter example, some of the steam is generated in open-ended jackets surrounding the lower parts of the reaction tubes and upstanding from a reservoir of water which effects the final cooling of the issuing products. The water-gas enters round the jackets, passes upwards, mixes with the steam, passes upwards round the actual reaction zone, then turns back downwards through the reaction tubes. Just before the turn-back additional steam may be admitted from an external boiler.

B. M. VENABLES.

Drying drum with attachment for cooling the material before discharge. BÜTTNER-WERKE A.-G. (G.P. 458,071, 28.12.26).—The device at the discharge end of the drying drum which serves to prevent ingress of air into the drum is provided with a closed cover which is surrounded by a cylindrical or conical sheath so as to form an annular chamber in which the discharge from the drum is cooled by means of a current of cold air passing countercurrent to the stream of dried material. The cooling chamber is closed at the end at which the material enters it from the drying drum by a wall provided with openings to allow the cooling and drying air to escape, and at the opposite end with adjustable openings for the admission of regulated quantities of cooling air.

A. R. POWELL.

Drying and pulverisation of slimy or tacky substances suspended in liquids. SOC. ALSACIENNE DE CONSTRUCTIONS MÉCANIQUES (F.P. 634,981, 28.9.26).—

The mass is mixed with small spheres, cylinders, or rings which are kept in motion while the mixture is heated to expel the liquid. As the material dries, the motion of the spheres etc. breaks up the clots which would otherwise form hard cakes. A. R. POWELL.

Method and apparatus for dehydrating. G. A. SYKES (B.P. 323,740, 8.10.28).—The foodstuff, wood, hide, or other fibrous material is contained in trays between partitions and surrounded by flues within a common casing. Dampers are provided so that hot gases may be drawn (a) through the partitions in contact with the material in either direction, or (b) past the ends of the partitions, leaving the material in a still atmosphere. In the latter case heat is supplied by combustion of gas within the same casing and reaches the material mainly by "radiation," while the atmosphere in contact with the goods tends to become saturated with moisture from the goods, aided in the early stages by trays of water if necessary. These periods of "closed" heating alternate with periods when the gases are drawn over the material and effect the removal of moisture.

B. M. VENABLES.

Pulverising apparatus. O. CRAIG, ASSR. to RILEY STOKER CORP. (U.S.P. 1,739,213, 10.12.29. Appl., 19.10.26).—Two pulverisers (with feeders) and one mixing and conveying fan are driven by the same motor. The apparatus is suitable for a mixture of anthracite and bituminous coal which need different designs of pulverisers for efficient operation. B. M. VENABLES.

Apparatus for pulverising material. W. J. A. LONDON, ASSR. to PEABODY ENG. CORP. (U.S.P. 1,737,800, 3.12.29. Appl., 9.5.28).—In an air-borne system a coarse pulveriser operates on open circuit with a separator, and a fine pulveriser operates in closed circuit with the same separator. The mills may be of different types; e.g., the first may be of the impact type and suitable for comparatively moist coal, the second an attrition mill which, as a rule, will only operate successfully on coal that has been partly dried by the air current.

B. M. VENABLES.

Centrifugal pulveriser. O. A. KREUTZBERG (U.S.P. 1,737,854, 3.12.29. Appl., 7.6.23).—From a centrifugal pulveriser the powdered material is drawn out in an air current through an axial hole in one end of the casing, the conduit rises to a separator, and the elbow is formed with a sloping lower side, which forms a chute down which the oversize from the separator will slide back to the mill against the air current.

B. M. VENABLES.

Mixing mill. D. C. HEIM (U.S.P. 1,739,149, 10.12.29. Appl., 4.1.29).—A mill for proportioning and mixing powdered materials, such as dusting powders, insecticides, etc., is described. The minor constituents of the mixture are measured into tall vessels with hinged bottoms, and are placed upright in a hopper. The main constituent is distributed around them, and, on withdrawal of the tall vessels, columns of the minor constituents are left surrounded by the bulk material. The whole is then withdrawn from the hopper by rotating brushes, and falls through a screen into a container (preferably a shipping drum) containing a

collapsible dasher or loose framework (removed before shipment). The drum is then closed and rotated horizontally by another part of the same machine, while the blending and sifting operations are performed on the next batch.

B. M. VENABLES.

Mixing apparatus. A. D. MACLELLAN (U.S.P. 1,738,440, 3.12.29. Appl., 18.8.26).—A rotating drum has buckets attached to the interior of both ends and radial pans at the middle of the length; the latter catch a portion of the material spilt by a bucket and deliver it to a later bucket at the other end. A screw conveyor in a tube runs right through the drum, the casing of which is cut away at one side only, within the drum, and is capable of rotation through 180° by hand, so that when the opening is up the screw will receive mixed material from the pans and deliver it out of the mixer; when the opening is down the screw will receive feed material from the other end and deliver it into the mixer.

B. M. VENABLES.

Mixing machine. J. F. FIELD (U.S.P. 1,739,066, 10.12.29. Appl., 1.11.28).—A screw-conveyor in a trough runs axially through a mixing drum, the screw and drum revolving in opposite directions and being reversible together. The drum is provided with longitudinal, radial blades upon which are inclined vanes. The screw-conveyor is surrounded by a hopper outside the drum at one end, to the open top of which the unmixed material is supplied and from which it is conveyed into the drum. After mixing and on reversal the material is brought back to the same hopper, whence it falls through a spout which was previously closed by a slide.

B. M. VENABLES.

Feed re-pulper for sedimentation apparatus. J. GREGORICH, Assr. to DORR Co. (U.S.P. 1,739,302, 10.12.29. Appl., 21.7.27).—The apparatus described is suitable for insertion in the feed launders of the intermediate stages of a continuous, countercurrent decantation plant, and may be driven from the shaft of the appropriate pump for thick pulp. It comprises a rocking shaft with stirring arms depending into the feed launder conveying a mixture of thick pulp from the next lower thickener and clear liquor from the one above. The motion of the arms is transverse to the flow of pulp.

B. M. VENABLES.

Apparatus for conveying solid materials through pipes by means of compressed air. T. F. MILLER (B.P. 324,103, 18.10.28).—Means for charging pulverulent material into the conveying conduit containing compressed air are described. The material passes in succession through a charging hopper, an air-lock chamber, an equalising chamber where the intermittent discharge of the air-lock is converted into a steady discharge by means of a worm-conveyor, a further paddle conveyor which also serves to aerate the material, and finally into the main air-stream.

B. M. VENABLES.

Apparatus for distillation of finely-divided material. G. SAUERBREY MASCHINENFABR. A.-G. (G.P. 457,478, 9.10.23. Addn. to G.P. 456,891; B., 1930, 176).—The apparatus of the prior patent is modified by the provision of several inclined drums arranged one above the other in such a manner that the

material to be distilled falls directly from the outlet of one drum to the inlet of the next lower drum.

A. B. MANNING.

Mechanical dissolution apparatus. E. LE BOS (F.P. 635,285, 2.5.27).—The apparatus comprises a trough in which the dissolution takes place, a funnel for charging the trough with a regular supply of solid material, and a flap-valve for regulating the supply of solvent, the whole being adapted for automatically regulating the density of the solution.

A. R. POWELL.

Rotating extraction drum. V. HÄNIG & Co., (G.P. 458,997, 11.10.23).—The drum is provided with a removable plane or slightly curved sieve plate, so arranged that it may be inserted at any time during the extraction process and permits the complete removal of all the liquid from the drum before air is drawn through the filter cake.

A. R. POWELL.

Centrifugal separators. AKTIEB. SEPARATOR (B.P. 317,827, 19.8.29. Swed., 23.8.28).—A separator of the "purifier" type, with two outlets, is convertible into a "clarifier," with one outlet, by fitting a dome round the neck of the bowl instead of by taking it apart and changing the top inner disc.

B. M. VENABLES.

Centrifugal separator. H. NÆSS (U.S.P. 1,738,258, 3.12.29. Appl., 20.2.28. Norw., 18.6.25).—A form of centrifugal separator (with internal cones) adapted to cleaning by flushing without taking apart is described.

B. M. VENABLES.

Centrifugal liquid purifiers. PRESTON STREET COMBING Co., LTD., and J. W. ADAMS (B.P. 323,914, 4.1.29. Addn. to B.P. 296,178; B., 1928, 773).—In modification of the prior patent, the interior of the bowl is shaped so as to give a fair lead for the solid material to each jet from any internal direction, and the guiding surfaces may be continued between the cones by means of ribs or the like.

B. M. VENABLES.

Apparatus for treating liquids. A. JENSEN (U.S.P. 1,738,953—4, 10.12.29. Appl., [A] 9.11.26, [B] 6.6.27).—A helical tubular stirrer is rotated in a liquid of which the temperature is to be rapidly changed, a heating or cooling fluid being forced through the tube by external means. In (A) the container of liquid is adjusted to the stirrer; in (B) the reverse process takes place, the stirrer being carried on a crane-like structure, counter-weighted, and with flexible connexions to the fluid pump, so that the stirrer may be lowered into the container in any position within range.

B. M. VENABLES.

Apparatus for dephlegmation. P. J. DUNCAN, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,738,766, 10.12.29. Appl., 16.5.27).—A fractionating tower is provided with "trays" in which no bubbling takes place, and hence the back-pressure is low. The "trays" are formed from two or more plates, curved in such a way that the gases whirl as they pass through; the liquid descends through holes in the plates, which are small enough to remain drowned.

B. M. VENABLES.

Fractional-distillation apparatus. R. B. CHILLAS, JUN., Assr. to ATLANTIC REFINING Co. (U.S.P. 1,738,036, 3.12.29. Appl., 22.9.25).—In a tower each bubbling plate is formed of a number of separate receptacles with up-turned edges which are spaced apart from each other.

The junctions are covered by caps, some of which have deeply depending flanges and prevent upward passage of the vapours, whilst others have shallower flanges and permit bubbling.

B. M. VENABLES.

Gas separator [cleaner]. A. H. RUBY, Assr. to CONTINENTAL OIL CO. (U.S.P. 1,739,093, 10.12.29. Appl., 17.6.26).—A filter for insertion in pipe-lines for natural gas (or for other purposes) is described. The filtering medium is steel wool or other fibrous material, and is contained in trays supported clear of the sides of the container by a central rod which also supports baffle plates extending to the walls of the casing; the plates are perforated over a portion, the perforations being staggered in different stories. On removal of the top dome the whole interior can be lifted out by the central rod and replaced by a clean one.

B. M. VENABLES.

Dust and like separators. MATTHEWS & YATES, LTD., and O. STOTT (B.P. 324,204, 1.1.29).—The volute of a fan is continued as a circular conduit surrounding the fan. The conduit, but not the volute proper, is divided by plates which are parallel to the course of the air and are provided with skimming devices to remove the layers of air in which the dust has been concentrated centrifugally.

B. M. VENABLES.

Apparatus for separating and/or classifying suspended matter from fluids. H. WARING, and ASSOCIATED LEAD MANUFACTURERS, LTD. (B.P. 323,719, 4.9.28).—The dusty gases are blown tangentially into the top (base) of a space between imperforate outer and perforated inner inverted cones having separate outlets at their apices for coarse and fine dust. The base of the cones is closed by an apertured disc, and above each aperture extends a slightly conical filter-bag, the upper ends of the bags being flexibly supported by, e.g., a lever and weight. Each bag in turn is shut off from the air pressure by means of an obturating plate rotating under the apertures in the disc.

B. M. VENABLES.

Filter for air, gases, and vapours. S. CANTEAU (F.P. 634,377, 14.5.27).—The apparatus comprises a number of filter discs laid one above the other in a canalised container in such a manner that the air can circulate around them, and between the lower and upper disc of each element is fixed a perforated metal ring.

A. R. POWELL.

Manufacture of filter material with a rough surface for air and gas filters. DELBAG-ENTSTAUBUNG GES.M.B.H. (G.P. 458,249, 24.8.24).—Filling rings for gas-cleaning towers are given a rough surface by coating them with sand, powdered porcelain, or the like, mixed with a binding material. The dust is said to adhere more efficiently to a rough than to a smooth surface.

A. R. POWELL.

Filling material for gas-washing towers. I. G. FARBENIND. A.-G. (G.P. 457,966, 3.7.25).—The material consists of small pressed-metal funnels with corrugated walls. This shape admits of almost complete utilisation of the surface, as the individual funnels touch one another only in certain spots and not over appreciable areas as with the usual type of filling rings.

A. R. POWELL.

Preparation of mixtures of compressed gases. ROMBACHER HÜTTENWERKE, and J. I. BRONN (G.P. 458,125,

16.9.22).—Cylinders containing the compressed gases to be mixed at a uniform pressure are connected through one or more similarly operating reducing valves or pressure regulators with a high-pressure pipe-line connected with the containers to which the mixture is to be supplied. The desired proportion of the individual gases delivered to the pipe-line is obtained by using cylinders having volumes in this desired ratio or by using cylinders of equal size, but in numbers corresponding with this ratio.

A. R. POWELL.

Mixing gases and liquids. T. W. BARBER (B.P. 323,758, 8.10.28).—One fluid is delivered in the form of a thin sheet and the other through jets which impinge on the sheet. The former may be pumped through an annular nozzle, and the latter axially through the same nozzle and then through small apertures in the dividing wall. The feed conduits may communicate with opposite ends of a balance chamber with floating piston to equalise the pressures.

B. M. VENABLES.

Apparatus for recovery [with adsorbent carbon] of gases and vapours. O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,737,822, 3.12.29. Appl., 2.7.24).—The solid absorbent is supported between upper and lower mesh-work across the middle part of a cylindrical vessel with horizontal axis. Pipes for media for regulation of temperature during absorption, for heating during regeneration, and for subsequent cooling are evenly spaced throughout the absorbent, and the ends are so connected that there is equal flow through each. Precautions are also taken for even distribution of the in- and out-going gases.

B. M. VENABLES.

Apparatus for dissolving gases in liquids, especially for the preparation of chlorine water. G. BRAAM (Dutch P. 17,747, 7.7.19).—The water in the absorption apparatus is forced by the pressure of the chlorine into an upper reservoir from which it automatically cascades back into the absorption vessel and is there agitated by an automatic stirring device.

A. R. POWELL.

Apparatus for spectral analysis of liquid mixtures. CHARBONS ACTIFS & PROC. E. URBAIN (B.P. 313,438, 30.5.29. Fr., 11.6.28).—The substances are examined in a light-absorption chamber which is maintained at a temperature high enough to ensure that all constituents present are vaporised, while the sample is introduced into, and volatilised in, a side chamber which is heated gradually so that constituents may be volatilised in succession. Both chambers are heated by surrounding electric resistors, separately controllable, and a connexion is provided through which the pressure may be regulated. To break the capsule containing the sample the apparatus may be rotated round the axis of the absorption chamber.

B. M. VENABLES.

Flow pyrometer. A. SCHACK and M. WENZL, Assrs. to SIEMENS & HALSKE A.-G. (U.S.P. 1,746,012, 4.2.30. Appl., 2.7.27. Ger., 8.7.26).—See B.P. 274,096; B., 1928, 42.

Apparatus for disintegrating, grinding, reducing to fibres, mixing, or felting humid or dry materials. E. STERZL (U.S.P. 1,745,727, 4.2.30. Appl., 9.8.27. Ger., 20.8.26).—See B.P. 276,356; B., 1928, 657.

Production of water from steam. P. FABER, AssT. to A.-G. BROWN, BOVERI & Co. (U.S.P. 1,745,364, 4.2.30. Appl., 4.1.28. Ger., 5.1.27).—See B.P. 283,158; B., 1929, 456.

Furnace walls. A. REPPMAN (B.P. 324,346, 22.10.28).

[Cinder trap for boiler] furnaces. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. R. SMITH (B.P. 303,009, 18.12.28. U.S., 24.12.27).

Refrigerating machines. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEN. ELEKTRICITÄTS-GES. (B.P. 303,864, 10.1.29. Ger., 11.1.28).

Heat-insulating material (F.P. 636,263).—See IX.
Purification of gases (G.P. 458,876 and 458,948).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Moisture and combined water contained in coal. S. IKI (J. Soc. Chem. Ind., Japan, 1929, 32, 371—372 b).—Water absorbed by coal at a certain humidity cannot all be equally driven off by drying. The four constituents of coal (α , β , γ , and ulmin) have characteristic absorbing properties, their absorptive power increasing in the order γ , β , α , and ulmin. The lower the caking power and the degree of natural carbonisation, the larger is the total amount of absorbed moisture and chemically combined water. The relation of weathering to water content has been studied. Weathering increases the total water content and decreases the ratio of absorbed moisture to combined water. F. J. WILKINS.

Working of horizontal retorts. G. PERCIVAL (Gas J., 1930, 189, 319—323).—The horizontal retort setting at H.M. Fuel Research Station is of regenerator type, and may be heated from a built-in producer or by gases obtained from external sources. A main chimney damper is fitted so that a slight pressure can always be maintained without alteration of dampers at the regenerator outlet. All control was eventually obtained from the chimney damper, breeze from the fire-front being removed and the primary air-slides full open. Secondary air was adjusted to give 18—19% CO₂ in the waste gas, and spray water was 0.6 lb. per lb. of coke gasified; these conditions avoided possible overheating and made the clinker easier to handle. When water-gas was used for heating it was found that the temperature of the waste gas was 430°, of the combustion chamber 1350°, and of the retort (internal) 1050°, compared with 490°, 1350°, and 1100—1130°, respectively, in the case of producer gas. Only when the carbon dioxide content of the waste gas was reduced to 14%, by admission of more secondary air, was the waste gas temperature raised to 490° when using water-gas. The rate of carbonisation, depending on internal retort temperature, was shown to vary with the waste gas temperature, and to be raised by increasing the volume alone of the waste gas. Heating by coal gas has been accomplished by mixing it with about 2½ vols. of flue gas drawn in by means of a suitable injector. Experiments are also briefly recorded in which attempts were made to use breeze as producer fuel; the resistance of the fire was overcome by forcing in air with a steam-jet, but the loss of fuel through the bars was excessive unless it was mixed with some coal. R. H. GRIFFITH.

Heating of retorts by gas from a low-temperature carbonisation plant. A. A. MCFARLANE (Gas J., 1930, 189, 383—384).—A setting of 40 Glasgow retorts, normally heated by producer gas, has been adapted to burn gas from a Maclaurin plant, and comparative tests have been carried out with both systems. It is found that the modified form gives higher and more uniform temperatures and an increase from 67.97 to 72.92 therms per ton of coal; further advantages are due to absence of clinker and of producer repairs. A second retort setting is being modified to work either with Maclaurin or producer gas. R. H. GRIFFITH.

Present requirements of coke ovens. L. H. SENSICLE (Gas J., 1930, 189, 260—261).—Attention is called to developments which reduce heat losses from coke-oven settings, particularly shortening of the time of carbonisation by improvements in heat transfer, and dry-quenching of coke. The latter may provide steam equivalent to about 6.5 therms per ton of coal carbonised. R. H. GRIFFITH.

Comparisons of silica-walled ovens and semi-silica-walled ovens in the same battery. E. C. DIXON (Gas World, 1930, 92, Coking Sect., 14—16).—In a battery of 21-in. Otto ovens, some have been relined with silica. It was found that with these the flues were hotter at the end of the carbonising period, and that the duration of each charge was less than with semi-silica walls. Using the same coal, the time of heating was reduced by 2—5 hrs. and the coke produced was of more uniform size and contained less volatile matter. R. H. GRIFFITH.

Mine rescue apparatus: the S.M.R.B. gas mask. S. H. KATZ and C. S. W. GRICE (Safety in Mines Res. Bd., 1930, Paper No. 57, 37 pp.).—An investigation, undertaken jointly with the U.S. Bureau of Mines, to develop a gas mask which would afford protection against irrespirable mine atmospheres as long as they contained sufficient oxygen to support life, a condition indicated by continued burning of a flame safety lamp, is described. It is concluded that if the use of the gas mask is permitted, the design, operation, and testing of which is described in detail, it should prove of considerable advantage in facilitating rescue and recovery work and in any operations where respiratory protection is required. C. B. MARSON.

Review of the Koppers "C.A.S." process as applied to British conditions. H. KOPPERS (Gas World, 1930, 92, Coking Sect., 13).—The author replies to criticisms of the above process by Smith (B., 1930, 89). R. H. GRIFFITH.

Catalytic reduction of carbon monoxide at ordinary pressure. III. **Production of liquid hydrocarbons with cobalt-copper-thoria catalyst.** S. KODAMA (J. Soc. Chem. Ind., Japan, 1929, 32, 285—286 B; cf. A., 1929, 773).—Carbon monoxide is reduced by hydrogen in presence of a catalyst consisting of 3 pts. of cobalt, 1 pt. of copper, and 0.45 pt. of thoria at a lower temperature than when a copper or copper-cobalt catalyst is used. Water, benzene, and petroleum are formed; unless the gas velocity is less than 2 litres/hr. the amount of carbon dioxide and methane hydrocarbons formed is very small, so that the final gas may

be repeatedly used in order to raise the oil yield and to reduce the gas loss. The reaction first occurs at 160°; the most suitable temperature is 250°.

S. K. TWEEDY.

Catalytic processes for utilisation of coal-tar crudes. A. O. JAEGER (Gas J., 1930, 189, 262—263).—A lecture. Selective catalytic oxidation can be successfully applied to coal-tar fractions, particularly those containing anthracene, in order to remove non-aromatic substances. When crude anthracene is treated thus, it is possible to carry the process still further and obtain anthraquinone, and naphthalene may similarly be converted into naphthaquinone, phthalic anhydride, or maleic acid.

R. H. GRIFFITH.

Amounts of aromatic hydrocarbons in petrol-eums from Niigata Prefecture. Extraction of xylenes from crude petroleum. S. IMORI and U. KIKUCHI (Bull. Inst. Phys. Chem. Res., Tokyo, 1929, 8, 984—991).—Maki petroleum (Niigata, Echigo Prov.) contains 0.07% of benzene, 0.59% of toluene, and about 5% of xylenes; Amazé petroleum contains 0.07, 0.53, and about 2% in the same order. The xylenes can be extracted from the crude oils by means of sulphonation of the 145—165° fraction.

R. A. MORTON.

Analysis of the benzene fractions of Polish petroleum, and its chemical composition. J. WINKLER (Przemysl Chem., 1930, 14, 1—18).—The use of 92—94% sulphuric acid is recommended for the determination of unsaturated constituents of straight-run benzene; in the case of benzene produced by cracking, the distillate after removal of unsaturated hydrocarbons by this method (cf. Kattwinkel, B., 1928, 78) still contains about 4% of these substances, which should be removed by the former method. Aromatic hydrocarbons can be removed only by the use of 100% sulphuric acid, or by Kattwinkel's phosphorus pentoxide-sulphuric acid reagent. Naphthenes are determined from the depression of the aniline point (Minchin and Nixon, B., 1928, 699). The average percentage composition of 8 straight-run Polish benzines is: unsaturated hydrocarbons 0.3—2.8, aromatic hydrocarbons 7.5—17.0, naphthenes 13.1—28.2, paraffins 61.1—77.7; the corresponding values for a sample of Cross-cracked benzene are 31.5, 9.4, 4.8, and 54.3. The Riccardo toluene value of these benzines varies from 11.7 to 21.1, so that in most cases the addition of benzene is not necessary.

R. TRUSZKOWSKI.

Sakhalin crude oils. S. S. NAMETKIN and E. M. SHAKHNAZAROVA (Nef. Choz., 1929, 16, 419—421).—Characteristics of crude oils from West and East Nutovo are described.

CHEMICAL ABSTRACTS.

Sludge of transformer oils. I. Formation factors of sludge. T. YAMADA (J. Soc. Chem. Ind., Japan, 1929, 32, 317 B).—Sulphur and unsaturated hydrocarbons are both necessary for sludge formation. The former resists acid formation.

C. W. GIBBY.

Influence of added sulphur on the oxidation of the less refined transformer oils. S. MIZUSHIMA and T. YAMADA (J. Soc. Chem. Ind., Japan, 1929, 32, 316—317 B).—Added sulphur increases sludge formation in the case of the less refined transformer and switch

oils. The sludge is not formed by the same reaction as that which causes acidity.

C. W. GIBBY.

Universally applicable substance for the calibration of calorimeters. W. A. ROTH (Chem.-Ztg., 1930, 54, 77—78).—Technical petroleum which has been freed from readily volatile constituents by means of a current of heated air passed through it serves as an excellent standard material for the determination of the water equivalent of calorimeters. Its heat of combustion can be determined with great accuracy; furthermore, the addition of a few drops (accurately weighed) to such difficultly combustible materials as blast-furnace coke and gas carbon in the calorimetric bomb greatly facilitates the determination of the heats of combustion of these substances.

H. F. HARWOOD.

Differential thermostat. Withycombe. Stopcock for gas-analysis apparatus. SHEPHERD.—See I.

Density of mineral oils. JÄRVINEN.—See XII.

See also A., Feb., 166, **Gaseous combustion** (DAVID and DAVIES). 167, **Upper explosive limits of methane-oxygen mixtures** (COOPER and WIEZEVICH). **Spontaneous inflammation of gaseous mixtures** (MONDAIN-MONVAL and QUANQUIN). **Effect of pressure on inflammable mixed gases** (BERL and BAUSCH). **Gaseous explosions** (SOUDERS and BROWN). 168, **Oxidation processes in motor fuel** (BERL and WINNACKER). 169, **Rate of oxidation of ferrous hydroxide and sulphide to ferric hydroxide** (GLUUD and RIESE). 190, **Origin of coal and petroleum** (STADNIKOV). 202, **2:3-Dimethylnaphthalene from coal tar** (KRUBER). 203, **Pyrogenic decomposition of chrysene with hydrogen at high pressures** (ORLOV and LICHATSCHEV).

PATENTS.

Method and apparatus for low-temperature distillation. A. E. WHITE. FROM INTERNAT. BITUMEN OIL CORP. (B.P. 323,523, 30.7.28).—The material is passed continuously through an inclined rotary retort wherein it is heated at a relatively low temperature (e.g., 290°) at the feed end, and at a higher temperature (e.g., 500—600°) towards the discharge end. The water vapour and lighter volatile products are withdrawn by suction from the feed end of the retort, while the fixed gases and higher-boiling volatile products are simultaneously withdrawn by suction from the discharge end of the retort, whereby a neutral point at substantially atmospheric pressure is maintained within the retort between the two zones. A current of air and/or inert gas may be passed through the low-temperature zone, if desired, in order to modify the caking properties of the coal. A current of steam may be passed through the high-temperature zone to prevent undue cracking of the volatile products evolved.

A. B. MANNING.

Carbonisation of material containing oil, fat, tar, etc. P. PLANTINGA (U.S.P. 1,738,202, 3.12.29. Appl., 26.4.24).—The apparatus comprises a carbonising chamber and a communicating auxiliary chamber so arranged that any liquids produced in the latter will not run by gravity into the former, but may be drawn off and collected. The material is fed into the auxiliary chamber, and passes thence into the carbonising cham-

ber. Hot inert gas is circulated continuously through one of the chambers countercurrent to the flow of the material therein, and through a heater. The other chamber is externally heated. The process may be applied to garbage.

A. B. MANNING.

Carbonisation apparatus for vegetable matter.

SOC. D'ETUDES POUR LES COLONIES ET L'ETRANGER, and M. JUIEL (F.P. 635,682, 8.6.27. Belg., 18.6.26).—The carbonising chambers consist of U-tubes terminating in a heating chamber.

A. R. POWELL.

Gasification of finely-divided, earthy, brown coal.

STAATLICH SÄCHSISCHE HÜTTENWERKE, and P. ROSIN (G.P. 457,726, 30.11.23).—The generator is provided with a grate which produces a fine and uniform subdivision of the air blast. The material is fed on to the grate to a height not greater than 40 cm., and the air pressure is maintained sufficiently low to prevent any crater formation.

A. B. MANNING.

Production of carbon black.

C. MATLOCK, Assr. to MONROE-LOUISIANA CARBON CO. (U.S.P. 1,738,716, 12.12.29. Appl., 5.1.21).—A hydrocarbon gas, *e.g.*, natural gas, preferably preheated, is burned in a regulated quantity of preheated air, insufficient for complete combustion. The products are cooled and the carbon black is separated therefrom by filtration or by electrical precipitation. Suitable apparatus is described and figured.

A. B. MANNING.

Active carbon of high density. SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 635,832, 6.10.26).—Sawdust, wood shavings, or other cellulosic material is heated with concentrated sulphuric acid and a small quantity of nitric acid.

A. R. POWELL.

Regeneration of absorptive carbon.

SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 636,165, 12.10.26).—The adsorbed liquid, *e.g.*, benzene, is removed by means of steam in a closed circuit. For the removal of adsorbed water superheated steam is used. Provision is made for the condensation of part of the water in the circuit, the remainder being reheated and used again.

A. R. POWELL.

Manufacture of carburetted water-gas.

P. T. DASHIELL, Assr. to U.G.I. CONTRACTING CO. (U.S.P. 1,738,493, 3.12.29. Appl., 28.10.21).—A heavy oil in a finely-divided form is subjected to the action of radiant heat in a vertical carburettor devoid of chequer brick. The gases and vapours formed enrich the current of water-gas passing through, whilst the solid residue is deposited in granular form on the bottom of the chamber, from which it may readily be removed. After each carburetting period the carburettor is reheated by the combustion of fuel gas therein.

A. B. MANNING.

Emulsions and other mixtures of tars, bitumens, oils, etc. P. GLOESS and M. MARINI (F.P. 633,687, 6.9.26).—The tars etc. are mixed with algin and worked up into emulsions with water or other materials. The products are more stable, plastic, and adhesive, etc. than those not containing algin; they are especially valuable for road-construction purposes.

A. B. MANNING.

Heat exchange between two non-miscible liquids of similar density, *e.g.*, benzol wash oil and

water. A. OTT (G.P. 457,967, 15.3.25).—First direct and then indirect cooling is used. A device provided with cooling surfaces is connected to the vessel wherein the direct cooling of the wash oil is brought about. The cooling device may be placed below the other vessel and the two may be combined within a single containing vessel.

A. B. MANNING.

Conversion of hydrocarbon oils.

E. E. BARTELS, Assr. to STANDARD OIL CO. (U.S.P. 1,741,045, 24.12.29. Appl., 29.11.26).—Oil is passed through a heating coil under 250–500 lb. pressure, at such a speed that no carbon is deposited, and is heated to temperatures ranging from 410° to 540°, according to the nature of the oil. From this coil the oil is passed into the first of a series of four vertical chambers maintained at conversion temperature and containing a supply of oil. Vapours are taken from the top of each chamber to a common main leading to a dephlegmator. Oil is passed from the first chamber to the succeeding chambers by an overflow line which delivers into distributing devices contained in the second and third chambers. These devices divide the oil between the chambers in such a way as to give the best results. The cracking stock is preheated by bringing it in contact with the vapours in the dephlegmator before passing it through the heating coil.

T. A. SMITH.

Treatment of oil.

E. W. GARD, B. G. ALDRIDGE, and H. J. MULTER (U.S.P. 1,739,898, 17.12.29. Appl., 16.11.26).—The removal of finely-divided tar from acid-treated lubricating oils is facilitated by passing the oils through capillary filters. The tar particles on passing through the filter are caused to agglomerate and separation is then possible. The apparatus used consists of a series of filter-plates separated by settling chambers from which tar can be removed. The plates may be of natural pumice or "Filtros" plates, made by fusing together a mass of silica particles at their points of contact. During oil refining the oil is passed through apparatus of this type for each of the operations of dehydration, acid tar and alkali removal. Similar apparatus may be used for acid-treated gasoline.

T. A. SMITH.

Purification and hydrogenation of heavy mineral oils, tars, and suspensions of coal in oil.

INTERNAT. BERGIN COMP. VOOR OLIE EN KOLEN CHEMIE (Addn. F.P. 32,509, 7.7.26, to F.P. 559,787. Cf. B.P. 213,661; B., 1924, 503).—The oils etc. are first freed roughly from oxygen, nitrogen, and sulphur compounds by heating with strong alkalis or alkaline earths under hydrogen pressure, and are then refined by treatment with sodium under hydrogen pressure. Porous materials, *e.g.*, fuller's earth, and also metallic oxides may be added to the alkalis or alkaline earths. By using several autoclaves in series the process may be made continuous.

A. B. MANNING.

Dehydration of oil. E. W. GARD, B. G. ALDRIDGE, and H. J. MULTER (U.S.P. 1,739,834, 17.12.29. Appl., 16.11.26).—Crude oil emulsion is first freed from mud by passing it slowly through a downwardly flowing stream of water. It is then passed through heat exchangers and up through a filter-plate to remove any solid matter which has escaped the water wash. This filter can be

cleared by forcing water, steam, or air through it in the reverse direction to the oil flow. The oil is then passed through a series of "Filtros" plates, the capillaries in which cause the water particles to agglomerate. The water collects in the chambers between the plates and is run off.

T. A. SMITH.

Prevention of knocking in internal-combustion engines. IMPERIAL CHEM. INDUSTRIES, LTD., and S. COFFEY (B.P. 323,463, 26.6.28).—Organic radicals which are capable of existence in the free state, *e.g.*, the triaryl-methyls, or substances which can generate such free radicals at elevated temperatures, *e.g.*, hexa-arylethanes, triarylmethyldiarylamines, tetra-arylhydrazines, diarylnitrosoamines, etc., but which do not contain metals or metalloids, are added to motor spirits in order to prevent knocking.

A. B. MANNING.

Manufacture of high m.p. wax. F. W. SULLIVAN, JUN., ASSR. to STANDARD OIL Co. (U.S.P. 1,740,125, 17.12.29. Appl., 23.7.24).—Crude paraffin wax containing not more than 5% of oil is refined by distillation in a vacuum (5 to 10 mm.) or with copious steam, at temperatures below 345°. If steam-distillation is used, the condensed water should be at least twice the volume of the wax distilled. A wax melting at 50–51° gives 68% of residual wax of m.p. 54°, whereas only 5–15% is obtained by "sweating." The distillate melts at about 45.5°.

T. A. SMITH.

Apparatus for manufacture of carbonised fuel. S. R. ILLINGWORTH, ASSR. to ILLINGWORTH CARBONIZATION Co., LTD. (Re-issue 17,572, 28.1.30, of U.S.P. 1,645,861, 18.10.27).—See B., 1927, 868.

Treatment of bodies [active carbon] of highly porous character. E. BERL (U.S.P. 1,744,735, 28.1.30. Appl., 8.3.24. Ger., 16.3.23).—See G.P. 411,953; B., 1925, 619.

Biological purification of waste liquors from gas works, ammonia-recovery plants, tar factories, and similar industries. H. BACH (U.S.P. 1,745,397, 4.2.30. Appl., 2.12.25. Ger., 10.12.24).—See G.P. 426,422; B., 1926, 646.

Trucks for use in quenching coke. DR. C. OTTO & Co. G.M.B.H. (B.P. 303,376, 31.12.28. Ger., 29.12.27).

Automatic control apparatus for use in manufacture of water-gas and other cyclic processes. HUMPHREYS & GLASGOW, LTD., ASSEES. of SOC. DE CONSTRUCTION D'APPAREILS POUR GAZ À L'EAU ET GAZ INDUSTRIELS (B.P. 310,508, 4.3.29. Fr., 27.4.28).

Gas burners. H. GUHL (B.P. 309,580, 21.3.29. Ger., 13.4.28).

Thermal reactions (F.P. 569,342 and B.P. 324,158). **Pulverisers** (U.S.P. 1,739,213 and 1,737,800). **Gas separator** (U.S.P. 1,739,093).—See I. **Light hydrocarbons** (B.P. 308,712).—See III. **Contact material**. (B.P. 324,541).—See VII.

III.—ORGANIC INTERMEDIATES.

Determination of acetone in air. N. P. KOMAR (Ukrain. Chem. J., 1929, 4, 349–360).—The accuracy of Messinger's method for the determination of small amounts of acetone is demonstrated, and details are

given for the determination of acetone in air. The air is passed into an evacuated absorption flask, into which iodine and sodium hydroxide solutions are introduced by means of a tap-funnel. After about 30 min. the acetone is completely absorbed by the alkaline iodine solution and is then determined in the usual way.

T. H. POPE.

Detection of benzene in mixtures. E. STOCK (Farben-Ztg., 1930, 35, 897).—On shaking benzene or mixtures containing it with an ammoniacal solution of nickel cyanide that has been rendered turbid by addition of acetic acid, a bluish-white precipitate of $\text{Ni}(\text{CN})_2\text{NH}_3\text{Ph}$ is obtained. Details of the preparation of the reagent and carrying out of the test, which may be used for detection and determination of benzene, are given.

S. S. WOOLF.

Utilisation of coal-tar crudes. JAEGER.—See II. **Higher alcohols from waxes.** HIROSE.—See XII. **Glycerin by fermentation.** TOMODA.—See XVIII.

See also A., Feb., 172, **Catalytic dehydrogenation of methyl alcohol and formaldehyde** (GHOSH and BAKSHI). 191, **Pyrolysis of hydrocarbons: isobutene** (HURD and SPENCE). 192, **Identification of mercaptans** (WERTHEIM). 202, **2:3-Dimethylnaphthalene from coal tar** (KRUBER). 203, **Preparation of primary and secondary amines** (GUYOT and FOURNIER). 205, **Degradation of nitrated 1-diazo-2-naphthol-4-sulphonic acid** (RUGGLI and others). 225, **Quinazolines** (AGGARWAL and others).

PATENTS.

Decomposing and hydrogenating heavy organic compounds to produce light hydrocarbons. J. M. F. D. FLORENTIN and A. J. KLING (B.P. 308,712, 30.1.29. Fr., 27.3.28).—The whole or a part of the heat required for the reaction is supplied by preheating the compressed hydrogen, which is introduced into the reaction chamber at a temperature somewhat above that at which the reaction is carried out. After separation of the light hydrocarbons from the issuing current of gas and vapour, the unused hydrogen is re-introduced into the reaction chamber. Part of the gas, however, is withdrawn from the apparatus and purified before being re-admitted with the fresh hydrogen.

A. B. MANNING.

Manufacture of halogen derivatives of organic compounds. M. PÓLÁNYI and S. VON BOGDANDY (B.P. 296,021, 26.7.28. Ger., 23.8.27. Addn. to B.P. 289,795; B., 1930, 182).—In the process of the prior patent vapour of cadmium or zinc is used to initiate the reaction.

C. HOLLINS.

Production of oxygen-containing organic compounds [formic acid etc. by hydration of carbon monoxide]. SOC. CHIM. DE LA GRANDE-PAROISSE AZOTE ET PROD. CHIM. (B.P. 296,049, 17.4.28. Fr., 24.8.27).—Special catalysts for hydration of carbon monoxide at high temperatures and pressures comprise mixtures containing (a) one of the elements aluminium, silicon, cerium, titanium, zirconium, thorium, and (b) one of the elements potassium, sodium, magnesium, chromium, molybdenum, manganese, copper, silver, zinc, cadmium, mercury, lead, arsenic, antimony, bismuth, and vanadium. Temperature and pressure

are adjusted to preclude formation of hydrogen. Preferably one or more of the reaction products is introduced into the reaction zone. [Stat. ref.]

C. HOLLINS.

Manufacture of acetaldehyde. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 288,213, 4.4.28. Ger., 4.4.27).—Vinyl acetate is boiled with aqueous acetic acid and phosphoric acid (or other acid catalyst) and the acetaldehyde formed is removed by distillation through a column. A continuous process is described.

C. HOLLINS.

Manufacture of acetaldehyde and acetic acid. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 321,241, 9.8.28).—Acetylene saturated with water vapour is passed at 100–200° over a mercury catalyst, e.g., mercuric orthophosphate, or mercuric vanadate, or a mixture of mercuric sulphate and silver or tin vanadate.

C. HOLLINS.

Production of concentrated acetic acid. H. SUIDA (B.P. 295,641, 15.8.28. Ger., 15.8.27).—For the extraction of acetic acid in the liquid phase, phthalic and other aromatic esters are used as solvents. Butyl and ethyl phthalate are mentioned specifically.

C. HOLLINS.

Continuous extraction of acetic acid from its aqueous solutions. SOC. ANON. DES DISTILLERIES DES DEUX SÈVRES (B.P. 307,868, 2.6.28. Belg., 15.3.28).—Aqueous acetic acid and the extracting solvent circulate in countercurrent through alternate mixing and settling vessels, the aqueous layer from each settler passing forward to the next mixer and the solvent layer backward to the previous mixer.

C. HOLLINS.

Manufacture of acetone [from acetic acid]. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 299,720, 29.10.28. Ger., 29.10.27).—In the catalytic preparation of acetone the effect on the catalyst (ceria) of variations in temperature or in rate of gas flow is avoided by diluting the acetic acid vapour either with excess of acetic acid or with steam. The vapours may be preheated before entering the catalyst chamber.

C. HOLLINS.

Manufacture of ethyl acetate free from alcohol. F. SCHEUERMANN (G.P. 455,584, 25.12.24).—The ester is freed from alcohol by washing with 10% acetic acid and then distilled, whereby a product containing 95–97% of ester free from alcohol is obtained together with a residual liquor consisting of 50–80% acetic acid, which is returned to the esterification still. Fractional distillation of the wash-liquors from the first-named treatment affords alcohol and further quantities of ester.

A. R. POWELL.

Manufacture of urea and substitution products thereof. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 321,566, 19.11.28).—Ammonia or amines are passed with carbon monoxide or dioxide over carbides of silicon, iron, nickel, cobalt, titanium, or alkaline-earth metals at 450–500°.

C. HOLLINS.

Hydrogenation of crotonaldehyde. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 321,253, 22.8.28).—Hydrogen introduced through a perforated central pipe-lead mixes gradually with crotonaldehyde vapour (to which steam and/or hydrogen

may be added) in the catalyst chamber; over-heating is thus avoided.

C. HOLLINS.

Dry distillation, especially of calcium butyrate. H. DOLTER (F.P. 634,959, 23.9.26).—The material, mixed with a volume greater than its own of spheres constructed of metal, kieselguhr, clay, etc., is treated in a rotating horizontal drum with a current of a non-reacting gas preheated to a temperature sufficiently high to effect the distillation. The gas, after it leaves the drum, is cooled to condense the distillation products and is returned to the process.

L. A. COLES.

Manufacture of ketonic acid esters. DR. A. WACKER GES. F. ELEKTRO-CHEM. IND. G.M.B.H. (B.P. 304,118, 6.11.28. Ger., 14.1.28).—Butyl and higher esters of acetic and higher acids react readily with the corresponding sodium alkoxide to give butyl and higher alkyl acetoacetates, the alcohol being distilled off (with some alkyl acetate etc.) as formed. Butyl acetoacetate, b.p. 100–103°/16 mm., and amyl acetoacetate, b.p. 105–108°/16 mm., are obtained in 90% and 80% yield, respectively.

C. HOLLINS.

Manufacture of nitrogenous condensation products from acetylene and ammonia. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 321,177, 27.7.28).—Pyridine bases suitable for denaturing alcohol are the main products when acetylene and ammonia are passed at 250–300° over a catalyst containing highly dispersed metallic cadmium, lead, gold, or bismuth, preferably on a porous carrier-promoter; e.g., active silica is impregnated with cadmium nitrate, with or without aluminium nitrate, dried, and reduced with hydrogen at 300–400°.

C. HOLLINS.

Manufacture of carboic acid [phenol]. S. J. LLOYD AND A. M. KENNEDY, ASSYS. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,735,327, 12.11.29. Appl., 21.3.25. Renewed 28.10.26).—Chlorobenzene vapour is passed with steam over silica gel.

C. HOLLINS.

Manufacture of [nuclear] alkylated phenols and their hydrogenation products. W. P. WILLIAMS. FROM SCHERING-KAHLBAUM A.-G. (B.P. 320,638, 17.7.28).—In the processes of B.P. 254,753 and 274,439 (B., 1928, 740; 1929, 164) ordinary hydrogenation catalysts may be employed if the temperature is above the decomposition point of the dihydroxydiarylmethane, or just below it if a porous catalyst is used. Thymol is produced from $\beta\beta$ -(2:2'-dihydroxy-4:4'-dimethyldiphenyl)propane.

C. HOLLINS.

Catalytic hydrogenation of aromatic bases. I. G. FARBENIND. A.-G. (B.P. 295,033, 1.8.28. Ger., 6.8.27).—For hydrogenation of arylamines (e.g., ethylaniline, o-toluidine) in the liquid phase, the nickel or cobalt catalyst is mixed with alkali, e.g., sodium carbonate or calcium oxide.

C. HOLLINS.

Manufacture of [halogenated] derivatives of terephthalic acid. A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 321,548, 7.11.28).—2-Chloro- or 2:5-dichloro-*p*-xylenes are completely chlorinated in the side-chains and the products are converted into chloro- and dichloro-terephthalic acids by warming with concentrated sulphuric acid. Bromoterephthalic acids are similarly prepared. The 2-chloro- and 2-bromo-tere-

phthalic acids may be nitrated to give the corresponding 5-nitro-acids, m.p. 265° and 260—262°, respectively.

C. HOLLINS.

Production of halogen-substituted organic sulphonic acids and their salts [wetting-out and emulsifying agents]. ORANIENBURGER CHEM. FABR. A.-G., Assecs. of CHEM. FABR. MILCH A.-G. (B.P. 289,841, 3.5.28. Ger., 3.5.27).—Chlorosulphonic acid is used in presence of manganese dioxide for the simultaneous condensation, halogenation, and sulphonation of aliphatic, aromatic, or hydroaromatic compounds, especially of castor oil or a mixture of ground-nut oil fatty acid, wool fat, and benzene, to give wetting-out and emulsifying agents. The process is also useful for preparation of chloronaphthalenesulphonic acids.

C. HOLLINS.

Production of styrene and homologues thereof. NAUGATUCK CHEM. CO., Asscs. of O. H. SMITH (B.P. 298,152, 7.7.28. U.S., 4.10.27).—Ethylbenzene is chlorinated below 30° to give a mixture of α - and β -chloroethylbenzenes, which is passed without separation through a hot tube at 500—725°; nuclear homologues of ethylbenzene are similarly treated. *o*-, *m*-, or *p*-Chloroethylbenzene also yields styrene by elimination of the nuclear chlorine at the same temperature.

C. HOLLINS.

Manufacture of salts of the urea of *m*-aminobenzoyl-*m*-amino-*p*-toluyl-1-naphthylamine-4:6:8-trisulphonic acid. G. M. DYSON and A. RENSHAW (B.P. 321,580, 5.12.28. Cf. B.P. 314,909; B., 1929, 807).—The alkali salts are converted by means of lead acetate and ammonia into lead salts, which are treated with dilute sulphuric acid in calculated amount; after removal of lead sulphate by filtration the solution is neutralised with ammonia and evaporated in a vacuum.

C. HOLLINS.

Manufacture of arylcarboxylic acid amide-*o*-thioglycollic acids and hydroxythionaphthens [*o*-amidocarbonylarylthioglycollic acids and thioindoxyls]. I. G. FARBENIND. A.-G. (B.P. 295,694, 17.8.28. Ger., 17.8.27).—*o*-Cyanoarylthioglycollic acids are converted by alkaline hydrogen peroxide at 40—70° into the corresponding amides, which give thioindoxyls by the action of caustic alkali at 95°, no isolation of the amide being necessary. The conversion of 5-ethoxy-2-cyanophenylthioglycollic acid into 6-ethoxythioindoxyl by way of the amide is described. C. HOLLINS.

Manufacture of condensation products of naphthalene and naphthalene derivatives. I. G. FARBENIND. A.-G. (B.P. 295,990, 1.8.28. Ger., 22.8.27. Addn. to B.P. 265,601 and 273,665; B., 1928, 740).—Naphthalene or tetrahydronaphthalene is condensed with propylene, butylene, or higher olefine in presence of aluminium chloride.

C. HOLLINS.

Production of borneol and isoborneol. J. A. TUNING (B.P. 321,442, 2.8.28).—In the manufacture of borneol from pinene or camphene, improved yields are obtained by using *o*-benzoylbenzoic anhydride or the mixed acetic anhydride as esterification agent.

C. HOLLINS.

Purification of benzanthrone. NEWPORT Co. (B.P. 305,536, 25.1.29. U.S., 6.2.28).—A solution of

crude benzanthrone in concentrated sulphuric acid is diluted with water to 65% acid concentration and pure benzanthrone is extracted with toluene at 60°.

C. HOLLINS.

Production of highly halogenated derivatives of pyranthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,161, 30.7.28).—More than 4 halogen atoms are introduced by halogenating (especially brominating) pyranthrone in chlorosulphonic acid in presence of iodine, selenium, etc. at 65—70°. Pentabromopyranthrone is a fast scarlet vat dye. [Stat. ref.]

C. HOLLINS.

Conversion of gaseous aliphatic ethylene hydrocarbons into liquid hydrocarbons. E. RICARD, Assr. to SOC. RICARD, ALLENET & CIE. (U.S.P. 1,745,028, 28.1.30. Appl., 24.7.23. Belg., 9.8.22).—See B.P. 202,311; B., 1925, 64.

Production of *N*-monoalkyl derivatives of aminophenols. W. TRAUBE and E. HELLRIEGEL (U.S.P. 1,746,064, 4.2.30. Appl., 9.9.27. Ger., 15.11.26).—See B.P. 280,553; B., 1929, 236.

[Manufacture of] acid diazonium salts of arylsulphonic acids. F. KELLER and K. SCHNITZSPAHN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,744,903, 28.1.30. Appl., 24.12.25. Ger., 19.1.25).—See B.P. 269,212; B., 1927, 647.

Production of hydroxyanthraquinones. J. THOMAS and H. W. HEREWARD, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,744,815, 28.1.30. Appl., 5.5.24. U.K., 18.2.24).—See B.P. 246,529; B., 1926, 312.

IV.—DYESTUFFS.

See A., Feb., 205, Azo dyes (RUGGLI and others). 215, New vat dyes from β -aminoanthraquinone (RIESZ and FEIKS).

PATENTS.

Manufacture of xanthen dyes and their use. IMPERIAL CHEM. INDUSTRIES, LTD., A. COULTHARD, and E. H. RODD (B.P. 320,345, 7.7.28).—The xanthenes of B.P. 314,825 (B., 1929, 711) are condensed in presence of phosphoryl chloride etc. with secondary or tertiary arylamines having a free *p*-position, yielding rhodamines with three basic groups. The products may be sulphonated to give acid dyes. 3:7-Tetramethyl- or -tetraethyl-diaminoxanthone is condensed, for example, with ethyl- α -naphthylamine or *p*-tolyl-*m*-phenetidine.

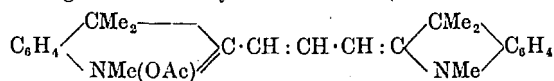
C. HOLLINS.

Bromination products of 6:6'-dichloro-4:4'-dimethylthioindigo and their manufacture. NEWPORT Co. (B.P. 292,904, 23.4.28. U.S., 25.6.27).—The thioindigo named is brominated in concentrated sulphuric acid to introduce less than 2 atoms of bromine.

C. HOLLINS.

Manufacture of dyes of the polymethine series. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (B.P. 321,155, 27.6.28).—The condensation product from formaldehyde and 2 mols. of a cycloammonium salt having a reactive α -methylene or α -methyl group is oxidised in a non-alkaline medium, and the product is treated with alkali to eliminate acid. Thus 2-methylene-1:3:3-trimethylindolenine is condensed with formalde-

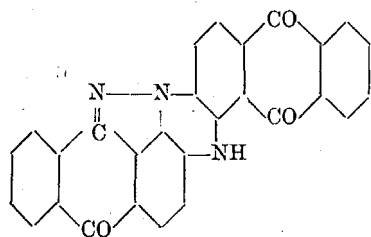
hyde and oxidised in benzene or chloroform solution with *p*-benzoquinone or lead peroxide in presence of acetic acid to give the indocyanine acetate (annexed formula).



Similarly, methylenediquinaldine methiodide is oxidised to pinacyanol base.

C. HOLLINS.

Manufacture of vat dyes [of the pyrazolanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,585, 15.12.28, 27.2. and 6.4.29).—Pyrazolanthrone having a free 3-position are condensed with 2-halogeno-1-aminoanthraquinones, or 3-halogeno-pyrazolanthrone with 1-aminoanthraquinones, in nitrobenzene in presence of copper and an acid-binding agent.



The products may be improved by halogenation. The vat dye (annexed formula) from pyrazolanthrone with 2-bromo-1-aminoanthraquinone gives bluish-violet shades; with 2-bromo-1-

amino-4-hydroxyaminoanthraquinone blue-green, grey after chlorination; from 3-bromopyrazolanthrone and 1:4-diaminoanthraquinone, green-grey, bordeaux after chlorination.

C. HOLLINS.

Manufacture of [red] vat dyes [of the pyrazolanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,703, 9.8.28).—4-Chloro-1:8-naphthalimide or an *N*-derivative is condensed with pyrazolanthrone and the product is fused with alcoholic alkali at 115–120° to give a red vat dye. Very pure shades result from *N*-alkyl derivatives or on subsequent alkylation.

C. HOLLINS.

Production of indanthrone bodies [halogenation of diarylaminoindanthrones for green vat dyes]. R. J. LOVELUCK, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 321,192, 30.4.28).—4:4'-Dianilinoindanthrones, prepared from 2-halogeno-1-amino-4-anilino- (or -toluidino-)anthraquinones, are dichlorinated or dibrominated in nitrobenzene to give fast green vat dyes. C. HOLLINS.

Dyes and dyeing. [Separation of leuco-esters of vat dyes from metal and pyridine.] R. S. BARNES, J. E. G. HARRIS, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 321,462, 11.8.28).—Decomposition of the sulphuric ester of leuco-vat dyes during isolation is prevented by first removing all copper, *e.g.*, by heating with caustic alkali and filtering, before distilling off the pyridine in steam.

C. HOLLINS.

Production of *N*-dihydro-1:1':2:2'-anthraquinoneazine fast to chlorine. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,397, 11.7. and 12.11.28, and 25.3.29).—Impure indanthrones, *e.g.*, indanthrene-blue RS or its methyl or sulphonic derivatives, are treated at 30–70° with manganese dioxide and sulphuric acid; unsulphonated indanthrones may further be treated with a mild sulphonating agent in presence

of boric acid. High yields of chlorine-fast product are claimed.

C. HOLLINS.

Azo dyes and their application [to wool and viscose silk]. BRIT. DYESTUFFS CORP., LTD., and R. BRIGHTMAN (B.P. 321,483, 21.8.28. Addn. to B.P. 294,284; B., 1928, 704).—In the dyes obtainable according to the prior patent the nitro-group is reduced, the resulting aminoazo dye being then diazotised and coupled with an aromatic amine or phenol or a sulphonic or carboxylic derivative of such. The final product must contain as end-component at least one 2:8-aminonaphtholsulphonic acid or an *N*-substituted derivative thereof.

C. HOLLINS.

Manufacture of azo dyes [for cotton]. SOC. CHEM. IND. IN BASLE (B.P. 299,791, 31.10.28. Switz., 31.10.27).—An arylamine containing no hydroxyl groups is diazotised and coupled through two non-hydroxylated middle components (α -naphthylamine or Cleve acid) with *J*-acid in acid, neutral, or alkaline medium to give grey to green-grey direct dyes. Suitable first components are aniline, β -naphthylamine-4:8-disulphonic acid, metanilic acid, Cleve acid.

C. HOLLINS.

Manufacture of [azo] dyes [containing chromium]. SOC. CHEM. IND. IN BASLE (B.P. 289,094, 23.4.28. Switz., 23.4.27).—Blue dyes with affinity for cotton are obtained by coupling diazotised 5-nitro-*o*-aminophenol with *N*-aryl-*J*-acids in alkaline solution, and chroming the product.

C. HOLLINS.

Manufacture of [azo] dyes containing chromium. SOC. CHEM. IND. IN BASLE (B.P. 295,594, 13.8.28. Switz., 13.8.27).—Chromable azo dyes are heated with sodium sulphide solution and chromium hydroxide, preferably with the addition of sulphite-cellulose liquor, glycerin, sucrose, etc. and caustic alkali. Nitro-groups are reduced at the same time to amino-, azo-, or azoxy-groups. The products dye silk, wool, cotton, and viscose silk or may be used for colouring varnishes.

C. HOLLINS.

Manufacture of developing dyes. I. G. FARBENIND. A.-G. (B.P. 303,026, 24.12.28. Ger., 24.12.27).—Secondary disazo dyes of the type: first component \rightarrow non-phenolic middle component \rightarrow benzenoid *p*-coupling amine, or similar trisazo dyes containing a second non-phenolic middle component, are *m*- or *p*-nitrobenzoylated and the nitro-group is reduced to give direct dyes which may be diazotised and developed on the fibre for browns. Examples are: *G*-acid \rightarrow Cleve acid \rightarrow *m*-toluidine; β -naphthylamine-4:8-disulphonic acid \rightarrow *m*-toluidine \rightarrow 2:5-dimethoxyaniline; aniline-2:5-disulphonic acid \rightarrow Cleve acid \rightarrow α -naphthylamine \rightarrow *m*-toluidine.

C. HOLLINS.

Manufacture of dyes [in dry, finely-divided form]. S. W. DUCKWORTH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 320,359, 9.7.28).—Dyes, especially vat dyes and insoluble dyes for acetate silk, obtained by drying aqueous pastes with or without dispersing agents, are finely ground, *e.g.*, in a ball mill, with ether, chloroform, or other low-boiling liquid. The product is a readily dispersible powder.

C. HOLLINS.

Production of body colours. "KOLLOIDCHEMIE" STUDIENGES M.B.H., J. B. CARPZOW, M. MARCH, R. LENZ-MANN, and H. SANDERS (B.P. 319,808, 30.5.28).—A mud

containing 20–60% of colloidal “unsaturated” silicon compounds (*i.e.*, compounds in which the silicon and oxygen are in the ratio $x:2x-1$) is treated with organic or inorganic dyes (which may also be formed *in situ*) in presence of metal salts capable of reacting with the mud to form silicates; the water is then removed. The mud may be activated by preliminary keeping in absence of air, if desired with addition of anaerobic bacteria and suitable nutrients. Examples are magenta, Turnbull's blue, ferric oxide (from the blue by calcination in presence of ammonia), and aniline-blue.

C. HOLLINS.

Manufacture of vat dyes. II. T. BUCHERER, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,745,004, 28.1.30. Appl., 24.2.27. Ger., 29.5.25).—See B.P. 252,745; B., 1927, 838.

Halogenated pyranthrones (B.P. 321,161). **Benzanthrones** (B.P. 305,536).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Straw. II. Production of furfuraldehyde from oat husks and oat straw. O. ROUTALA and O. KUULA.

III. Action of enzymes on the hemicelluloses. O. ROUTALA and H. SOINI (Suomen Kem., 1929, 2, 124–135, 135–146).—II. Hydrolysis of oat husks containing 29.33% of pentosans by means of zinc chloride gave 4.21% of furfuraldehyde, whilst with aluminium chloride the yield was 6.76%. Heating with 3.57% sulphuric acid solution at 145° and under 4 atm. for 7 hrs. gave 6.34%, whereas 9.30% resulted from treatment under similar conditions with a 3.3% phosphoric acid solution for 15 hrs. About 4% of the weight of the original material was present in the residue after hydrolysis as sugars fermentable with yeast. From oat straw containing 26.67% of pentosans a 9.57% yield of furfuraldehyde was obtained by the phosphoric acid treatment.

III. Straw treated so as to liberate its hemicelluloses is not fermented by malt extract, and the waste liquor from the production of sulphite-cellulose from the straw is not fermented by yeast. Straw, cotton, and pine-wood yield the same triacetates and cellobioses. A 40% zinc chloride solution dissolved 76.81% of a straw containing 19.4% of lignin in 24 hrs.

R. CUTHILL.

Brittle straw in rye. J. FRATEUR (Natuurwetensch. Tijds., 1930, 12, 2–5).—The second crop from a crossing of Petkus and Eecloo rye gave 133 plants, of which two were very brittle. The grain from these was sown, and the proportion of brittle plants increased rapidly. Examination revealed no histological basis for the brittleness, but chemical investigation showed a very low proportion of crude cellulose (27.7–29.8%, against 57.4–60% for normal straw), and a very high proportion of extractable matter (64.7–66.8%, against 33.2–35.8% for normal straw), which contained abnormally high proportions of pentosans.

S. I. LEVY.

Carbonisation of wool. W. KEGEL (Leipziger Monatsschr. Textil-Ind., 1929, 44, 219; Chem. Zentr., 1929, ii, 953).—A discussion.

A. A. ELDRIDGE.

Bamboo. II. Pulp-making from the bamboo “Mōsō-chiku.” Y. UYEDA (J. Cellulose Inst., Tokyo,

1930, 6, 3–5; cf. B., 1928, 477).—The yields and composition of the pulps obtained in the digestion of bamboo by the soda, the sulphite, and the bisulphite processes are compared. In the soda process the pulp obtained contained a high proportion (22.81% *av.*) of pentosan, whereas by the other methods both lignin and pentosan contents were largely removed. The yields of pulp by the three methods are 54.7, 48, and 52%, respectively.

F. R. ENNOS.

Change of viscosity during ripening, and the spinning of viscose. G. KITA, S. IWASAKI, and S. MASUDA (J. Cellulose Inst., Tokyo, 1930, 6, 19–26).—Viscose prepared with soda of more than 18% concentration and with carbon disulphide equivalent to about 50% of the cellulose undergoes the usual changes of viscosity during ripening, and there is no improvement in the threads so produced compared with those obtained from normal viscose.

F. R. ENNOS.

Cellulose acetate. I. Acetylation of cotton cellulose. Y. KAMI, M. INUBUSHI, and K. KITAZAWA (J. Cellulose Inst., Tokyo, 1930, 6, 11–18).—Viscosity determinations on the esterified cellulose indicate that the minimum quantities (by wt.) of reagents required for complete acetylation are acetic anhydride 3 pts., acetic acid 3 pts., and sulphuric acid 0.2 pt. for 1 pt. of cotton wadding, and 2.5, 2.5, and 0.2 pt., respectively, for 1 pt. of linters pulp.

F. R. ENNOS.

Apparatus for concentration of sulphite-[cellulose] waste liquors. ANON. (Chem.-Ztg., 1930, 54, 99).—An evaporator for concentrating the waste liquors of the paper industry without foaming or the formation of hard crusts comprises a boiler with heating system consisting of a number of concentric rings each of which is built of two cylindrical, metal shells with upper and lower strengthening rings welded on to form a chamber through which the heating steam is passed. The liquor to be evaporated circulates rapidly through the annular spaces between the heating rings. The heating surface of a 7-ring boiler is 120 m.² All the auxiliary apparatus is arranged below the boiler so that the evaporating surfaces can be readily reached for cleaning, which is effected by means of a mechanically operated metal brush. A basket-shaped chamber above the heating rings is provided with a rotating beating device which serves to break up any foam that forms. Data are given for the capacity and power consumption of four sizes of this type of evaporator.

A. R. POWELL.

Theory of rosin sizing of paper. H. ROSCHIER (Suomen Kem., 1929, 2, 48–54).—The author's conclusions (B., 1928, 851) have been questioned by Öman, who considers the fibres always absorb aluminium ions and that the sizing is effected by free resin independent of the acidity, aluminium hydroxide being detrimental to the sizing rather than improving it. Experiments are described which confirm the author's previous results, and which show that aluminium resinate, aluminium hydroxide, and free resin are of great importance in sizing (although free resin and the aluminium ions in the fibres cannot themselves bring about sizing), whilst aluminium ions are of little or no importance. Thus lime-free, ashless filter-paper fibres take up almost no

aluminium ions from alum solution, but the fibres can nevertheless be satisfactorily sized. Benzene extracts 82% of the rosin used for a drawing-paper made at p_H 4.8, the extract containing 81% of aluminium resinate, *i.e.*, the aluminium and resin are chemically combined.

S. K. TWEEDY.

Oxidation of alkali-cellulose by ageing and its importance in the manufacture of artificial silk.

G. KITA and I. SAKURADA (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1929, 12, 1—20).—See B., 1929, 749.

See also A., Feb., 205, **Action of aromatic diazo-compounds on lignin and cellulose** (KÜSTER and DAUR).

PATENTS.

[Centrifugal apparatus for] treating flotalant materials [degumming of silk]. J. J. McKEON, Assr. to TOLHURST MACHINE WORKS, INC. (U.S.P. 1,739,162, 10.12.29. Appl., 27.2.26).—The material (raw silk) to be cleaned by alternate solvent extraction and centrifuging is treated in bulk in a free condition and remains in one detachable centrifugal basket during all stages of the process.

B. M. VENABLES.

Improvement of (A) artificial fibrous material, (B) artificial fibres or fabrics. L. LILIENTFELD (B.P. 323,731—2, 2.7.28).—(A) Regenerated cellulose silks show a reduced tendency to crease, improved elasticity and extensibility, a softer feel, and a lustre more closely resembling that of natural silk, without any deleterious effect on their strength, if they are treated, preferably in an unstretched condition, with alkaline reagents such as caustic alkali solutions (5—74% calc. as NaOH), organic bases, or alkali sulphide solutions containing more than 15% $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ at temperatures between 25° and 150°. The duration of the treatment may be 1 min. for caustic alkali solution or 5 min. for alkali sulphide solution. This process may be applied to mixed fabrics containing, *e.g.*, cotton and cellulose silks, and is particularly suitable for the treatment of Lilientfeld silk. The results of 43 experiments on materials so treated are tabulated. (B) The effects produced by the above process are also obtainable at the ordinary temperature (15—25°) with caustic alkali solutions containing more than 30% of caustic alkali (calc. as NaOH).

D. J. NORMAN.

Preparation of cellulose masses [from straw etc.]. CELLULOSE ET PAPIERS (SOC. DE RECHERCHES ET D'APPLICATIONS) (F.P. 569,492, 26.10.22).—Straw or other raw fibrous material is rapidly and uniformly digested with a caustic alkaline liquor in a digester provided with an inner, open, cylindrical vessel; the material is circulated upwards by, *e.g.*, a helical stirrer or pump in the inner cylinder and then downwards through the outer space.

A. J. HALL.

Manufacture of fine viscose filaments. VEREIN. GLANZSTOFF-FABR. A.-G. (G.P. 458,790, 9.8.19).—In the manufacture of viscose silk in which strongly acid (sulphuric acid) spinning baths are used, also a viscose solution having a degree of ripeness suitable to the acidity of the bath, and spinning jets of the usual dimensions, an addition is made to the bath of products obtained by hydrolysing cellulosic materials by means of mineral acids.

A. J. HALL.

Coagulation or precipitation of viscose. L. LILIENTFELD (B.P. 323,747, 2.7.28).—For this purpose warm or hot caustic alkali solution or alkali sulphide solution is used either alone or in conjunction with other suitable agents. Thus a viscose solution containing 6.5% of cellulose and 8% of caustic soda is filmed on glass and then immersed in 18% caustic soda solution at 104° for 6—10 min. Fourteen other examples are given.

D. J. NORMAN.

Manufacture of cellulose esters. KODAK, LTD., Assees. of H. T. CLARKE, C. J. MALM, and R. L. STINCHFIELD (B.P. 287,880, 27.3.28. U.S., 28.3.27).—Mixed esters of cellulose are obtained by treating cellulose with an organic monocarboxylic acid and a lower aliphatic acid anhydride, both the acid and the anhydride entering into the reaction. A solvent (chloroacetic acid) and an esterification catalyst (zinc chloride, perchlorates, etc.) are desirable. For mixed esters insoluble in benzene the ratio of higher to lower acyl groups should be within certain limits; in each C_{24} cellulose molecule the number of acyl groups above C_8 should be between 0.3 and 4.0, whilst the acyl groups C_1 to C_5 should exceed 8 but be less than 11.6. The most useful film-forming esters are those containing 9 acetyl groups and about 3 stearyl and/or palmityl groups. A method for determining the proportion of acyl groups is detailed.

C. HOLLINS.

Manufacture of mixed esters of cellulose. KODAK, LTD., Assees. of H. T. CLARKE and C. J. MALM (B.P. 290,571, 4.5.28. U.S., 14.5.27. Addn. to B.P. 287,880; preceding abstract).—In the process of the prior patent the higher acid is first treated with the lower (acetic) anhydride, the lower acid is distilled off, a solvent (chloroacetic acid) is added, and cellulose is esterified with the resulting mixture in presence of a catalyst (magnesium perchlorate).

C. HOLLINS.

Manufacture and treatment of cellulose derivatives. H. DREYFUS (B.P. 320,842, 18.4.28).—Cellulose is acylated with chloro- or bromo-acetic anhydrides. The halogen may subsequently be replaced by hydroxyl, amino-, alkylamino-, or other groups. Mixed esters may also be made.

C. HOLLINS.

Sizing of paper. L. F. DOBLER (F.P. 569,363, 19.10.22).—The paper is sized and passed through rollers heated at 100—175° so that the resinous constituents melt and the size is distributed homogeneously through the material.

L. A. COLES.

Manufacture of transparent paper resistant to fat and moisture. RÉALISATION DES BREVETS FRANÇ. AMOUROUX & Co., Assees. of F. C. WICKEL (F.P. 631,645, 16.11.26. Ger., 14.8.26).—Highly calendered sulphite-cellulose paper is coated on both sides with a fluid, resinous, formaldehyde condensation product and passed between rollers at 120—150°.

A. J. HALL.

Manufacture of cellulose esters. KODAK, LTD., Assees. of H. T. CLARKE and C. J. MALM (B.P. 289,853, 1.5.28. U.S., 4.5.27).—See U.S.P. 1,690,620; B., 1929, 203.

Manufacture of foils, films, ribbons, etc. from viscose and similar cellulose solutions. E. CZAPEK and R. WEINGAND (U.S.P. 1,745,247, 28.1.30. Appl., 1.9.27. Ger., 13.9.26).—See B.P. 277,309; B., 1928, 810.

Stencil sheet for use in duplicating. A. DE WAELE, Assr. to D. GESTETNER, LTD. (U.S.P. 1,744,755, 28.1.30. Appl., 6.7.27. U.K., 8.7.26).—See B.P. 298,705; B., 1929, 14.

Production of compound yarns or threads. O. RASCH (B.P. 314,521, 30.5.9. Ger., 30.6.28).

[Spreader device for] manufacture of very smooth, varnished, flexible fabric. G. A. LANGVERTE & CIE. (B.P. 309,866, 15.4.29. Fr., 16.4.28).

Production of thin films or strips from solutions of cellulose or its derivatives [by extrusion]. H. HAMPEL (B.P. 324,648, 28.8.28).

Decorated [toilet] articles and their manufacture. (SIR) G. C. MARKS. From DU PONT VISCOLOID CO. (B.P. 323,473, 29.9.28).

Method of paper drying and apparatus therefor. H. A. GILL. From J. O. ROSS ENGINEERING CORP. (B.P. 324,745, 20.11.28).

[Multi-layer] sheet or strip material for packing, covering, decoration, etc. WOLFF & CO. KOMM.-GES. AUF AKT., E. CZAPEK, and R. WEINGAND (B.P. 324,326, 19.10.28).

Artificial masses etc. (B.P. 321,258).—See XIII. **Feeding-stuff** (F.P. 623,663).—See XIX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Improving the [wetting-out] action of treatment liquids in the textile industry. H. T. BÖHME A.-G. (B.P. 297,383, 20.8.28. Ger., 20.9.27).—The wetting-out properties of aromatic sulphonic acids substituted in the nucleus by groups having three or more carbon atoms are preserved in highly concentrated alkaline solutions, such as those used in mercerising, by adding thereto a phenol or similar substance. In an example a mixture of sulphonated ricinoleic acid, tricresol, and dipropyl-naphthalenesulphonic acid is used. A. J. HALL.

Preparation of effect threads. CHEM. FABR. VORM. SANDOZ (B.P. 323,500, 323,515, and 323,548, 24.7.28. Addns. to B.P. 280,493; B., 1928, 812).—(A) The process of the earlier patent may be satisfactorily carried out at temperatures above 25°, and hence in a shorter time, if the activity of the acetylating bath is reduced by using either a smaller quantity of the catalyst or a less active one. Thus, bleached cotton yarn (100 pts.) is treated for $\frac{1}{2}$ — $\frac{3}{4}$ hr. at 80° with a bath containing zinc chloride (30 pts.), acetic anhydride (250 pts.), and glacial acetic acid (720 pts.). (B) Greater economy and a product of softer feel are obtained if the above process is carried out with the theoretical quantity of acetic anhydride or only a slight excess thereof. (C) The cellulose is treated first with one or more of the constituents of the acetylating mixture and later with the remaining constituents. D. J. NORMAN.

Treatment of textile fabrics. BRIT. CELANESE, LTD., and R. S. FELGATE (B.P. 323,210, 20.9.28).—In the production of effects on fabrics composed of or containing cellulose acetate, the fabric is first embossed and the effect material is then applied to the upraised portions by a roller or similar device. This process is parti-

cularly applicable for the production of local saponification effects and subsequent cross-dyeings. Suitable apparatus is described. D. J. NORMAN.

Treatment of artificial silk threads and filaments. J. BRANDWOOD (B.P. 323,216, 26.6.28).—The tendency of artificial silk, when dyed in the form of, e.g., cheeses or while wound on perforated spools, to show unevenness, and with parti-coloured dyeing a ragged edge, may be obviated by first treating the silk with a paraffin wax emulsion which is not in itself soluble in the dye liquor. The emulsion is preferably introduced into the centre of a perforated spool and forced through the thread mass, and, at the same time, between the individual filaments of the thread, by centrifugal action. D. J. NORMAN.

Bleaching fibres with ozone. E. CRESPI and M. P. OTTO (F.P. 622,646, 2.2.26).—Raw textile fibres are treated for 7 hrs. at 3 atm. pressure in a closed vessel with a 6% solution of caustic soda containing 1% of Turkey-red oil, then washed, soured with dilute hydrochloric or oxalic acid, again washed, and then bleached while moist in a chamber with air containing 4.5–9.0 g. of ozone per m.³ A. J. HALL.

Bleaching of straw. R. FALANDRIN (F.P. 618,007, 22.6.26).—Straw is treated with a mixture containing sulphur dioxide and an acid salt, such as an acid sulphate or oxalate. A. J. HALL.

Dyeing with anthraquinone vat dyes. CHEM. WORKS, FORMERLY SANDOZ (B.P. 300,662, 17.11.28. Ger., 18.11.27).—Dye liquors used in dyeing printed reserve fabrics with anthraquinone vat dyes by the glucose-caustic soda process are stabilised by addition to the vat of phenols or naphthols to which 2–10% of hydrogenated aromatic substances (*cyclohexanols*, *methyl-cyclohexanols*, hydrogenated naphthols or naphthalene) has been added. The decomposition and re-oxidation of the dye liquors is thereby prevented, and leuco-compounds set free in the liquors remain in solution or in a finely-dispersed form. A. J. HALL.

Dyeing of cellulose acetate silk. GILLET ET FILS (F.P. 570,264, 17.11.22).—The silk is successively treated for a long time with cold potassium permanganate solution, washed, treated with sodium bisulphite solution, treated with hydrochloric acid until the brown colour disappears, and dyed with basic dyes. L. A. COLES.

Production of special [cross-dyeing] effects on mixed fabrics of cotton and acetate silk. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,027, 30.4.28).—The cotton in the mixed fabric is dyed with a sulphide dye (preferably vatted with hyposulphite instead of sulphide) in presence of a borate, hydrogen carbonate, or other weak-acid salt of an alkali metal (sodium if sodium sulphide is used). The acetate silk in the fabric remains uncoloured and may afterwards be dyed, if desired, with acetate silk dyes. C. HOLLINS.

Dyeing and printing [over-printing with soluble esters of leuco-vat dyes]. J. MORTON, J. I. M. JONES, and STANDFAST DYERS & PRINTERS, LTD. (B.P. 321,191, 26.4. and 10.12.28).—Cloth is printed with a soluble

leuco-ester together with an oxidant which does not affect the leuco-ester but is a resist against an ordinary vat dye used in the subsequent over-printing or padding. The leuco-ester and oxidant may be applied to yarn which is then woven with untreated yarn, and the whole over-printed or padded. A suitable oxidant is sodium *m*-nitrobenzenesulphonate. C. HOLLINS.

Printing of fabric with pigment dyes. SOC. ALITHER & GUEX, and W. HUGENTOBLE (F.P. 570,093, 2.11.22).—A printing paste is used consisting of the pigment dye, cadmium-yellow, and linseed oil diluted with turpentine or glycerin. A. J. HALL.

Improvement of vegetable textile materials. L. LILIENFELD (B.P. 323,174—5, 2.7.28).—(A) The materials are mercerised by means of an alkali sulphide solution containing not less than 25% of alkali sulphide (calc. as $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) at a high or moderately raised temperature. Cotton yarn treated for 5 min. in crystalline sodium sulphide solution of 100% concentration at 115° showed an increase in strength of 66% in the dry state and 59% in the wet state as against 16.5% and 17.3%, respectively, for the same yarn mercerised with 22.5% caustic soda solution. (B) Mercerisation is effected by concentrated solutions of caustic alkalis (20–74%) at temperatures well above 100°, *e.g.*, 120–150°; thus cotton yarn mercerised in a stretched condition with a 50% caustic soda solution at 140° for 5 min. showed increases in strength in the dry and wet states of 68% and 42.5%, respectively, as against 48% and 26.6%, respectively, for the same yarn mercerised with 50% caustic soda solution at 100°. Further, the lustre obtained at the higher temperature is superior to that at 100°. D. J. NORMAN.

Treatment of fabrics or yarns composed of natural celluloses. Treatment of mixed fabrics or yarns composed of natural celluloses and wool or natural silk. Treatment of fabrics containing artificial silk. W. MARSHALL (B.P. 323,345—6 and 323,307, [A, B] 12.12.28, [C] 7.11.28).—(A) The handle and lustre of materials made from natural celluloses are improved and the risk of damage is minimised if, after mercerisation or other treatment with alkali solution, the material is washed in an aqueous solution containing one or more of the following protective agents: sodium or potassium chloride, sulphate, nitrate, chlorate, sodium thiosulphate, sucrose, glycerin, and glucose. A solution containing 8% or more of common salt is suitable at a temperature between 15° and 104°, preferably 43°. The use of such protective agents prevents damage to (B) animal fibres and (C) regenerated cellulose silk fibres when mixed fabrics are treated with caustic alkali solutions, and in the latter case promotes level dyeing of the cellulose silk fibres. In the case of fabrics containing animal fibres the washing should be conducted at a low temperature. D. J. NORMAN.

Proofing of woollen or other cloth. F. D. TOYNE, and HUNT & WINTERBOTHAM, LTD. (B.P. 323,250, 30.8.28).—The cloth is impregnated with rape, linseed, or similar oil containing a relatively small quantity of lead and/or manganese, colophony and/or turpentine, and also, if desired, a cobalt compound known as

“linnælite.” When dry the treated fabric is resilient, substantially impermeable to water, oil, or similar liquids, and is particularly suitable for printers’ blankets. D. J. NORMAN.

Treatment [waterproofing] of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 323,501, 31.7.28).—Insoluble metal soaps may be introduced into such cellulose materials by treating the latter simultaneously or successively with a swelling agent and a soluble metal salt, *e.g.*, chloride, sulphate, or thiocyanate of tin, aluminium, iron, or chromium. Alternatively, the salt solution, if sufficiently concentrated, may itself act as a swelling agent. Suitable swelling agents include aqueous solutions of acetic acid, lactic acid, alcohol, acetone, diacetin, phenol, urethanes, guanidine, etc. The treated material, with or without rinsing, is subsequently treated with solutions of soluble soaps or of the fatty acids themselves. In certain circumstances either the soluble soap or the metal salt may be dissolved in the spinning liquor. The resulting products have a subdued lustre and show improved resistance to water. D. J. NORMAN.

Sizing of fibrous materials. TEINTURERIE DE LA RIZE (F.P. 618,284, 1.7.26).—The fibrous materials are impregnated with the sizing mixture in a closed vessel *in vacuo*, excess of the mixture is run off, and the fibrous material exposed to a current of warm ozonised air.

A. J. HALL.

Treatment of felts made from animal hairs. H. HAAKH and H. SUBAL (U.S.P. 1,745,417, 4.2.30. Appl., 7.6.27. Ger., 13.8.26).—See B.P. 275,939; B., 1928, 48.

Liquid centrifuging apparatus, more especially for washing machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 317,310, 5.4.29. Ger., 13.8.28).

Water-soluble condensation products (B.P. 321,190). **Wetting-out etc. agents** (B.P. 289,841).—See III. **Xanthen dyes** (B.P. 320,345). **Dyes and dyeing** (B.P. 321,462). **Azo dyes** (B.P. 321,483).—See IV. **Coloured varnishes** (B.P. 293,358).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Thermal production of phosphoric acid and high-percentage phosphates. E. V. BRITZKE and N. E. PESTOV (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 59, 5—160).—Laboratory yields of 90–99% were obtained from phosphates of low phosphorus content. Data concerning the production of phosphine and iron phosphide are recorded. By using lime, alumina, and silica, an aluminous cement was also obtained, but the higher temperature decreased the yield of phosphorus. Lime from the towers was converted into phosphate to a 50% P_2O_5 content. The precipitation of phosphorus pentoxide by the Cottrell apparatus, the theory of the separation of phosphoric acid from gases, and the deposition of fumes of phosphoric acid on the surface of lime or crude phosphate were examined. With lime a phosphate containing 58% P_2O_5 (water-soluble),

and with crude phosphate (15.6% P_2O_5) one containing 36% P_2O_5 (available) was obtained.

CHEMICAL ABSTRACTS.

Catalytic reduction of carbon monoxide. KODAMA.—See VII. **Changes in superphosphate during storage.** SHOJI and SUZUKI.—See XVI. **Determination of thallium in mouse poisons.** LEPPER.—See XXIII.

See also A., Feb., 151, **Adsorption of nitrogen peroxide by colloidal silicic acid** (SAPOSHNIKOV and others). 156, **Preparation of concentrated silicic acid sols** (CHALIZEV). 176, **Hydrates of bleaching powder** (NEUMANN and HÄBLER). **Phosphorescent compounds** (VERNITZ). **Yellow luminous phosphorescence** (VANINO and SCHMID). 183, **Electroanalysis of copper** (GUZMÁN and RANCAÑO). 262, **Iodides, bromides, and iodide-oxidases in marine algæ** (KYLIN).

PATENTS.

Apparatus for producing sulphuric acid. MANSFELD A.-G. F. BERGBAU U. HÜTTENBETRIEB, H. KREBS, and R. BORCHERS (B.P. 304,688, 11.1.29. Ger., 24.1.28).—In the manufacture of sulphuric acid of d 1.61 or over, by the nitric oxide process, the reaction vessels, comprising chambers, towers, and gas-reaction vessels, are constructed of cast iron, wrought iron, steel, mild steel, cast steel, or certain iron alloys. Provided that the concentration of the acid does not fall below d 1.61 there is considerably less action on the plant than when lead is used in the construction. W. J. WRIGHT.

Manufacture of hydrogen and phosphoric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,122, 20.10.28).—Hydrogen and phosphoric acid are obtained continuously and in the pure state by treating phosphorus with water or water vapour at 300–400° and at pressures up to 500 atm. The gaseous and liquid reaction products are removed and subjected separately or together to another pressure treatment, with addition of water or water vapour if desired. W. J. WRIGHT.

(A, B) **Oxidation of phosphorus in presence of steam.** (c) **Condensation of phosphoric acid.** COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES (ÉTABL. KUHLMANN) (F.P. 635,432, 2.6.27, 635,501, 3.6.27, and 635,765, 10.6.27).—(A) Phosphorus is converted into phosphoric acid by steam in the presence of a silicide of copper, of copper and nickel, or of copper, iron, and nickel. (B) Finely-powdered copper or nickel phosphide on a granular carrier is used as catalyst. (c) Phosphoric acid is recovered from the gases by absorption in hot dilute phosphoric acid, which is thereby brought up to the desired concentration. The remaining acid in the gases is removed by filtration.

A. R. POWELL.

Boiler for alkaline solutions. H. FRISCHER (G.P. 458,372, 17.5.25).—The boiler comprises a cast-iron pan with a wrought-iron cover. A. R. POWELL.

Production of barium hydroxide, alkali hydroxide or carbonate, and hydrochloric acid from barium sulphate [silica, and alkali chloride]. SOC. FRANÇ. DE SUCRATERIES (BREVET ET PROC. DEGUIDE) (F.P. 633,368, 26.4.27).—Barium sulphate is

heated with silica at 1500° or above and the polybasic silicate so obtained is ground and leached with water to extract barium hydroxide (cf. Swiss P. 122,352; B., 1929, 516). The gases evolved in the furnace treatment are converted into sulphuric acid in lead chambers or contact plant and the acid is used for the manufacture of hydrochloric acid and alkali sulphate from alkali chloride. Finally the alkali sulphate is dissolved in water and the solution treated with the barium hydroxide solution previously obtained to regenerate barium sulphate and yield alkali hydroxide. A. R. POWELL.

Caustic magnesia from impure magnesite and rocks containing it. STEIRISCHE MAGNESIT-IND. A.-G. (Austr.P. 109,012, 18.5.22).—The rock is burnt at such a temperature that only the magnesium carbonate is converted into oxide, the calcium carbonate and magnesium silicate remaining undecomposed. The product is then agitated in scouring cylinders, whereby the magnesia falls to a fine powder which is separated from the unchanged rock by screening. A. R. POWELL.

Preparation of hydrazine from ammonia by electrical discharge. I. G. FARBENIND. A.-G. (G.P. 454,699, 20.3.26).—A rapid stream of dry ammonia is passed through an ozonising apparatus operated with an alternating current of 500 periods at 10,000 volts, the issuing gases are condensed, and the liquid is subjected to fractional distillation to remove ammonia. Alternatively, the walls of the apparatus may be kept moistened with an aliphatic hydrocarbon oil, e.g., paraffin oil, which absorbs the hydrazine. A. R. POWELL.

Manufacture of hydrogen peroxide in concentrated solution. M. BODENSTEIN (G.P. 458,189, 25.12.26, and 458,190, 13.4.24).—(A) Barium peroxide is treated with sufficient phosphoric acid to form the soluble primary barium phosphate and the solution is then treated with hydrofluoric acid and barium peroxide alternately until a thick suspension of barium fluoride is obtained. After removing the insoluble barium fluoride by filtration, the clear liquor is treated with barium peroxide until it ceases to dissolve, then with barium carbonate to precipitate completely all the phosphoric acid and leave a concentrated solution of hydrogen peroxide. The barium fluoride precipitate is converted into nitrate by digestion with calcium nitrate and the nitrate is recovered and converted into peroxide by roasting. The calcium fluoride obtained is heated with sulphuric acid to regenerate hydrofluoric acid for further use. (B) Arsenic acid is used instead of phosphoric acid, and hydrofluosilicic or hydrofluoboric acid in place of hydrofluoric acid. Alternatively the barium may be precipitated by introducing gaseous hydrogen fluoride, silicon tetrafluoride, or boron trifluoride into the phosphate solution. The residual hydrogen peroxide is purified by fractional distillation *in vacuo*. A. R. POWELL.

Preparation of a stable hydrogen peroxide solution containing formaldehyde. A. RITTERSHOFER (G.P. 458,889, 10.8.27).—Formalin containing added potassium hydroxyquinolinesulphonate or other quinoline derivative is mixed with 30% hydrogen peroxide. A. R. POWELL.

Manufacture of sodium fluoride or bifluoride. STICKSTOFFWERKE GES.M.B.H., and E. HENE (G.P. 458,526, 28.10.26).—A concentrated brine is treated with ammonium hydrogen fluoride and the precipitate is washed with a solution of the precipitant, the wash-liquors being returned to the process for further use as a precipitant. The sodium hydrogen fluoride so obtained is free from chloride. A. R. POWELL.

Extraction of alkali iodide from water containing iodine. N. V. MIJNBOW EN HANDELSMAATSCHAPPIJ "SOEMBER ASSIN" (Dutch P. 17,807, 27.9.26).—The iodine is concentrated by adsorption with starch and subsequently extracted with alkali bisulphite or bicarbonate. A. R. POWELL.

Electrolytic production of hypochlorite solutions. F. CHEVRIER (F.P. 635,654, 21.12.26).—A high-tension anode is used in an acid electrolyte so that a large amount of ozone is generated in the anode compartment of the cell. A. R. POWELL.

Preparation of anhydrous chlorides of aluminium, iron, etc. VER. F. CHEM. U. METALLURG. PRODUKTION (G.P. 455,266, 9.3.24).—A mixture of hydrogen with a large excess of chlorine is passed over a hot mixture of the metal oxide and carbon whereby, owing to the heat of reaction of the hydrogen and chlorine, the metal chloride formed sublimes out of the reaction chamber. The gases from the condensation vessel are treated for the recovery of pure hydrochloric acid. A. R. POWELL.

Thermochemical treatment of bauxite with sulphuric acid. F. TRAWINSKI (F.P. 633,199, 4.4.27).—A thick suspension of finely-divided bauxite in water is added to concentrated sulphuric acid, the heat of dilution of which is thereby utilised in effecting the reaction. A. R. POWELL.

Preparation of an aluminium magnesium silicate (artificial mica). STUDIEN-GES. F. WIRTS. U. IND. M.B.H. (G.P. 458,475, 19.5.25).—A fused mass of magnesium chloride, quartz, alumina, feldspar, and calcium fluoride is subjected to electrolysis. A. R. POWELL.

Preparation of crystallised iron, copper, zinc, and nickel sulphates [from pickling liquors]. G. AGDE (G.P. 458,191, 23.12.26. Addn. to G.P. 431,581; B., 1926, 914).—The solution is cooled nearly to 0°, whereby the greater part of the ferrous sulphate crystallises out and the solution becomes correspondingly enriched in acid. The mother-liquor may be suitably diluted and used for further pickling in order to concentrate the sulphates of the other metals, which are subsequently recovered by the process claimed in the prior patent. A. R. POWELL.

Contact material for catalytic cracking and like operations. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 324,541, 11.2.29. Fr., 10.11.28).—The material, which is preferably worked up into small amular units, comprises activated aluminium silicate, not more than 30% of an argillaceous binder, and, e.g., about 3% of free alumina. L. A. COLES.

Hydrogen producer for intermittent working of contact chambers. P. GROSSMANN (G.P. 458,187,

9.7.26).—The contact chambers are long, narrow, and high rectangular flues provided with discharge openings in the narrow sides and heating flues along the long sides between each pair of chambers, the flues being divided by vertical walls into several compartments the heat of which can be separately controlled. Numerous horizontal floors of gas-permeable material covered with a thin layer of catalyst are evenly disposed through the contact chambers. A. R. POWELL.

Separate recovery of sulphur dioxide and hydrochloric acid from gases. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (F.P. 635,339, 31.5.27. Ger., 31.5.26).—The gas current is washed with water on the countercurrent system to obtain a final concentrated solution of hydrochloric acid and a gas with a high content of sulphur dioxide free from chlorine. A. R. POWELL.

Separation of ammonia from gases and mixtures of gases containing it. G. F. UHDE (U.S.P. 1,745,730, 4.2.30. Appl., 13.6.27. Ger., 15.6.26).—See B.P. 272,929; B., 1928, 482.

Manufacture of solutions of salts of alkali metal and aluminium. H. BRENEK, ASSR. to RHENANIA VER. CHEM. FABR. A.-G. (U.S.P. 1,745,734, 4.2.30. Appl., 3.7.24. Ger., 10.7.23).—See B.P. 218,998; B., 1925, 803.

Preparation of cyanamides of the alkaline-earth metals and magnesium. N. CARO, A. R. FRANK, H. H. FRANCK, and H. HEIMANN (U.S.P. 1,745,350, 4.2.30. Appl., 19.10.27. Ger., 28.10.26).—See B.P. 279,812; B., 1929, 53.

Manufacture of zeolites. S. DAHL-RODE, ASSR. to COCHRANE CORP. (U.S.P. 1,745,337, 28.1.30. Appl., 23.12.24).—See B.P. 245,092; B., 1927, 382.

Conducting thermal reactions (F.P. 569,342). Chlorine water (Dutch P. 17,747).—See I. Metals for metal carbonyls (B.P. 324,382).—See X. Treatment of bones (F.P. 634,361).—See XV. Fertilisers (Swiss P. 124,358 and F.P. 635,523). Seed-pickling material (G.P. 454,570).—See XVI.

VIII.—GLASS; CERAMICS.

Dependence of the solubility in water of the soluble substances of clays and ceramics on the temperature of calcination. P. P. BUDNIKOV (Ukrain. Chem. J., 1929, 4, [Tech.], 205—213).—The materials examined were Tschasov-Jarski and Nikolaevski clays, Cetlitski kaolin, porcelain from the Baranovski factory, dinas from the Krasogorovski factory, and earthenware from the Budjanski factory, these being extracted with water after being heated at various temperatures, and the electrical conductivity of the solutions measured. With Tschasov-Jarski clay, heating at 900° corresponds with the maximum formation of water-soluble products. With Nikolaevski clay the maximum solubility is obtained by heating at 750°, and with both this and the preceding clay the solubility is increased by boiling the material in water after calcination. Somewhat different results are obtained with the other materials. The varying behaviour is ascribed to chemical reactions between the components during the heating.

T. H. POPE.

Properties of various alumina-clay mixtures under various firing conditions. H. SALMANG and F. GOETH (Sprechsaal, 1929, 62, 503—506, 521—523, 539—543; Chem. Zentr., 1929, ii, 1728).—The behaviour of various mixtures of alumina and clay, when fired under load at 1410°, 1500°, or 1650°, was examined. In ordinary firing the porosity increases with increasing alumina content. Diminution of porosity on firing under load is least when the composition approaches that of mullite, the same composition corresponding with maximum stability. The softening point could be raised to 1630°. Scorification depended only on porosity. In kaolin-alumina mixtures the highest softening point was shown at the composition of sillimanite.

A. A. ELDRIDGE.

See also A., Feb., 150, **Solubility of quartz and clay in felspar** (PARMELEE and AMBERG). 156, **Reddening of glass containing gold and its birefringence** (TAMMANN and SCHRADER). 162, **Ceramics of refractories: system zirconia-beryllia** (RUFF and others). **Systems** $\text{BaO-Al}_2\text{O}_3\text{-SiO}_2$, $\text{ZnO-Al}_2\text{O}_3\text{-SiO}_2$, $\text{KNaO-felspar eutectic}$, and $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ eutectic (ZIMMER and NEFF). **Influence of alumina and silica on** ($\text{K}_2\text{O-Na}_2\text{O-Al}_2\text{O}_3\text{-SiO}_2$ eutectic (BUTTERWORTH and KATES). 183, **Determination of alumina in aluminosilicates** (KRASNOVSKI). 186, **Hygroscopic properties of glass** (SHISKAKOV).

PATENTS.

Substances transparent to ultra-violet light. CORNING GLASS WORKS, Assecs. of H. P. HOOD (B.P. 298,908, 15.10.28. U.S., 15.10.27).—Samples of commercial ultra-violet light transmitting window-glass artificially "solarised" by exposure to radiation from a quartz-mercury arc were restored to their original ultra-violet transmission values by heating at 550° for 15 min., or at 400° for 1 hr.

M. PARKIN.

Decoration of enamelled ceramic and metalised articles. "SFINX" SPOJENÉ SMALTOVNÝ A TOVARNÝ NA KOVOVE ZBOŽÍ, AKC. SPOL. (F.P. 634,669, 19.5.27. Czechoslov., 8.6.26).—The articles, after drying but before firing, are sprayed with aqueous solutions of suitable salts to produce the desired colours on firing.

A. R. POWELL.

Fusing metal particles into glazes and ceramic products. W. DÄLLENBACH (Swiss P. 124,309, 15.9.26).—The metal particles are first thinly coated with chromium or an alloy containing it.

L. A. COLES.

Fusible ceramic product for moulding. R. MOHR and H. BECKER, SEN. (G.P. 458,509, 27.11.26).—Fusible silicates, e.g., phonolite, are fused with calcium fluoride and, if necessary, aluminium, alkali, or alkaline-earth compounds, or minerals containing them. Colouring materials such as metal oxides or substances which promote crystallisation, e.g., loam or kaolin, may also be added.

A. R. POWELL.

Reactive packing for metallurgical refractories. C. S. HOLLANDER and E. L. HELWIG (U.S.P. 1,741,128, 24.12.29. Appl., 6.2.29).—To prevent escape of a charge, owing to refractory failure, from a crucible furnace, where such escape is liable to cause serious damage, e.g., to coils in electric induction furnaces, the

crucible is surrounded by a packing consisting of a mixture capable of reacting with the escaping charge to form a viscous mass at the prevailing temperature. E.g., for a clay-graphite retort containing zinc, the packing consists of 4 pts. of finely-ground silica and 1 pt. of sodium phosphate.

M. PARKIN.

Metallic deposits [on glass] (B.P. 324,363).—See X.

IX.—BUILDING MATERIALS.

Cements and moulding masses and their use in electrotechnics. W. NAGEL and J. GRÜSS (Wiss. Veröff. Siemens-Konz., 1929, 8, [2], 174—186).—The change in weight and the percentage alteration in volume during storage for 3 months have been determined for numerous magnesium oxychloride cements made from chemically-prepared magnesia and from burnt magnesite. The latter, in general, gave the most satisfactory cements, the best ratio of $\text{MgO}:\text{MgCl}_2$ being 2—10:1. Above or below this ratio there is a tendency for the cement to swell and become distorted or porous. The ageing process is somewhat retarded by the addition of inert fillers, but ceases sooner, and the fillers prevent any segregation of the constituents taking place. Zinc oxychloride cements with a composition approximating to $5\text{ZnO}, \text{ZnCl}_2$ are remarkably free from volume changes after a slight contraction during the first 5 days. The most resistant of the magnesium oxychloride cements to atmospheric disintegration is that with the composition $4\text{MgO}, \text{MgCl}_2$ containing a suitable filler.

A. R. POWELL.

Manufacture of anhydrite cement. P. P. BUDNIKOV (J. Appl. Chem. Russia, 1929, 2, 389—393).—The best catalyst is a mixture of sodium hydrogen sulphate and copper sulphate. Anhydrite cement can be regenerated without further addition of catalysts. Lime reduces the strength. Variations in the strength with time are probably due to the formation of an unstable complex hydrate.

CHEMICAL ABSTRACTS.

Influence of magnesia, ferric oxide, and soda on the temperature of liquid formation in certain Portland cement mixtures. W. C. HANSEN (Bur. Stand. J. Res., 1930, 4, 55—72).—The influence of ferric oxide, magnesia, and soda on the temperature at which melting starts when they are added separately or together to mixtures of lime, alumina, and silica has been determined. Mixtures of lime, alumina, and silica approximating to the composition of Portland cement start to melt at 1455°. When ferric oxide is added the temperature of liquid formation is reduced to 1340°; with magnesia, to 1375°; with soda, to 1430°; with ferric oxide and magnesia, to 1300°; and with all three compounds, to 1280°. The following compounds were identified in the clinkers: $3\text{CaO}, \text{SiO}_2$, $2\text{CaO}, \text{SiO}_2$, $3\text{CaO}, \text{Al}_2\text{O}_3, \text{MgO}$, and $4\text{CaO}, \text{Al}_2\text{O}_3, \text{Fe}_2\text{O}_3$.

C. J. SMITHELLS.

Mixed Portland cements. V. S. NAGAI (J. Soc. Chem. Ind., Japan, 1929, 32, 373—374 B).—Results of studies of physical properties of mortars prepared from a new mixed Portland cement "Neo-solidit" are described.

F. J. WILKINS.

See also A., Feb., 161, **Dehydration of calcium sulphate** (YAMANE).

PATENTS.

Heat-insulating material. A. C. D. BOULANGER (F.P. 636,263, 20.6.27).—The material comprises the ash of burnt coal-dust, which is applied in admixture with sodium silicate solution or is vitrified and cut, *e.g.*, into rings for covering pipes for conveying superheated steam. L. A. COLES.

Intimate admixture of materials [e.g., the constituents of Portland cement]. WICKING'SCHE PORTLAND CEMENT- U. WASSERKALKWERKE (G.P. 458,637, 4.6.25).—The coarsely mixed material is passed repeatedly through a compressed-air or suction conveyor which returns it to the container. L. A. COLES.

Production of building material. E. A. LUNDIN, Assee. of E. W. TILLBERG (Swed.P. 60,338, 23.12.24).—The dust and smaller particles separated from broken-up brick waste are ground finely and mixed with lime and/or Portland cement and then with the coarser particles of the brick, and the mixture, moulded into blocks, is treated with steam under pressure. L. A. COLES.

Production of artificial marble. V. MOLINARI (F.P. 636,140, 8.10.26).—Moulded blocks of gypsum or alabaster, after a rapid drying, are dipped successively into solutions containing alum, potassium sulphite, and, *e.g.*, zinc, iron, or copper sulphate, and are then polished. L. A. COLES.

Wood preserving and disinfecting, and seed-grain immunising. A. CARPMIEL. From I. G. FARBENIND. A.-G. (B.P. 321,396, 1.6.28).—An aromatic mercuri-compound is applied, for the preservation and disinfection of wood or for the immunisation of seed, either in intimate mixture with an inert filter (talcum, kieselguhr), or in aqueous solution or suspension. Suitable compounds are phenyl, tolyl, or naphthyl mercuriacetate, mercurated nitrobenzene, anisole, or ethyl *p*-aminobenzoate, and toluene-*p*-sulphonyl or salicylsulphonyl derivatives of aniline mercuriacetate. Arsenical compounds may be added. C. HOLLINS.

Drying or treating wood and other materials. H. C. BABEL (B.P. 324,333, 18.10.28).—See U.S.P. 1,687,822; B., 1929, 56.

[Mixing] treatment of concrete. CLINTON MOTORS CORP. (B.P. 308,207, 25.2.29. U.S., 19.3.28).

Apparatus for manufacturing bituminous compounds. A. H. TYLER (B.P. 324,424, 2.11.28).

Dehydration (B.P. 323,740).—See I. Emulsions of tars etc. (F.P. 633,687).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Eutectic cast iron. B. OSANN (Giesserei, 1929, 16, 565—567; Chem. Zentr., 1929, ii, 1736).—The eutectic point can be determined by slowly cooling and expressing the fluid portion immediately before solidification. The expressed material is then analysed. The formation of the eutectic is considered theoretically.

A. A. ELDRIDGE.

Superheating of cast iron. P. BARDENHEUER and K. L. ZEYEN (Giesserei, 1929, 16, 733—746; Chem. Zentr., 1929, ii, 1736—1737).—For iron containing not

less than 3.2% C, superheating on fusion increases the strength; with lower carbon content increase of superheating is accompanied by diminished resistance to bending (and, if little phosphorus and sulphur are present, also to drawing). Alloys containing boron, chromium, etc. are insensitive to heating. A. A. ELDRIDGE.

Works' supervision in the manufacture and working of special steels and in carrying out the necessary tests. R. HOHAGE (Stahl u. Eisen, 1930, 50, 93—97).—The selection of the raw materials, the melting and casting processes, and the methods used in further working of special steels are briefly described, together with methods of testing the quality of the metal at various stages of the work and precautions to be taken to ensure that satisfactory properties are obtained. A. R. POWELL.

Effect of cold-drawing on the magnetic properties of a carbon steel. W. S. MESSKIN (Arch. Eisenhüttenw., 1929—1930, 3, 417—425; Stahl u. Eisen, 1930, 50, 105—106).—The remanence of a plain carbon steel with 0.78% C decreases considerably with light rolling, but increases again with heavier rolling; the maximum permeability changes similarly, but with a greater reduction than 30% it decreases again. The coercivity increases steadily during rolling to about double its original value when the thickness has been reduced 80.8%; on subsequently annealing, the coercivity decreases with rise of temperature rapidly to 450° and then somewhat more slowly. Maximum remanence is obtained after annealing at 600°; this value is greater than that measured before rolling. Cold-deformation of steel has a similar effect on the magnetic properties to that of quenching; *e.g.*, on annealing the quenched steel at 450° a secondary maximum of coercivity is obtained similar to that produced by annealing heavily cold-rolled steel at the same temperature. In a molybdenum steel with 0.96% C and 2.18% Mo, the same but less well-defined phenomena were observed. A. R. POWELL.

Quenched steels. K. TAMARU (Sci. Rep. Tôhoku Imp. Univ., 1929, 18, 473—490).—Magnetic observations of the A0 transformation of a 0.95% C steel showed that its magnitude varies with the form of the cementite particles, being greatest with pearlitic cementite and least with the globular form. Tempering a quenched high-carbon steel (1.69% C) results in the transformation of α - into β -martensite at 110°, of the retained austenite through α - into β -martensite at 240°, and finally of β -martensite into ferrite and cementite at 260°. The maximum in the magnetisation curve at 300—350° corresponds with the complete separation of cementite from martensite in a finely-divided and almost colloidal state; at a slightly higher temperature coagulation of the cementite occurs and the magnetisation curve falls. A similar fall in specimens very slowly heated at 110° is attributed to the same cause. The rate at which specimens etch in a 5% alcoholic solution diminishes during the transformation of α - into β -martensite. The density of pure iron, extrapolated from a series of curves showing the density of steels of varying carbon content after different heat-treatments, is 7.882, to which point all the curves converge. Similarly, the density of pure

γ -iron, extrapolated from curves for austenitic steels, is 8.10, and decreases linearly by 0.06 for every 0.5% C and by 0.017 for every 2% Mn present. The change of length of quenched steel during tempering and the electrical resistance at 20° of austenitic steels containing 2–20% Mn have been measured. A. R. POWELL.

Nature of martensite crystals. K. HONDA (Sci. Rep. Tôhoku Imp. Univ., 1929, 18, 504–516).—A review of recent work and a discussion of some of the modern theories dealing with the nature of martensite. Matsushita's theory with some slight modifications (cf. Tamaru, preceding abstract) is considered to be more in accord with the facts than that of Hanemann and Schröder (Arch. Eisenhüttenw., 1928–29, 2, 763).

A. R. POWELL.

Steel ingots. W. H. HATFIELD (Proc. South Wales Inst. Eng., 1930, 45, 523–562).—A recapitulation of the practice of casting steel ingots, including methods for diminishing heterogeneity and segregation.

C. A. KING.

Density of some iron alloys in the liquid state. C. BENEDICKS (Compt. rend., 1930, 190, 114–116. Cf. B., 1924, 1014, and Berlin, B., 1927, 487).—The method is that previously used. Densities have been determined at various temperatures up to 1700° and the results are expressed by (i) sp. vol.-temperature curves for alloys with carbon, nickel, and silicon; (ii) a sp. gr.-composition curve for iron-carbon alloys: this shows a discontinuity at 4.4% C, corresponding with that in the liquidus; (iii) sp. vol.-composition curves for alloys with aluminium, carbon, chromium, molybdenum, nickel, phosphorus, silicon, and tungsten at 1600°, and for iron-nickel alloys also at 1500° and 1550°, at which temperatures these alloys, in general, show specific volumes in excess of those deduced from the additive law, the maximum excess corresponding to the composition Fe₂Ni, i.e., approximately that of Invar. Alloys containing 26–40% Ni show negative dilatation, with apparently a maximum sp. vol. at about 1600°.

C. A. SILBERRAD.

Abnormal rusting. F. WILBORN (Farben-Ztg., 1930, 35, 945).—Wolff's observations (B., 1929, 988) on the dissolution of iron by paint films, the iron plate remaining bright, are confirmed in the case of a nitro-cellulose lacquer and a Spanish red oxide-boiled oil paint. In the latter case blisters, which were formed after immersion of the test panel, were broken and the metal surface was examined. Photomicrographs show that the iron is practically free from corrosion and that rust is imbedded in the paint film. S. S. WOOLF.

Water-works' corrosion problems. I. D. VAN GIESEN (J. Amer. Water Works' Assoc., 1930, 22, 36–48).—Open, steel equalising tanks are subject to severe corrosive conditions as the top layer of water rarely changes, there being but a single bottom connexion. The effect is worst on the inside portion facing south, as this receives the full heat of the sun besides being alternately wet and dry. The corrosion may be reduced by covering the tank and by the use of a suitable paint, but a water-emulsified asphalt paint is not recommended owing to the effect of heat on the air particles entrained therein unless emulsified

under vacuum. Cases of corrosion in cast-iron mains are shown to be due to the crystallisation of combined carbon into the form of graphite in the presence of a highly ionised soil. Mutual corrosion (of dissimilar metals) may be prevented by using the same metal throughout, or, if this be impossible, by coating the pipes with asphaltum compound for several feet in both directions from the point of metal change. The difference in corrodibility between cast iron and wrought iron is emphasised. C. JEPSON.

Effect on the recrystallisation of copper of a second metal which forms a limited series of solid solutions and induces age-hardening. O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [2], 157–173).—The recrystallisation temperature of cold-rolled copper (70% deformation) is increased by about 100° by the addition of 0.05–0.1% of zinc, aluminium, phosphorus, silicon, tin, magnesium, or beryllium. Addition of larger quantities of these elements within the limit of solid solubility does not further affect the temperature at which recrystallisation begins, but it raises the final temperature to which it is necessary to heat the metal to obtain complete recrystallisation. The rate of reduction of hardness at the minimum recrystallisation temperature is greater for the four first-named alloying constituents than for the other three. In the case of copper with 2% Mg, 3% Si, or 2% Be, the softening due to removal of work-hardness at 350° is more than counterbalanced by age-hardening effects.

A. R. POWELL.

Effect of recrystallisation on the technical behaviour of lead. F. BRENTHEL (Z. Metallk., 1930, 22, 23–25).—Chemical analysis of lead is not sufficient to determine whether the metal is suitable for use in chemical plant as the crystalline structure has far more influence on the behaviour of lead towards acids than has its composition. Thus lead refined by the Parkes process recrystallises rapidly at 180°, and its structure then consists of large, regularly oriented crystals; sulphuric acid attacks this lead very vigorously at 160–240°, the attack proceeding inwards along the grain-boundaries so that the metal soon disintegrates. Lead refined by the Pattinson process, however, always contains small quantities of copper, and its structure remains stable, homogeneous, and finely crystalline at 180°; attack on this lead by sulphuric acid does not commence until 300°, and disintegration does not occur until well above this temperature. Pattinson lead is also very resistant to failure by fatigue, whereas Parkes lead soon recrystallises when subjected to constant vibration, and intercrystalline fracture then rapidly ensues.

A. R. POWELL.

Influence of temperature on the impact strength and hardness of aluminium alloys. W. SCHWINNING and F. FISCHER (Z. Metallk., 1930, 22, 1–7).—The hardness of 99.5% aluminium and of 98/99% aluminium falls with increasing temperature from –80° to 200° in a smooth curve from 50 and 45 to 25 and 20, respectively, for 1-mm. sheet, whereas that of lual falls from 105 at –80° to 99 at 40°, 80 at 130°, and 55 at 200°. At the ordinary temperature aluminium regains its original hardness after heating, whereas the hardness of

lantal slowly falls with increase of the temperature at which it has been heated. The actual hardness figures vary somewhat with the thickness of the sheet. The impact strength of bars with a 45° rounded notch decreases between -40° and 0°, then increases with further rise of temperature for scleron, and falls very slightly for constructal and lantal, but more rapidly for duralumin. These results are compared graphically with those obtained for a 56% Cu brass and a 0.51% C steel. The angle to which lantal can be bent round a rod without breaking increases in a smooth curve with the temperature, and is the greater the higher is the ratio of the rod diameter to the thickness of the sheet. The effect of ageing at the ordinary temperature on the bending and impact tests is not very marked in the case of lantal.

A. R. POWELL.

Commercial chromium-plating. R. SCHNEIDERWIND (Dept. Eng. Res., Univ. Michigan, Circ. Series No. 3, 1930, 60 pp.).—Technical information necessary for the design and operation of commercial chromium-plating baths is given, and the process of deposition of chromium from chromic acid baths is discussed. Equipment is considered under the headings of motor generators, rheostats, tanks, fume-exhausting apparatus, temperature-control devices, racks, contacts, and anodes. The effects of variations in plating conditions and solutions are discussed in detail. Notes on throwing power, stripping, inspection, defects, and costs are also included.

C. J. SMITHELLS.

Throwing powder in chromium-plating. H. L. FARBER and W. BLUM (Bur. Stand. J. Res., 1930, 4, 27—53).—It is shown on general principles that there is little hope of radically improving the throwing power of chromic acid baths used in chromium plating. The conditions giving the best throwing power have been investigated, and the following are recommended: (a) a high temperature, such as 55°; (b) a high current density, such as 35 amp./dm.²; (c) a low concentration of chromic acid, such as 150—250 g./litre; (d) a low sulphate content, such as CrO₃/SO₄ = 200. These conditions require about 6 volts, and for lower voltages a more concentrated solution should be used with lower temperature and current density.

C. J. SMITHELLS.

Electrodeposition of nickel. I. Effect of p_H and of various buffering agents; the presence of oxygen in the deposits. J. B. O'SULLIVAN (Trans. Faraday Soc., 1930, 26, 89—93).—Nickel deposited by electrolysis from buffered nickel sulphate solutions becomes smoother and finer-grained as the p_H of the solution is increased. This cannot be explained by assuming that the simultaneous evolution of hydrogen prevents the growth of large crystals (Kohlschütter, B., 1924, 300), or that the metal is initially deposited in an unstable form which rapidly changes into a more stable modification (Glasstone, A., 1927, 24), but may be accounted for by the simultaneous deposition of colloidal nickel hydroxide or basic salt (cf. Kohlschütter and Schacht, A., 1913, ii, 287). This view receives support from the observation that the deposit contains a small amount of oxygen.

R. CUTHILL.

Electrolytic deposits on aluminium and its alloys. BALLAY (Compt. rend., 1930, 190, 305—308).—

The mechanical or chemical etching of the surface of aluminium or its alloys, which is necessary to bring about the satisfactory adherence of an electrolytic deposit, results in difficulties when the plated surface has to be polished. A process is described for nickel-plating the aluminium, thus forming a surface on which other metals may then be readily deposited. The surface is prepared by dipping the aluminium or its alloy in a hydrochloric acid solution of ferric chloride at the b.p. or nearly so. A slight deposit of iron is thus obtained, and, on subsequently plating with nickel in a hot bath at a high current density, a strongly adherent deposit can be rapidly prepared. Iron solutions suitable for all alloys are comprised within the following limits: Fe, 6—22 g./litre; HCl, 0.10—0.70 g.-mol./litre.

M. S. BURR.

Making the cuprous oxide film on the surface of copper [plates]. Its efficiency of rectification [of electric currents]. Y. KATO and T. MURAKAMI (J. Soc. Chem. Ind., Japan, 1929, 32, 339—340 B).—By heating the plates at 800—1026° in air, they acquire a film of cupric oxide which subsequently becomes reduced by the metal in contact therewith to cuprous oxide. The rectifying elements thus obtained have a ratio of the electrical resistance in one direction to that in another of at least 300 : 1.

F. J. WILKINS.

Recovery of austenitic steels. A. MICHEL and P. BÉNAZET (Rev. Mét., 1929, 26, 455—463).—See B., 1929, 358.

Boiler plugs. FREEMAN and others.—See I.

See also A., Feb., 147, **System cadmium-antimony** (HALLA and ADLER). **System chromium-carbon** (KRAICZEK and SAUERWALD). **Silver-zinc alloys** (PETRENKO and PETRENKO). 148, **Platinum-iridium alloys** (NEMILOV). **Rhodium-bismuth alloys** (RODE). **Palladium- and gold-antimony alloys** (GRIGORIEV). **Iron-beryllium and -boron alloys** (WEVER). **System iron-manganese** (SCHMIDT). **Nickel-copper alloys at low temperature** (KRUPKOWSKI and DE HAAS). 149, **Silver-copper alloys** (WEINBAUM). 161, **System copper-zinc** (RUER and KREMERS). **System copper-oxygen** (VOGEL and POCHER). 176, **Pure powdered mercury** (KRAUSS and MÄHLMANN). 177, **Extraction of rare earths from gadolinite** (PEARCE and HARRIS). 183, **Spectroscopic analysis of manganese ores** (PIÑA DE RUBIES and DORRONSORO). **Determination of tungsten** (DOTREPPE).

PATENTS.

Preparation of slimes derived from wet washing of [blast-furnace] gas for sintering. A. J. BOYNTON, Assr. to H. A. BRASSERT & Co. (U.S.P. 1,738,940, 10.12.29. Appl., 27.10.27).—The sludge obtained by the wet washing of the gases from the iron blast furnace is filtered and the cake dried without crushing. The dry mass is then broken through a ½-in. screen and sintered directly after addition of about 14% of moisture, which causes the mass to swell to a maximum volume.

A. R. POWELL.

Roasting of zinc blende. BALZ-ERZÖSTUNG GES. M.B.H. (B.P. 309,476, 11.10.28. Austr., 11.4.28. Add. to B.P. 303,055; B., 1929, 923).—In a multiple-hearth

furnace with hollow hearths alternately fixed and movable, a pipe is provided for withdrawing gases from the lower hearths and returning them to upper hearths or to the bottom of the kiln at a velocity sufficient to penetrate into the ore layers. Means are provided for introducing fresh air into the pipe system if required.

A. R. POWELL.

Melting furnace for readily fusible metals [e.g., zinc]. W. BUSS (G.P. 458,351, 4.11.25).—The furnace comprises a collecting hearth, heated with gas or oil burners, above which is a melting vessel provided with separately heated overflow tubes leading to the hearth and totally enclosed so that the metal in them does not come into direct contact with the heating gases.

A. R. POWELL.

Furnace [for treatment of scrap metal etc.]. F. MENNE, Assr. to H. F. HOEVEL (U.S.P. 1,739,250, 10.12.29. Appl., 7.6.28. Ger., 24.6.27).—An elongated furnace is described in which billets or boxes of material are placed across the hearth and progressed in steps. When stationary, the billets rest on outer rails composed of water-cooled tubes; when to be moved the inner rails rise, lift the billets, move them forward, and then fall and recede without the billets. B. M. VENABLES.

Annealing of wire- or band-rings of iron or another metal by inductive heating. R. ARPI and G. J. M. DAHLQUIST (B.P. 323,835, 24.10.28).—The rings are welded together in parallel or in series, and mounted to form one or more secondary coils in a transformer, which is so arranged that the primary is outside a cylindrical furnace chamber, and the portions of the core adapted for the mounting of the rings are in the shape of water-cooled, heat-insulated rods inside the furnace chamber. [Stat. ref.]

A. R. POWELL.

Manufacture of steel. T. F. BAILY (U.S.P. 1,739,278, 10.12.29. Appl., 11.3.26).—Iron and steel scrap is fed together with pig iron into the upper end of an inclined, rotating, cylindrical furnace fired by an oil burner arranged in an opening in the axis of the furnace at the lower or discharge end. This end of the furnace is lined with basic brick, whilst the central portion has an acid lining, and the upper portion a lining of wear-resisting metal. The charge is preheated by the waste gases in the throat of the furnace, melted in the acid-lined zone, and partially refined in the basic-lined zone under an oxidising flux which removes the phosphorus. The metal is then discharged into an electric furnace for the finishing processes.

A. R. POWELL.

Making ductile high-silicon steel sheets. L. R. CURRY, Assr. to ALLEGHENY STEEL CO. (U.S.P. 1,739,126, 10.12.29. Appl., 27.7.27).—Silicon steel containing more than 4% Si may be rolled readily into thin sheets if it is annealed at the rolling temperature for 1–4 hrs. prior to rolling.

A. R. POWELL.

Reconditioning of steel cutting-tools. HASSENFORDERER & CIE. (F.P. 634,370, 14.5.27).—Tools which have become unusable owing to overheating are hardened by immersion in a fused mixture of talc, fish-liver oil, colophony, borax, soot, sodium chloride, bone ash, and leather ashes.

A. R. POWELL.

Determination of the sharpness, cutting power,

and permanence of the cutting edge of blades, tools, etc. K. HONDA (G.P. 458,052, 25.12.26. Jap., 20.1.26).—The loaded tool is drawn backwards and forwards with a regular motion over a pile of thin sheets of paper or fabric. The number of sheets cut is a measure of the cutting power, and the number of movements required to reduce the cutting power to half its original value is a measure of the permanence of the edge. (Cf. B., 1927, 782.)

A. R. POWELL.

Metallic element [single-crystal iron rod]. L. W. MCKEEHAN, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,738,307, 3.12.29. Appl., 11.4.27).—A polycrystalline iron rod is passed slowly through an electrically heated tube furnace at 1400°, and is cooled rapidly to 200° at the exit end by means of steel rollers and by a current of cold hydrogen which is passed through the furnace in the opposite direction to the travel of the rod. In this way a very small portion of the rod is subjected at one time to a temperature of about 900°, at which grain growth of α -iron is rapid. The rate of travel of the rod is so adjusted that, as every portion passes through the zone at 900°, the crystals grow to such an extent that a single crystal occupies the whole cross-sectional area; this crystal then grows along the whole length of the rod. The optimum rate appears to be about 200 cm./hr. Single-crystal iron has a high permeability and a small hysteresis loop.

A. R. POWELL.

Production of finely-divided metallic materials. J. H. WHITE, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,739,052, 10.12.29. Appl., 31.1.29).—The material, e.g., iron or nickel-iron alloy, is melted together with a small quantity of the sulphide of one of its constituents, so as to obtain on casting a brittle ingot which may readily be reduced to fine powder. The process is especially suitable for preparing magnetic materials for dust cores.

A. R. POWELL.

Production of [metallic] materials in finely-divided form [for magnetic dust cores]. J. E. HARRIS, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,739,068, 10.12.29. Appl., 31.1.29).—The material, e.g., iron or an iron-nickel alloy, is melted with a small quantity of iron oxide or sulphide to render it brittle, then granulated in water, dried, and ground to a powder.

A. R. POWELL.

Corrosion-resistant ferrous alloy. J. T. HAY (B.P. 323,845, 30.10.28).—The alloy consists of iron with 0.4–0.5% Cu, 0.05–0.1% Mo, up to 0.05% C, 0.1–0.15% Mn, and less than 0.005% Si, 0.005% P, and 0.04% S. [Stat. ref.]

A. R. POWELL.

Treatment of [tin] ores. D. H. McINTOSH, Assr. to G. L. OLDRIGHT (U.S.P. 1,737,888, 3.12.29. Appl., 24.12.26).—Tin ores containing pyrites and other sulphides are heated first at 800–850° for about 3 hrs. and then at 1000° for at least 1 hr., both operations being conducted in a reducing atmosphere. The product is leached with sulphuric acid with or without the addition of ferric sulphate. When the ferrous sulphide has all dissolved, the residue is roasted in an oxidising atmosphere and the copper and lead are extracted by a brine leach, leaving a residue of almost pure cassiterite.

A. R. POWELL.

Production of zinc. R. LEPSON (U.S.P. 1,738,910, 10.12.29. Appl., 24.6.27).—Zinc is recovered from lead blast-furnace slag by melting the slag with 10% of its weight of coke in a continuously operating electric furnace of the submerged-arc type, whereby zinc and lead are volatilised and a silicon-iron alloy is obtained below a lime-ferrous silicate slag. The gases are withdrawn at 1100° or above, through a short tuyère situated just above the surface of the bath, into a large, rectangular, iron cooling chamber provided with numerous vertical, water-cooled pipes suspended from the roof. The gas stream is thus cooled rapidly to 300°, and the zinc is thereby deposited as a very fine powder on the outer surfaces of the cooling tubes, from which it is periodically detached by the aid of reciprocating plungers working loosely on the tubes. This fine zinc dust is especially suitable for use in the purification of electrolytic zinc liquors, for effecting precipitation of other metals from their solutions, and as a reducing agent in chemical operations. A. R. POWELL.

Concentration of [tin] ores. Flotation of cassiterite. (A, B) R. S. HANDY and (A) R. R. BEARD, Assrs. to PATINO MINES & ENTERPRISES CONSOL., INC. (U.S.P. 1,737,716—7, 3.12.29. Appl., [A] 13.9.28, [B] 13.11.28. Bolivia, [B] 5.12.27).—(A) The finely-powdered ore is agitated with 8 lb. of sodium silicate per ton and 28 times its weight of water. After sufficient settling, the supernatant colloidal suspension is removed and the crystalline residue subjected to flotation with pine oil in an acid circuit to remove sulphides. Tin is recovered from the tailings by flotation with 1 lb. of oleic acid and 0.2 lb. of cresol per ton. (B) Finely-ground tin ore is subjected to flotation in an alkaline circuit containing sodium carbonate, sodium silicate, and copper sulphate, using oleic acid or a soluble oleate, with or without an oil, as flotation agent. The concentrate contains the cassiterite and pyrites, the latter being subsequently removed by flotation in an acid circuit. A. R. POWELL.

Bearing [copper-tin-lead] alloy. H. M. WILLIAMS, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,738,163, 3.12.29. Appl., 11.4.27).—The constituents of the alloy in a finely-powdered form are thoroughly incorporated in a mechanical mixer, the mixture is formed into the desired shape by a pressure of 75,000 lb./in.², and the shapes are heated in an inert atmosphere for 3 hrs. at 775°, whereby the volatile constituents are removed, leaving a porous bearing which absorbs a high proportion of lubricant. Suitable mixtures comprise (a) copper 90, tin 8, lead 10, phosphor-tin 2, graphite 6, and salicylic acid 2—4 pts.; (b) copper 68, lead 25, graphite 5, and phosphor-tin 2%, with or without salicylic acid; or (c) copper 80, lead 10, graphite 8, and tin 10 pts., with 0.5 pt. each of phosphor-tin and ammonium chloride. The phosphor-tin prevents segregation of the lead during heating. A. R. POWELL.

[Aluminium] alloys. P. BERTHELEMY and H. DE MONTLY (F.P. 634,751, 20.5.27).—A homogeneous alloy of cadmium with aluminium is obtained if magnesium is added at the same time. The alloys are claimed to be resistant to corrosion by sea-water, Sorel cement, and thiosulphate. A. R. POWELL.

Acoustic castings of aluminium or its alloys. VEREIN. ALUMINIUM-WERKE A.-G. (G.P. 458,312, 19.10.24).—The acoustic properties of aluminium alloy bells, gongs, etc. are improved by quenching or slow cooling from a high temperature, followed by ageing at the ordinary temperature when magnesium is the alloying constituents, or at higher temperatures according to the nature of the alloy. A. R. POWELL.

Preparation of alkali metals and alkaline-earth metals. S. G. S. DICKER. From N. V. PHILIPS' GLOEI-LAMPENFABR. (B.P. 323,718, 3.8.28).—A suitable non-volatile, non-hygroscopic compound of the alkali or alkaline-earth metal is heated in a vacuum or an inert gas with finely-powdered titanium, zirconium, or hafnium, whereby the alkali or alkaline-earth metal is liberated. The process is especially applicable to the production of small quantities of caesium or barium inside vacuum tubes or radio valves to act as getters; e.g., a small pellet or rod of caesium sulphate, bisulphate, or dichromate with 4—20 times its weight of zirconium is heated at 300—500°, when an energetic reaction takes place with the liberation of 50—97% of the caesium. A. R. POWELL.

Production of calcium. W. KROLL (G.P. 458,493, 5.2.26).—An alloy of lead and calcium, obtained by heating lead with calcium carbide or with mixtures which produce calcium carbide, is made the anode in a bath of molten calcium salts. A. R. POWELL.

Purification of molten metals. R. WALTER (F.P. 634,436, 14.5.27. Ger., 18.5.26).—The molten metal is stirred under a flux comprising a mixture of alkali carbonate or chloride with 1—38% of calcium fluoride. Metals so treated are claimed to be free from gas inclusions and internal stress. A. R. POWELL.

Manufacture of [metals for production of] metal carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,382, 27.9.28).—Metal oxides are reduced by heating above 500° in the presence of hydrogen, carbon monoxide, hydrocarbons, etc., and, to prevent sintering, the metals, as soon as reduction is complete, are cooled rapidly in a non-carburising atmosphere preferably consisting of or containing hydrogen. L. A. COLES.

Manufacture of metallic deposits from metal carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,363, 12.10.28).—The carbonyl is introduced at low pressure into a vessel in which the article to be coated, e.g., a glass plate, is maintained at a temperature above that of decomposition of the carbonyl while the walls of the vessel are kept cold by water-cooling. To ensure better adherence the article to be coated may first be washed with a solution of potassium hydroxide or other cleansing agent. If the deposit is made on a soluble substance, e.g., a large crystal of sylvine, a thin transparent film of the metal is obtained by subsequent dissolution of this substance. The process is particularly adapted for the production of plane iron mirrors on glass and for the preparation of thin iron foil. A. R. POWELL.

Electrolytic process and apparatus [for silver-plating]. LANGBEIN-PFANHAUSER-WERKE A.-G. (G.P.

455,506, 5.11.26).—In order to obtain a thicker deposit of silver on the parts of plated articles which are most subject to wear, a large flat disc of metal, preferably silver, is placed in the solution facing the anode and the back of the disc is connected with a much smaller silver disc by means of a metal conductor rod, the back of both discs and the connecting rod being insulated from the solution, so that the current passing through the electrolyte to the larger disc is concentrated in the smaller disc, which faces the part of the cathode to be plated more thickly, and hence a higher current density is applied to this part while a lower current density is applied to the neighbouring parts. A. R. POWELL.

Protection of metal parts in electrolytic baths. ULLSTEIN A.-G. (G.P. 458,284, 28.9.27).—The metal parts which are not to be plated, *e.g.*, heating coils, are made anodic to the cathode by means of an opposed *E.M.F.* just equal to the direct *E.M.F.* between the cathode and the part to be protected. A. R. POWELL.

Ceramic vessel for chromium-plating baths. Electroplating with chromium. CHROM-IND. M. WOMMER (F.P. 635,699 and 635,700, 9.6.27. Ger., [A] 9.6.26, [B] 7.12.26).—(A) The plating bath is provided with a hollow rim with perforations just above the surface of the liquid in the bath so that by coupling the rim with a suction tube dangerous vapour and spray may be removed from the surface of the bath. (B) Inorganic colloids, *e.g.*, silica or sodium silicate, are added to the usual chromium-plating bath. Bright, adherent deposits are thus obtained at current densities (up to 15 amp./dm.²) higher than usual.

A. R. POWELL.

Protective coatings of chromium. ELEKTRO-CHROM-GES.M.B.H. (G.P. 458,632, 9.8.24).—The metal is first electroplated with cadmium, zinc, or lead, then with chromium. The intermediate layer protects the metal from penetration of corrosive media through imperfections in the chromium plate. A. R. POWELL.

Production of compact chromium deposits from molten electrolytes. H. SCHMIDT (G.P. 458,494, 1.9.26).—The bath consists of a chromium compound dissolved in a molten compound of an alkaline-earth metal, aluminium, magnesium, zinc, or manganese, together with an alkali borate.

A. R. POWELL.

Electroplating on aluminium and its alloys. E. P. M. GAT and E. M. L. CARRIÈRE (F.P. 635,002, 25.5.27).—The cleaned metal is plated first with a thin layer of iron in a ferrous sulphate bath, then with nickel or other suitable metal in the usual plating bath.

A. R. POWELL.

Electrolytic deposition of alloys. B. LEECH and F. HAMMOND (B.P. 323,765, 4.7.28).—Pulverulent iron-nickel alloys may be produced electrolytically on a nickel cathode by electrolysis of a slightly acid chloride solution of the two metals, using a composite anode composed of nickel, iron, and graphite in such proportions that the nickel content of the solution remains constant while the iron content gradually increases. A nickel-iron alloy containing 78.7% Ni is always produced from a bath in which the nickel-iron ratio is 2—5:5—16. This alloy behaves as a single metal

when used as anode in an electrolytic cell, and hence may be deposited on other metals in a smooth coating which is highly resistant to corrosion. From sulphate solutions of tin and copper similar alloys containing 80% or 85.72% Sn may be produced in powder form by suitable adjustment of the conditions.

A. R. POWELL.

Direct production of steel or steel alloys from titaniferous ores and iron sands. F. A. DE SILVA and C. G. CARLISLE (U.S.P. 1,745,360, 4.2.30. Appl., 30.7.27. U.K., 18.5.27).—See B.P. 290,442; B., 1928, 527.

Purification of metals. F. WEVER, Assr. to KAISER-WILHELM INSTITUT F. EISENFORSCHUNG (U.S.P. 1,745,464, 4.2.30. Appl., 18.1.27. Ger., 27.1.26).—See B.P. 265,213; B., 1928, 489.

Jacketed metal drums. F. KRUPP A.-G. (B.P. 310,531, 22.4.29. Ger., 28.4.28).

Thermal reactions (F.P. 569,342).—See I. **Treatment of pickling liquors** (G.P. 458,191).—See VII. **Decoration of metallised articles** (F.P. 634,669). **Packing for metallurgical refractories** (U.S.P. 1,741,128).—See VIII. **Separation of materials** (B.P. 312,684).—See XI.

XI.—ELECTROTECHNICS.

High-voltage phenomena in insulating oil. T. NISHI and K. OHTSUKA (Bull. Inst. Phys. Chem. Res., Tokyo, 1929, 8, 999—1013).—The charge on the surface of glass plates immersed in four qualities of transformer oil has been studied.

R. A. MORTON.

Cements and moulding masses. NAGEL and GRÜSS.—See IX. **Chromium-plating.** SCHEIDEWIND; FARBER and BLUM. **Electrodeposition of nickel.** O'SULLIVAN. **Electrodeposits on aluminium and its alloys.** BALLAY. **Copper plates for rectifying.** KATO and MURAKAMI.—See X.

See also A., Feb., 141, **Magnetic nickel-manganese alloys** (KAYA and KUSSMANN). 183, **Electro-analysis of copper** (GUZMÁN and RANCAÑO).

PATENTS.

[Electromagnetic] separation or sorting of solid materials. F. KRUPP GRUSONWERK A.-G. (B.P. 312,684, 23.4.29. Ger., 31.5.28).—A number of devices making use of the differing electrical conductivity, dielectric constant, permeability, or other quality of a constituent of a mixture are described. The thermionic valves, discharge tubes, or other primary relays actuated by the very feeble currents produced as the materials to be sorted (*e.g.*, raw mining products) are brought piece by piece within the range of influence of the electric or electromagnetic fields are arranged in unstable "tipping" connexion.

B. M. VENABLES.

Electrical purification of gases. ELEKTR. GAS-REINIGUNGS-GES.M.B.H., and H. ROHMANN (G.P. 458,876, 14.10.22).—All the spark-discharge electrodes in the apparatus are operated with the same voltage, but by varying the thickness of the wires, the sharpness of the points, and the temperature of the electrodes the intensity of the discharge is increased from the front of the apparatus to the back to correspond with the decreasing

concentration of the dust in the gases as they pass through the precipitating chamber. A. R. POWELL.

Electrode for electrical purification of gases. H. ZSCHOCKE (G.P. 458,948, 10.5.24).—The electrode consists of a central wire provided with a series of small cylindrical or conical conducting bodies regularly disposed along it. These bodies protect the wire from corrosion, from damage by arcing, and from becoming coated with impurities, and act as discharge points for the high-tension current, not by point action, but by reason of the well-known corona effect. A. R. POWELL.

Inductor furnace. E. F. NORTHRUP, ASSR. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,744,983, 28.1.30. Appl., 2.8.28).—See B.P. 316,659; B., 1930, 245.

Electroplating apparatus. J. G. NEWBY and C. B. JERRED (U.S.P. 1,744,792, 28.1.30. Appl., 20.9.27. U.K., 9.10.26).—See B.P. 279,616; B., 1928, 22.

X-Ray installations. N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 309,997, 8.4.29. Holl., 19.4.28).

[Gas-filled, multi-filament] electric incandescence lamps. GEN. ELECTRIC CO., LTD., and J. P. BOWEN (B.P. 324,390, 25.10.28).

[Vent for] electric accumulators. CHLORIDE ELECTRICAL STORAGE CO., LTD. (B.P. 324,533, 5.2.29).

Conducting chemical reactions (G.P. 458,756).—See I. **Hypochlorite solutions** (F.P. 635,654). **Artificial mica** (G.P. 458,475).—See VII. **Packing for metallurgical refractories** (U.S.P. 1,741,128).—See VIII. **Annealing iron rings** (B.P. 323,835). **Finely-divided metallic materials** (U.S.P. 1,739,052 and 1,739,068). **Alkali or alkaline-earth metals** (B.P. 323,718). **Calcium** (G.P. 458,493). **Silver-plating** (G.P. 455,506). **Electrolytic baths** (G.P. 458,284). **Chromium-plating** (F.P. 635,699 and 635,700; G.P. 458,632 and 458,494). **Plating on aluminium** (F.P. 635,002). **Deposition of alloys** (B.P. 323,765).—See X.

XII.—FATS; OILS; WAXES.

Laurel fat (*Oleum lauri*). A. HEIDUSCHKA and J. MÜLLER (Arch. Pharm., 1930, 268, 114—128).—An investigation has been made of the fatty acids contained in laurel fat. The following constants of the commercial fat are new or differ considerably from the recorded figures: $[\alpha]_D^{20} +6.6^\circ$, acid value 15.3, ester value 190.7, acetyl values 19.7 (apparent) and 11.4 (true), volatile acids 0.034%, water 0.94%, ash 0.03%. After removal of essential oil (1.1%) by steam-distillation, the fat was hydrolysed with alcoholic potassium hydroxide, acidified, and chlorophyllins were separated by treatment with bicarbonate solution. The fatty acids had $d_4^{15} 0.9075$, m.p. 25—26°, setting point 20.18°, butyrefractometric index 44.4 at 25°, $n_D 1.4496$, $[\alpha]_D^{20} +3.23^\circ$, neutralisation value 225.2, mol. wt. 249.2, iodine value 66.36. The mixture was esterified with alcohol and sulphuric acid and the ethyl esters were fractionally distilled under 2 mm. pressure. No fatty acids lower than lauric acid were present. The laurate was readily separated and was followed by a mixture of palmitate, oleate, and linoleate. The last-named was analysed by the lead salt-ether method, and α -linoleic acid was

determined by the bromide method. The composition of the fatty acids deduced from this was: lauric acid 30.2, palmitic 11.1, oleic 39.8, α -linoleic 11.0, and β -linoleic acid 7.9%. It was shown definitely that myristic acid was absent, and no more than a trace, if any, of stearic acid was present. R. K. CALLOW.

Action of silent discharge on oils, fats, and fatty acids. II. **Effects of gaseous media.** I. Y. IWAMOTO (J. Soc. Chem. Ind., Japan, 1929, 32, 259—260 B).—Oleic, linoleic, and linolenic acids when exposed to a silent discharge of 10,000 volts in gaseous media are attacked the more severely the smaller is the mol. wt. of the gaseous medium. Hydrogenation and polymerisation occur rapidly in hydrogen, slowly in nitrogen, and not at all in sulphur dioxide and carbon monoxide. As compared with the reaction in hydrogen, the rate of polymerisation in nitrogen is much greater than that of hydrogenation in this gas. S. K. TWEEDY.

Combined determination of water and fat in organic material. H. BULL (Tidsskr. Kjem. Berg., 1930, 10, 5—7).—The material (10 g.) is weighed out into a flask fitted with a graduated side tube, and boiled under reflux with 50 c.c. of trichloroethylene, the flask being inclined so that the expelled water collects in the graduated side tube. After reading off the volume of water in this, the solvent remaining in the tube is returned to the flask by cautiously inclining the latter, and an aliquot portion of the liquid is withdrawn by a pipette. The extract is evaporated in a weighed flask over a flame and the last traces of solvent are removed by evacuating the flask while heating it at 100°, admitting air, and then re-evacuating; after $\frac{1}{2}$ hr. the flask and extracted fat are weighed. If the material under examination is a powder the extract must be filtered before withdrawing the sample; this is preferably accomplished by inserting a thimble made from filter paper into the liquid in the flask, and pipetting off the clear liquid from its interior. A correction must be applied to the quantity of fat obtained, due to the increase in volume of the solution caused by the dissolved fat; for this purpose tabulated figures are given.

H. F. HARWOOD.

Comparison of the Hanus and Kaufmann methods for determination of iodine value. S. JUSCHKE-WITSCH (Chem. Umschau, 1929, 36, 385—386).—The Kaufmann method was found to give excellently concordant results, was convenient and rapid in use, and was cheaper than the Hanus method; the reagent also had good keeping properties. E. LEWKOWITSCH.

Density of fats, fatty acids, and mineral oils. K. K. JÄRVINEN (Suomen Kem., 1929, 2, 111—115).—Values of d for various fats, fatty acids, and mineral oils at 20—100° are recorded. For the liquids, d diminishes by about 0.007 for a rise in temperature of 10°.

R. CUTHILL.

Soya-bean oil extraction. I. M. MASHINO (J. Soc. Chem. Ind., Japan, 1929, 32, 256 B).—The residue after the extraction of soya-bean oil with an azeotropic mixture of hydrocarbons and lower alcohols is suitable for the raw material of foodstuffs. After extraction at 40—50° with a mixture of gasoline and methyl alcohol,

the azeotropic mixture separates into two layers: the upper, gasoline, layer contains soya-bean oil; the alcohol layer contains carbohydrates, phosphatides, colouring matter, and other impurities. The lowering of the separation temperature increased the concentration of the oil in the upper layer, which process is also aided by the water content of the soya bean.

C. C. N. VASS.

Chaulmoogra oil from the seeds of *Hydnocarpus heterophylla*. D. R. KOOLHAAS (Rec. trav. chim., 1930, 49, 109—111).—Extraction of the dry kernels with light petroleum gives a yellow oil, d_4^{25} 0.952, n_D^{25} 1.4679, $[\alpha]_D^{25}$ +43.1°, acid value 0.86, iodine value 73.3, saponif. value 194. Hydrolysis of the oil with alcoholic potassium hydroxide, purification of the potassium salts, esterification of the free acids, and subsequent fractional distillation and further hydrolysis affords hydnocarpic and chaulmoogric acids, m.p. 59° and 68°, respectively.

H. BURTON.

Oil from the seeds of *Butea frondosa*, Roxb. M. C. T. KATTI and B. L. MANJUNATH (J. Indian Chem. Soc., 1929, 6, 839—845).—The oil, obtained in 18% yield by extracting the crushed seeds with light petroleum, has d_4^{25} 0.8983, n_D^{25} 1.4650, f.p. 15°, saponif. value 178, iodine value 67.2, acetyl value 23.6, acid value 18.1, Hehner value 88.6, and Reichert-Meissl value 0.5. Hydrolysis of the oil with alcoholic potassium hydroxide solution, separation of the saturated (36%) and unsaturated (64%) acids by Twitchell's method (A., 1921, ii, 662), and subsequent purification through the methyl esters show the presence of oleic, linoleic, palmitic, lignoceric, and higher saturated acids. Sitosterol is present in the unsaponifiable matter (2.3%) of the oil.

H. BURTON.

Negative catalysts of the hardening of fatty oils. VIII. S. UENO and T. YUKIMORI (J. Soc. Chem. Ind., Japan, 1929, 32, 318—321 B).—Catalysis of the hardening of fatty (sardine) oils by nickel is retarded by the presence of minute quantities of nitric acid and ammonia; nitrogen, ethylene, methane, and carbon dioxide have no effect.

C. W. GIBBY.

Narcotic action of anæsthetics towards reducing catalysts [for the hydrogenation of fatty oils]. S. UENO (J. Soc. Chem. Ind., Japan, 1929, 32, 321—323 B).—The catalytic activity of platinum and nickel in the hydrogenation of sardine oils is reduced by treatment with anæsthetics.

C. W. GIBBY.

Determination of tung oil in stand oils. H. WOLFF, G. ZEIDLER, and J. RAKINOWICZ (Farben-Ztg., 1930, 35, 896—897).—The coagulation of the tung oil content of stand oils by iodine (McIlhiney, Marcusson) and by zinc chloride (Scheiber) does not afford a method of determining the tung oil. The chief sources of error are the frequent failure of the oil to coagulate, and the fact that linseed oil yields insoluble fractions under the stipulated conditions. No reliable method is at present available, but the determination of the refractive index (n_D^{40}) of the fatty acids isolated from the stand oil is considered to give more trustworthy figures than the coagulation method, the tung oil content being expressed by $(n - 1.4714)/0.162$. High results are given by thin

oils and bodied mixed oils, whilst thick and blended stand oils tend to give low results.

S. S. WOOLF.

Manufacture of higher alcohols and soaps from waxes. I. Odourless soap and wax alcohol from sperm-head oil. M. HIROSE (J. Soc. Chem. Ind., Japan, 1929, 32, 253—254 B).—The oil is saponified with sodium hydroxide at above 150°; addition of a small quantity of wax alcohol aids the reaction. The greater part of the wax alcohol is removed by distillation of the soap at 200° under 13 mm. The wax alcohol, which forms about 22% of the oil, consists chiefly of oleyl and cetyl alcohols. The residue is a hard, greyish-yellow, odourless soap, which contains no free alkali and dissolves almost clearly in warm water; its lathering power is good at low temperatures, but unsatisfactory at 40°.

C. C. N. VASS.

Insecticide soaps. VAN DER MEULEN and VAN LEEUWEN.—See XVI.

See also A., Feb., 259, Constituents of *Illicium religiosum* (CHEN).

PATENTS.

Manufacture of soap. G. LAPORTE (F.P. 577,923, 18.2.24).—Boiling coconut oil (60 kg.) is run slowly into a solution of 5—7 kg. of sodium hydroxide and 5 kg. of sodium carbonate in 45 kg. of water, the mixture is heated to the b.p., and 5 kg. of powdered colophony and a solution of 5 kg. of sodium chloride in 25 kg. of water are added; after boiling for 20 min. the product is run into moulds.

L. A. COLES.

Water-soluble hydrocarbon soaps. R. VIDAL (F.P. 634,983, 23.9.26).—Fats, oils, or fatty acids are saponified with alkali hydroxides and alkali hypochlorites or aldehydes; e.g., 10 kg. of oleic acid and 10 kg. of vaseline oil are made into a paste with 10 kg. of sodium hypochlorite solution (d 1.16) and 3 kg. of sodium hydroxide solution (d 1.33), whereby a semi-solid soap is obtained. The hypochlorite may be replaced by 1—1.5 kg. of 50% acetaldehyde solution; after 24 hrs. the soap formed will dissolve in water without separation of oily particles.

A. R. POWELL.

Refining of fatty oils. E. E. AYRES, JUN., and L. H. CLARK, Assrs. to SHARPLES SPECIALTY CO. (U.S.P. 1,737,402, 26.11.29. Appl., 15.7.22).—The fatty oils are heated to 60—80° and agitated with small amounts (1—5%) of water or dilute (e.g., 2%) solutions of starch, salt, or acid, whereby the impurities dissolved in the oil are "hydrated" and precipitated in the form of an insoluble sludge which is easily removed by settling or centrifuging. The addition of excess water, which is indicated by the appearance of free water globules in the sludge, should be avoided.

E. LEWKOWITSCH.

Treatment of talloel [tall oil]. W. SCHULTZE (U.S.P. 1,736,802, 26.11.29. Appl., 30.9.27. Ger., 11.10.26).—Tall oil is treated by such esterifying agents (e.g., alcohol and concentrated sulphuric acid) that the liquid fatty acids only are esterified; the resin acids therefrom are removed by adding aqueous caustic soda lye and extracting the esters with a solvent (benzene). The fatty esters may be purified from unsaponifiable matter by distillation and the fatty acids recovered

by saponification; the resin acids are recovered by acidification and extraction of the soap solution.

E. LEWKOWITZCH.

Apparatus for purifying oils and fats *in vacuo* by means of steam or the like. W. GENSECKE (U.S.P. 1,746,078, 4.2.30. Appl., 26.8.24. Ger., 18.9.23).—See B.P. 222,093; B., 1925, 556.

Wetting-out etc. agents (B.P. 289,841).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colour testing [of pigments]. C. D. O. WINSLADE (J. Oil & Colour Chem. Assoc., 1930, 13, 3–7).—Mainly a discussion of the testing of the colour of pigments in full strength and on reduction. The comparison of "self colours" is best done at brushing consistency, as stiff pastes may show colour variations with slight differences in fluidity. This is particularly marked in the case of Turkey-red oxides. In connexion with the use of white pigments, other than zinc oxide, for reduction of coloured pigments, it is pointed out that different tones are obtained with zinc oxide, titanium white, lithopone, and white lead, there being a trend from blue to yellow in the order indicated. The harmful influence of excessive water-soluble salts is stressed. S. S. WOOLF.

Amorphous and crystalline resins and varnishes. D. VORLÄNDER (Z. angew. Chem., 1930, 43, 13–16).—With reference to the behaviour of numerous complex organic compounds of known constitution which behave under certain conditions as resins or varnishes, it is shown that the factor which determines whether the substance solidifies in the crystalline or amorphous state is the arrangement and nature of the groups in the molecule. Any dissymmetry in this arrangement, such as the presence of side-chains, *m*- and *o*-substitution, or an uneven number of side-chains, tends to render more difficult the assumption of a crystalline state on solidification. The formation of varnishes by polymerisation and oxidation appears to be due to a decrease in the symmetry of the molecule which permits permanent supercooling or hardening of the amorphous mass. A. R. POWELL.

Hydrocarbons and a high-tension discharge. BECKER.—See II.

See also A., Feb., 215, **Resins: preparation of pure α - and β -amyirin** (HORMMANN).

PATENTS.

Manufacture of coloured varnishes. SOC. CHEM. IND. IN BASLE (B.P. 293,358, 4.7.28. Ger., 4.7.27).—Pre-chromed or pre-coppered azo dyes derived from *o*-aminophenols are incorporated in varnishes or lacquers having a basis of cellulose nitrate or phenol-aldehyde products. C. HOLLINS.

Nitrocellulose composition [for artificial leather]. J. C. EMHARDT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,737,364, 26.11.29. Appl., 21.9.26).—A homogeneous nitrocellulose composition suitable for use on leather coatings and free from solvent softeners contains as softeners for the nitrocellulose a blown vegetable oil, *e.g.*, castor, cottonseed, rapeseed oil, and a non-volatile mineral oil (2–5% of the total

mineral and vegetable oil content). The mineral oil prevents the stickiness normally induced by the vegetable oil. S. S. WOOLF.

Manufacture of artificial masses, lacquers, and solutions containing both cellulose esters and rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,258, 27.8. and 21.12.28).—Unsaturated aliphatic esters, especially ethyl crotonate, are used as ingredients in lacquers of low viscosity containing cellulose nitrate or acetate and rubber. A spraying lacquer is produced, *e.g.*, by mixing ethyl α -crotonate (70 pts.), butyl cinnamate (5 pts.), methyl oleate (12 pts.), cellulose nitrate (10 pts.), and rubber (3 pts.). C. HOLLINS.

Manufacture of condensation products of cyanamide and formaldehyde. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 319,822, 28.6.28).—Cyanamide, especially an aqueous extract of "nitrolin," is condensed with formaldehyde either (a) in neutral or alkaline solution or (b) at higher temperatures in presence of dilute acid. Two different products result. C. HOLLINS.

Old rubber [lacquer] (B.P. 323,526).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Causes of deterioration of ebonite when exposed to light and air. D. M. WEBSTER and B. D. PORRITT (India-rubber J., 1930, 79, 239–243; cf. B., 1929, 828).—The surface film of ebonite, containing no free sulphur, develops distinct traces of sulphurous and sulphuric acids after exposure to light in a damp atmosphere for 4 weeks, the acids resulting apparently from oxidation of hydrogen sulphide which arises from decomposition of the rubber-sulphur compound in the ebonite. The rate of evolution of hydrogen sulphide from ebonite heated in hydrogen is rapid at first, but rapidly decreases, finally becoming practically uniform over a long period. After being heated, the ebonite is reddish in colour and less saturated than the original material; it is also more soluble in acetone or carbon disulphide. No appreciable proportion of free sulphur or volatile organic compounds appears to arise from ebonite below 250°. The bearing of the results on the electrical deterioration of ebonite is discussed. D. F. TWISS.

PATENTS.

Production of tubes from aqueous dispersions of rubber. DUNLOP RUBBER Co., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 323,519, 6.7.28 and 30.11.28).—A predetermined quantity of concentrated compounded latex calculated to give the desired thickness of wall is introduced into a horizontally mounted cylindrical mould, *e.g.*, a rotatable tube, of the necessary internal diameter. The aqueous mixture is compacted under the influence of centrifugal force while being subjected to the action of setting and/or coagulation. During the process the tube may be heated and a current of hot air passed over the surface of the dispersion, which also may contain chemical agents capable of facilitating its coagulation by heat. D. F. TWISS.

Manufacture of vulcanised rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,580, 18.10.28).—

Before vulcanisation there are added to crude rubber, or compounded rubber (which may contain natural and/or artificial rubber), resinous or asphaltic oxidation products of organic compounds of high mol. wt. or resinous or asphaltic conversion derivatives of these oxidation products. The mechanical properties, after vulcanisation, are thereby enhanced. D. F. TWISS.

Antioxidant or age-resister [for rubber]. GOOD-YEAR TIRE & RUBBER Co., Assecs. of A. M. CLIFFORD (B.P. 302,144, 27.8.28. U.S., 10.12.27).— α - or β -Naphthylamides of formic and other acids are applied as antioxidants in rubber mixes. Examples are form- α - and - β -naphthylamides, β -naphthylacet- β -naphthylamide, β -2-naphthylaminocroton- β -naphthylamide, form- β -2-naphthylaminooethylamide, and the product from ethyl acetoacetate and α -naphthylamine.

C. HOLLINS.

Refining of old rubber of all kinds and its application. H. E. POTTS. From H. PLAUSON (B.P. 323,526, 6.9.28).—Old rubber is treated with a volatile solvent, preferably a saturated organic compound, under pressure and with agitation at 160–180°; small amounts of an organic anhydride or superoxide or an alkaline-earth peroxide or superoxide being also added; benzoyl peroxide, acetic anhydride, and phthalic anhydride are mentioned. Fibrous or powdery fillers may be removed from the resulting solution by filtration or other means. The solution obtained may be used as such, or as an impregnating material for paper, leather, etc., or as a lacquer. D. F. TWISS.

Manufacture of [highly porous] rubber substance. H. BECKMANN (U.S.P. 1,745,657, 4.2.30. Appl., 1.7.25. Ger., 22.8.24).—See B.P. 238,870; B., 1927, 81.

Manufacture of [thermoplastic] rubber composition. H. GRAY, Assr. to B. F. GOODRICH Co. (U.S.P. 1,745,926, 4.2.30. Appl., 2.11.25).—See B.P. 260,637; B., 1927, 610.

Artificial masses (B.P. 321,258).—See XIII.

XV.—LEATHER; GLUE.

Action of alkali solutions on leather. M. BORODULIN (Trans. State Inst. Appl. Chem., Moscow, 1928, No. 10, 72–74).—Treatment of leather with alkali causes appreciable loss in weight, affecting first the non-nitrogenous portion; chrome-tanned leather is resistant, but "plant-tanned" leather was especially affected.

CHEMICAL ABSTRACTS.

Determination of water in glue. O. GERNGROSS (Kunstdünger- u. Leim-Ind., 1929, 26, 195–196; Chem. Zentr., 1929, ii, 1758).—Portions (10–50 g.) of glue are dissolved in 2.5 pts. of water, cooled to 30–35°, and a portion of the solution is dried in an aluminium vessel to constant weight. A. A. ELDRIDGE.

See also A., Feb., 258, **Influence of alcohol treatment on extraction of tannin from vegetables** (MICHEL-DURAND).

PATENTS.

Treatment of bones. GÉLATINES HASSELT & VILVORDE (F.P. 634,361, 13.5.27. Belg., 12.8.26).—The bones are treated with dilute phosphoric acid to remove calcium phosphate, which is subsequently recovered as

the mono- or di-hydrogen phosphate by addition of lime. The residual ossein or gelatin from the extraction process is colourless and free from arsenic. The phosphoric acid is regenerated from part of the extract by addition of sulphuric acid sufficient to remove the lime.

A. R. POWELL.

Plastic material. L. LEDERMANN, R. KOCH, and G. KÜPKER (F.P. 629,886, 26.2.27).—A mixture of animal and vegetable glue with glue obtained by the saponification of resin, together, if desired, with emulsions of celluloid, resin, etc. in a non-aqueous solvent, e.g., amyl acetate, is poured on to plates, dried, and hardened by treatment with solutions of formaldehyde, hexamethylenetetramine, or aluminium or chromium salts. L. A. COLES.

Treatment of leather-impregnating material. E. D. VAN TASSEL, JUN., Assr. to VAN TASSEL SOLE & LEATHER CORP. (U.S.P. 1,745,591, 4.2.30. Appl., 12.6.26).—See B.P. 293,062; B., 1928, 682.

Production of solid glue and gelatin in drop- or lens-shape. C. GREINER (U.S.P. 1,744,884, 28.1.30. Appl., 17.1.28. Ger., 28.3.27).—See B.P. 287,866; B., 1929, 30.

Method and apparatus [knife] for separating skin from flesh or depilating skins. M. MASSIN (B.P. 301,032, 21.11.28. Fr., 23.11.27).

Dehydration (B.P. 323,740).—See I. **Synthetic tanning agents** (B.P. 321,190).—See III. **Artificial leather** (U.S.P. 1,737,364).—See XIII.

XVI.—AGRICULTURE.

Physical chemistry of cultivated soils. R. A. HERZNER (Z. Pflanz. Düng., 1930, 15A, 342–361).—The nature of the electrical double layer surrounding soil particles is discussed. The adsorption potential of a number of soils, examined by means of the special apparatus described, varied between 6.0×10^{-3} and 0.4×10^{-3} volt. The charge carried by soil particles is dependent on the physical structure of the soil and on the composition and concentration of the aqueous solution in equilibrium with it. Measurements of the adsorption potential of soils afford indirect means of determining their nutrient requirements. Particles of medium and heavy soils of low nutrient content carry but small charges, those of light soils vary considerably.

A. G. POLLARD.

Soils. VI. Displaceable acid radicals. VII. Evolution of clay. J. CLARENS and S. NIKOLITCH (Bull. Soc. chim., 1930, [iv], 47, 104–109, 109–113; cf. B., 1929, 950).—VI. A graphical method is described for representing the displaceable acids and bases in soils.

VII. A possible mechanism for the transformation of felspar into clay is discussed. C. W. GIBBY.

Absorbing complex of soil. K. N. TARANOV (Ukrain. Chem. J., 1929, 4, [Tech.], 179–204).—The absorptive capacity of soil (black soil-clay) may be quickly and accurately determined by means of 0.2N-hydrochloric acid. Treatment with neutral salts of univalent ions (sodium, ammonium, and potassium chlorides and sodium acetate), although serving to remove particles of about 1 micron or less in diam.,

does not deprive the soil of absorptive power. Further separation of such particles in an alkaline medium (0.1N-sodium hydroxide) gives a further quantity of organic and mineral substances, but the residual soil still retains absorptive capacity. The impossibility of displacing completely the absorbing hydrogen ions by univalent ions, even in an alkaline medium, is also demonstrated by treating soil which has been subjected to electrodialysis.

T. H. POPE.

Base exchange and acidity [in soils]. P. VAGELER and J. WOLTERS DORF (Z. Pflanz. Düng., 1930, 15A, 329—342).—The mathematical basis of a number of methods for examining base exchange in soils is critically discussed and formulæ are developed for expressing the course of this phenomenon. The importance is emphasised of ascertaining well-defined limiting values for the exchange of bases, and of including these in any mathematical expression of results. Properties of free ions, notably their hydration, are not necessarily the same as those of ions adsorbed by soils. A. G. POLLARD.

Determination of soil acidity. D. L. ASKINASI and J. S. JARUSOV (Trans. Sci. Inst. Fertilisers, Moscow, 1927, No. 48, 5—36).—The value of the absorption capacity and acidity of soils depends on the reaction of the reagent used for extraction; the highest capacity is obtained by using free alkali. The "increased" capacity for base exchange is determined as follows. The soil is placed in a funnel free from carbon dioxide and air free from carbon dioxide is admitted; the soil is then treated with 0.1N-barium hydroxide and the excess removed by washing with boiling barium chloride solution, the barium chloride being washed out with water. Hydrogen carbonate in the wash-waters is determined by titration. The sum of the barium remaining in the soil and that calculated from the hydrogen carbonate titration, when expressed in terms of calcium, gives the value of the increased capacity. The different results for the capacity obtained by the use of calcium acetate, barium chloride, etc. represent the value of the active fraction of the absorbing complex of soils. For the determination of the hydrolytic acidity the soil was treated with N-sodium chloride in presence of an excess of calcium carbonate, the total calcium in the filtrate being determined. Treatment of a soil with weak acids decreases its absorptive capacity, which may be restored by treatment with sodium acetate. Liming increases the absorption capacity of, and therefore improves, podsol soils. CHEMICAL ABSTRACTS.

Simple electrode apparatus for measuring the hydrogen-ion concentration of soils. The quinhydrone-calomel electrode. W. SELKE (Z. Pflanz. Düng., 1930, 15A, 362—368).—The apparatus described enables the user to combine speed of equilibrium with accuracy. Diffusion potential is minimised.

A. G. POLLARD.

Pipette method of mechanical analysis of soils based on improved dispersion procedure. L. B. OLMSTEAD, L. T. ALEXANDER, and H. E. MIDDLETON (U.S. Dept. Agric., Tech. Bull. 170, 1930, 23 pp.).—The soil sample is prepared by treatment with hydrogen peroxide, but the usual hydrochloric acid treatment is omitted as being superfluous and in some cases undesir-

able. Special treatment is prescribed for soils rich in manganese dioxide, involving destruction of the latter with acetic acid. Soluble salts are removed by washing and filtration with Pasteur-Chamberland filters. Dilute sodium oxalate solution is used as dispersing medium. Sand fractions are removed by a 300-mesh sieve and the fine silt, clay, and colloid fractions determined by the pipette method.

A. G. POLLARD.

Field test for available phosphorus in soils. R. H. BRAY (Illinois Agric. Exp. Sta. Bull., 1929, No. 337, 591—602).—The reagent is prepared by dissolving ammonium molybdate (100 g.) in water (850 c.c.), filtering, cooling, and adding slowly, with stirring, a cold mixture of 36% hydrochloric acid (1700 c.c.) and water (700 c.c.); for use, 120 c.c. are diluted to 1 litre. The soil (1 pt.) is shaken with the solution (3 pts.) and, after settling, the solution is gently stirred with a tin or solder rod, the intensity of the blue or green colour produced varying with the quantity of available phosphoric acid present. CHEMICAL ABSTRACTS.

Biological oxidation of sulphur. II. Effect on the microflora of activated sludge. C. V. R. AYYAR and R. V. NORRIS. **III. Sulphur-oxidising organism from activated sludge.** C. V. R. AYYAR (J. Indian Inst. Sci., 1929, 12A, 275—277, 278—294; cf. B., 1928, 938).—II. As a result of the continued aeration of suspensions of sulphur in activated sludge the p_H value decreased from 7.2 to 2.1 in one month. The faecal, putrefactive, and chromogenic types of bacteria quickly disappeared, moulds persisted longer, but in the last stage (p_H 2.1) sulphur-oxidising organisms were almost the only survivors, and were therefore easy to isolate in pure culture.

III. The sulphur-oxidising organism isolated as described above proved on examination to be a new species having a higher S:C ratio than Joffe's and being capable of oxidising elemental sulphur to sulphuric acid without accumulating sulphur either within or without the cell. It can withstand p_H 6.6 and utilises carbohydrates to some extent, oxidising more sulphur in their presence. Certain inorganic salts, notably sodium silicate, have a stimulating action.

C. JEPSON.

Composting barnyard manure with sulphur and rock phosphate. W. G. FRIEDEMANN (Georgia Agric. Exp. Sta. Bull., 1929, No. 154).—Greater loss of nitrogen occurred in untreated compost than in compost containing sulphur. CHEMICAL ABSTRACTS.

Fertilising value of sewage sludge. G. S. FRAPS (Proc. XI Texas Water Works' School, 1929, 150—152).—In Imhoff tank sludge extensive decomposition has taken place, only resistant material remaining; sludge from the activated-sludge processes is, however, easily decomposed by soil bacteria. Comparative values (8—10% H_2O) are: N 0.88—2.99, 3.6—5.5; P_2O_5 0.47—2.48, 2.41—2.68; K_2O 0.2—1.44, —, respectively. CHEMICAL ABSTRACTS.

Movement of fertiliser salts in the soil. O. F. JENSEN (J. Amer. Soc. Agron., 1929, 21, 1113).—Downward movement of phosphorus was indicated.

CHEMICAL ABSTRACTS.

Change in the water-soluble phosphoric acid content of superphosphate during heap storage. I. T. SHOJI and E. SUZUKI (J. Soc. Chem. Ind., Japan, 1929, 32, 272—274 B).—Increase in the water-soluble phosphoric acid content of stored superphosphates prepared from Christmas phosphate is hindered when the superphosphate is kept at a fairly high temperature (60°). For this reason such superphosphate which is stored in heaps does not attain a sufficiently high soluble phosphate content in summer.

S. K. TWEEDY.

Effects of seed treatment on the germination and subsequent growth of wheat. D. C. SMITH and E. N. BRESSMAN (J. Agric. Res., 1930, 40, 25—36).—Ammonium thiocyanate proved decidedly toxic as a seed dressing; it reduced germination, growth of wheat sprouts, and the length of the roots of Kanred and White Winter wheat. Ethyl bromide was slightly toxic to root and sprout growth of White Winter wheat, and reduced germination particularly of this variety. Germinating wheat grains withstood 12 days at -12°, but this treatment did not break the dormant vegetative period of winter-wheat plants subsequently produced. Alternate freezing and thawing was more detrimental than continuous freezing.

E. HOLMES.

Crop experiments with "Kalkammonsalpeter." E. BLANCK and F. KLÄNDER (J. Landw., 1930, 77, 337—340).—In pot trials with oats, "Kalkammonsalpeter" (a mixture of ammonium nitrate and calcium carbonate) proved slightly inferior to sodium nitrate but better than ammonium sulphate or calcium cyanamide.

A. G. POLLARD.

Phosphate nutrition of cultivated plants. M. K. DOMONTOVICH (Trans. Sci. Inst. Fertilisers, Moscow, 1928, No. 52, 3—37).—Within the limits of physiologically balanced solution the p_H has no influence on the absorption of phosphorus by oats or buckwheat. Oats, maize, buckwheat, mustard, and peas showed no striking differences in the power to exhaust the phosphorus supply from a solution. The influence of the period of vegetation on the utilisation of soluble and insoluble phosphates by plants was also investigated. With decrease in the size of particles raw phosphates become available in culture solutions.

CHEMICAL ABSTRACTS.

Influence on plant growth of the addition to sand of clays of varying composition. E. BLANCK and E. F. VON OLDERSHAUSEN (J. Landw., 1930, 77, 331—336).—Pot experiments are described in which oats were grown in mixtures of sand and a number of clays. Higher yields were obtained with mixtures having p_H 4.5 approx. than with the more nearly neutral samples.

A. G. POLLARD.

Apparatus for watering pot cultures and determining the absolute water consumption. K. MEYER (J. Landw., 1930, 77, 327—330).—A portable apparatus is described whereby water is added to culture vessels till the latter attain a predetermined gross weight. The volume of water added is measured by a special apparatus.

A. G. POLLARD.

Effects of leaching with cold water on the foliage of fruit trees. I. Course of leaching of dry

matter, ash, and potash from leaves of apple, pear, plum, black currant, and gooseberry. T. WALLACE (J. Pomology, 1930, 8, 44—60).—The leaves, subjected to leaching for 4 successive days at ordinary temperatures, lost considerable proportions of potash, ash, and dry matter, the proportions leached being in the order named. The ratios of % potash/% ash, and % ash/% dry matter leached varied for different classes and varieties of fruit. Apple and gooseberry leaves leached readily, plum leaves showed great variations. Summation curves suggest that the same groups of materials were leached throughout the 4-day periods.

E. HOLMES.

Manuring of fruit trees. III. Effects of deficiencies of potassium, calcium, and magnesium, respectively, on the contents of these elements, and of phosphorus in the shoot and trunk regions of apple trees. T. WALLACE (J. Pomology, 1930, 8, 23—43; cf. B., 1927, 55).—From an examination of the effects on apple trees, Stirling Castle variety, grown in quartz sand with added nutrients, it is concluded that omission of potassium resulted in increased shoot growth, leaf scorch, and defoliation of shoots, of calcium in slightly increased shoot growth, relatively large leaves, and breakdown of leaf-tissue in patches, whilst omission of magnesium reduced shoot growth particularly after two seasons. Large reductions of potash and magnesia, but not of lime, in the ash of the trees was caused by lack of these bases. Omission of potash and lime gave a higher percentage of dry matter and a low percentage of ash, whilst omission of magnesia had the reverse effect. The effects on phosphoric acid in the plant were not striking.

E. HOLMES.

Decomposition and effect of straw- and peat litter-stall manure in soils. P. NIEMEYER (Landw. Versuchs-stat., 1929, 108, 305—348).—Comparison is made of the chemical changes occurring during the decomposition of manure made with straw and peat litter in soils of different types. Changes in the "total carbonaceous matter" are more rapid in straw manure than in peat, the difference being most marked in clay soils. Decomposition of pentosans follows similar lines, and in the case of peat is incomplete after 1 year. The decomposition of lignins is slower, and occurs at similar rates with both manures. With the progressive decomposition of cellulose and pentosans the soil humus becomes increasingly rich in lignins. Decreases in total nitrogen soon after the application of manure were greater from the straw than from the peat. After cropping, all added nitrogen from both materials was utilised. Changes in the easily-soluble nitrogen were similar, the proportion in peat manure being higher than in straw initially, but lower after cropping. The nitrate content of soils receiving peat manure was generally higher than with straw. No differences were observed in the citric-soluble phosphate in the two cases. Straw originally contained more easily-soluble potash than peat, but losses by leaching and chemical change in the treated soils were greater in the case of straw. Crop yields were generally higher from peat manure plots. This is attributed to the higher proportion of nitrogen available and to the more uniform

rate of decomposition of the peat manure during the growth period of the plant. Both field trials and Neubauer tests show that the proportional utilisation of nutrients is greater from peat manure than from straw.

A. G. POLLARD.

Compatibilities of insecticides. I. Fluosilicates and cryolite with arsenates. R. H. CARTER (J. Econ. Entomol., 1929, 22, 814—818).—Mixtures of lead arsenate with sodium, potassium, barium, or calcium fluosilicate in water do not show excessive amounts of soluble arsenic; calcium, manganese, magnesium, aluminium, and barium arsenates are largely decomposed in presence of fluosilicate. Cryolite inhibits the formation of soluble arsenic.

CHEMICAL ABSTRACTS.

Use of acidulated mercuric chloride in disinfecting potato tubers for the control of *Rhizoctonia*. J. G. LEACH, H. W. JOHNSON, and H. E. PARSONS (Phytopathol., 1929, 19, 713—724).—Treatment with mercuric chloride solution (0.2%) containing 1 vol.-% of hydrochloric acid is rapid (5 min.), and as satisfactory as that with soap in mercuric chloride solution (2 hrs.) or hot formaldehyde.

CHEMICAL ABSTRACTS.

Insecticidal properties of soaps against the Japanese beetle. P. A. VAN DER MEULEN and E. R. VAN LEEUWEN (J. Econ. Entomol., 1929, 22, 812—814).—The insecticidal efficiencies for *Popillia japonica* of the sodium and potassium soaps of the fatty acids of various oils are recorded.

CHEMICAL ABSTRACTS.

Sulphide-sulphur content as a basis for diluting lime-sulphur for spraying. G. M. LIST (Colorado Agric. Exp. Sta. Bull., 1929, No. 352, 14 pp.).—The insecticidal value of lime-sulphur is attributed largely to the ability of the calcium polysulphides to take up large quantities of oxygen. Determinations of the effective concentration of sulphide-sulphur for various pests are recorded.

CHEMICAL ABSTRACTS.

Arsenical injuries [to foliage] and correctives. J. M. GINSBURG (New Jersey Agric. Exp. Sta. Bull., 1929, No. 468, 1—16).—Zinc oxide, when added to arsenical sprays, eliminated soluble arsenic, but was itself toxic to peach and apple foliage; alumina, aluminium palmitate, calcium silicate, stannic oxide, charcoal, and powdered skim-milk did not reduce the injury. Ferric oxide or excess of lime slightly injured peach foliage; the former improves adherence.

CHEMICAL ABSTRACTS.

Injury of peach trees with *p*-dichlorobenzene. O. I. SNAPP and H. S. SWINGLE (J. Econ. Entomol., 1929, 22, 782—785).—Injury was caused in high temperatures. *p*-Dichlorobenzene dissolved in gasoline did not control the peach borer.

CHEMICAL ABSTRACTS.

Simultaneous determination of arsenic and copper, especially in insecticides. F. WESSEL (Chem.-Ztg., 1930, 54, 97—98).—The substance is dissolved in dilute hydrochloric acid and an aliquot part of the solution containing 0.03—0.04 g. of arsenic as arsenic acid is filtered off, treated with 1 c.c. of 10% calcium chloride solution and 8—10 c.c. of 10% ammonia in excess of that required for neutralisation, and thoroughly stirred until the precipitate becomes crystalline. After 10—12 hrs. the solution is filtered

the precipitate washed with a dilute ammoniacal solution of calcium chloride until colourless, and then dissolved in 5 c.c. of 10% hydrochloric acid, and the arsenic determined iodometrically. The ammoniacal filtrate is boiled to expel ammonia, concentrated if necessary, acidified with dilute sulphuric acid, and the copper determined iodometrically. If the arsenic in the original substance is all present as arsenious oxide the copper may be determined directly in the usual manner and the arsenic by titration with iodine in the presence of sodium hydrogencarbonate. If the copper is present as cuprous salt and the arsenic as trioxide, the substance is heated with sulphuric acid and hydrogen peroxide and the analysis finished by the first of the above methods.

A. R. POWELL.

See also A., Feb., 185, Immersion filter for phosphorus, calcium, and crude-fibre determinations (SIMON). 258, Rôle of enzyme action in seed germination (STEPHEN). 262, Nitrates and ammonium salts as sources of nitrogen for plants at constant p_H (PIRSCHLE). Relative importance of sulphur and phosphorus in plant nutrition (BERTRAND and SILBERSTEIN). Action of calcium and magnesium ions on seeded peas (BOLELOUCKY). Assimilation of phosphoric acid by barley (TUEVA).

PATENTS.

Fertiliser. CHEM. FABR. DR. HEPPES & Co., G.M.B.H., J. B. CARPZOW, and J. HEPPES (Swiss P. 124,143, 31.10.25. Ger., 31.10.24).—The use is claimed of mixtures of inorganic or organic vegetable nutrients with slime deposited from streams etc. and containing dead animal or vegetable material.

L. A. COLES.

Fertiliser. ELEKTIZITÄTWERK LONZA (GAMPEL U. BASEL), and E. LÜSCHER (Swiss P. 124,358, 18.12.26).—Unwashed filter residues obtained in the production of ammonium sulphate by treating gypsum with ammonia and carbon dioxide are treated with an equivalent weight of nitric acid of such concentration that a practically dry mixture of calcium nitrate, ammonium nitrate, and a little calcium sulphate is obtained.

L. A. COLES.

[Phosphatic] fertiliser. G. TRUFFAUT (F.P. 635,523, 3.6.27).—Crude phosphate rock is digested with aqueous phosphoric acid and finely-ground silica, the acid used being sufficient to decompose the carbonates and fluorides present and to convert 80% of the calcium phosphate into CaHPO_4 and 20% into $\text{Ca}(\text{H}_2\text{PO}_4)_2$. Limestone is then added until all the fluorine is precipitated and the solution filtered and evaporated for the recovery of the dissolved phosphates.

A. R. POWELL.

Fertiliser containing potassium compounds. B. T. G. V. H. O. MELLERBORG (Swed. P. 60,306, 5.7.24).—Mixtures of potassium ores with quick-lime and sufficient water to slake the lime are sealed up in autoclaves; the temperature and pressure generated render the potash water-soluble.

L. A. COLES.

Dry seed pickling material. CHEM. FABR. L. MEYER (G.P. 454,570, 27.11.23).—Pickling materials containing a fungicide and an acid or material capable of hydrolysis to furnish an acid, such as various mixtures of copper carbonate and anhydrous copper sulphate,

calcium arsenite, mercurous sulphate, copper acetate-arsenite, or arsenious acid, are claimed. E. HOLMES.

Method of pickling seed. BAYERISCHE STICKSTOFF-WERKE A.-G. (G.P. 457,995, 2.9.24).—A 1:1 mixture of seeds treated with different pickling materials, and immediately dried, is claimed to give increased germination and plants less susceptible to deleterious external conditions. E. HOLMES.

Increasing the germination of seed. I. G. FARBENIND. A.-G. (G.P. 456,556, 16.12.19. Cf. U.S.P. 1,565,237; B., 1926, 559).—It is claimed that seeds of barley and wheat may be disinfected, and germination increased, by treatment for $\frac{1}{2}$ hr. with solutions containing 0.25–0.5% of various specified aryl derivatives of arsenious acid, or with similar materials combined with copper sulphate, mercuric chloride, and mercury chlorophenols. E. HOLMES.

Insecticides etc. I. G. FARBENIND. A.-G., Assecs. of A. STEINDORFF and O. WEICKERT (G.P. 453,357, 19.3.22).—Insecticides containing copper compounds, sulphonic acids of high mol. wt. (e.g., sulphonated naphthol pitch, coal-tar pitch), waxes such as coumarone wax, colophony, and fillers such as lime or slaked lime are claimed. E. HOLMES.

Preparation of stable water-soluble insecticides etc. I. G. FARBENIND. A.-G., Assecs. of K. MARX and K. BRODERSEN (G.P. 456,019, 22.12.22. Addn. to G.P. 430,712; B., 1926, 893).—Water-soluble insecticides stable in air are prepared by incorporating with organic acids of high mol. wt., obtained by the oxidation of brown coal etc., alkali polysulphides with aluminium hydroxide or alumina, and arsenicals. E. HOLMES.

Materials with fungicidal and insecticidal properties. J. D. RIEDEL A.-G. (G.P. 453,193, 11.10.24).—Soap emulsions of the acetyl, benzoyl, and crotonaldehyde derivatives of hydrogenated naphthalene are stated to be better insecticides etc. than is hydrogenated naphthalene itself. E. HOLMES.

Material for combating plant pests. CHEM. FABR. DR. REIS G.M.B.H. (G.P. 454,899, 19.8.21).—A solution of *p*-dichlorobenzene in pyridine, emulsified with soft soap, is claimed to be effective against plant lice, red spiders, and caterpillars without damaging the plant or interfering with the flavour of vegetables or fruits. E. HOLMES.

Extermination of soil and other plant pests. E. DINKELMANN (Swiss P. 124,486, 16.2.27).—Soil disinfectants containing nicotine, ferrous sulphate, ammonium sulphate, or sodium nitrate, with quicklime or calcium salts are claimed to be useful when applied with liquid manure or water. E. HOLMES.

Sulphur preparations as plant disinfectants. I. G. FARBENIND. A.-G., Assecs. of M. MÜLLER-CUNRADI and M. SCHMIKING (G.P. 454,933, 21.12.23).—Finely-powdered sulphur or ammonium polysulphide solutions with humic acid or its salts are claimed as good spray materials. In an example, brown coal rich in humus is treated with ammonia, the mixture filtered, and the filtrate evaporated to dryness. The finely-powdered residue is mixed with 3 pts. of sulphur. E. HOLMES.

Preservation of [fresh] flowers and plants. LABORATORIUM MIGOHLA INT. K. KIEF (G.P. 455,528, 1.3.24).—The flowers etc. are treated with solutions containing 50% of alcohol, 45% of water, and 5% of boric or salicylic acid. E. HOLMES.

Production of liquid insecticide containing the effective ingredient of *Derris* species. T. OHORI and K. NAKAZAWA, Assts. to ZAIDAN HOJIN RIKAGAKU KEN-KYUJO (U.S.P. 1,745,907, 4.2.30. Appl., 10.2.28. Jap., 18.2.27).—See B.P. 285,797; B., 1929, 106.

Mixing mill (U.S.P. 1,739,149).—See I. Seed-grain immunising (B.P. 321,396).—See IX.

XVII.—SUGARS; STARCHES; GUMS.

How much non-sugar from the water used for diffusion passes into the juice [in beet-sugar factories]? V. STANĚK and J. VONDRÁK (Z. Zuckerind. Czechoslov., 1929, 54, 117–121).—The water supplies of some beet-sugar factories are liable, at times, to contain abnormal amounts of dissolved solids. To ascertain what proportion of the dissolved solids in the water passes into the juice the authors carried out comparative diffusion experiments with a laboratory battery, using distilled water alone and distilled water containing 0.1 and 0.5% of sodium chloride and urea. From the amounts of these substances found in the diffusion juices it is concluded that about one third of the dissolved matter in the water passes into the juice. If this is not removed by carbonatation it must lower the purity of the final juice and increase the amount of molasses obtained. The extra molasses due to 0.1% of mineral salts or 0.1% of organic non-sugar in the original water may amount to 0.25% of the weight of beets in the former case or 0.12% in the latter.

J. H. LANE.

Baryta-alumina clarification of cane-sugar products. J. H. HALDANE and N. DEERR (Facts about Sugar, 1929, 24, 928).—The following results were obtained for mixed juice, syrup, and waste molasses, respectively: direct polarisation with dry lead acetate (Horne) 13.31, 46.46, 34.18; double polarisation (Steuerwald) 13.49, 47.11, 35.93; direct polarisation with clarification by baryta and alumina, volume corrected, 13.24, 46.19, 32.83; double polarisation by inversion with sulphuric acid and clarification with baryta and aluminium sulphate (Coates and Shen) 13.40, 46.84, 35.18. CHEMICAL ABSTRACTS.

Clarification of sugar solutions for analysis. M. I. NAKHMANOVICH and S. L. BERMAN (Monograph, 184 pp.).—A review and bibliography.

CHEMICAL ABSTRACTS.

Capacity of crystallisation of green syrups treated with activated carbon, sulphur dioxide, and carbon dioxide. I. I. DOKHILENKO and G. A. SOKOLOV (Zhur. Sakh. Prom., 1929, 3, 20–34).—The velocity of crystallisation of green syrups treated with lime and carbon dioxide or sulphur dioxide (6%, calc. on sugar) is much greater than when norit (2%, calc. on sugar) is used. Norit effects greater removal of ash, but the decrease in surface tension is greater when norit is used. CHEMICAL ABSTRACTS.

Determination of the starch content in potatoes. SPROCKHOFF (Z. Spiritusind., 1930, 53, 35).—Unless the determination of starch in potatoes by the chemical method is based on a sample weighing at least 5 kg., *i.e.*, the weight used for the sp.-gr. method, comparable results from the two methods are not obtained, since individual potatoes from the same lot may show variations in the starch content amounting to 3.7%. In addition, the starch is not evenly distributed through the potato, and the starch content at the hilum end may exceed that at the top end by 1% or more. If possible, a correction should be made for the gas content of the potatoes when the starch is determined by the sp.-gr. method.

C. RANKEN.

Glycerin by fermentation. TOMODA.—See XVIII. **Swelling of dried beet slices.** MACH and HERRMANN.—See XIX.

See also A., Feb., 195, **Precipitation of sugars etc. as copper-barium hydroxide complexes** (FLEURY and AMBERT). 259, **Galactose from sea-weed "Tengusa"** (UYEDA).

PATENT.

Production of dextrose and laevulose from grape juice. SOC. DES ETABL. BARBET (Addn. 32,642, 17.4.26, to F.P. 615,942; B., 1927, 856).—The crystals of dextrose obtained by evaporation are separated from the syrup by centrifuging and are then washed with a concentrated dextrose solution. The mother-liquor is evaporated to obtain a second crop of impure dextrose crystals and a mother-liquor (d 1.4) which is decolorised with animal charcoal and allowed to crystallise to obtain pure white laevulose.

A. R. POWELL.

XVIII.—FERMENTATION INDUSTRIES.

Substances toxic to the Downy mildew of the hop. W. NEWTON and C. YARWOOD (J. Inst. Brew., 1930, 36, 67—68).—Sap bled from the cut tips of hop plants was toxic to the zoöspores of the Downy mildew, whereas that bled from cut basal stems was almost non-toxic. Sap which was obtained by pressure from ground hop and other plant material, such as nettles and bind-weed, was only slightly toxic. The antiseptic principle associated with the hop resins was probably responsible for the toxicity of the sap. Commercial pine resin dissolved in methyl alcohol and potassium hydroxide was more toxic than copper sulphate, and may be used as a control measure against the attacks of the Downy mildew.

C. RANKEN.

Prolonged fermentation of wort. STAIGER and GLAUBITZ (Z. Spiritusind., 1930, 53, 36).—A wort prepared from rye was seeded with yeast, and the content of alcohol, acid, and diastase as well as the amount of carbon dioxide evolved and the condition of the yeast were determined after periods of 24, 48, 72, 96, and 120 hrs. At the end of 72 hrs. the optimal production of alcohol was obtained, the amount of acid was normal, carbon dioxide had ceased to be evolved, and the quantity of diastase, although diminished, was sufficient. The appearance of the yeast was considered as normal, although there were 50% of cells present which were weakened or dead owing to the increasing alcohol content

and diminished quantity of nutrients. At the end of 96 hrs. the dead cells had increased to 60% and the diastase had completely disappeared, whilst at 120 hrs. almost all the cells had been destroyed, the acidity was higher, and the alcohol content had diminished slightly.

C. RANKEN.

Influence of aeration during fermentation on yeast condition and beer quality. F. WINDISCH (Woch. Brau., 1930, 47, 33—39, 45—51).—Parallel fermentations of 8-litre portions of wort, of which one set was aerated daily by stirring, showed distinctly higher rates of fermentation in the aerated sets. The yeast harvested from these deteriorated in quality throughout the fermentation, as shown by its appearance and its fermentative and reproductive powers. The observations were made on two bottom-fermentation yeasts. The increased rate of fermentation was due to the stimulating effect of aeration on reproduction, since it did not occur when high pitching rates were used, either with fresh yeast or with sediments to which fresh wort was added. Aeration after cell increase had reached its maximum, *i.e.*, on the 5th and 6th days of fermentation, also had no effect on the final attenuation. A series of three successive brewery fermentations, of which one set was aerated by pumping on the first three days, showed a slight increase of fermentation rate on the first two occasions, but again the yeast deteriorated progressively and in the third fermentation proved less vigorous than the unaerated yeast, in spite of the stimulating action on reproduction of the wort aeration. The beers from the third unaerated fermentation gave a clean normal sediment, that from the corresponding aerated wort being larger, looser, and of unsound smell, and containing hardly any normal yeast cells. On the basis of the results the practice of aerating to stimulate fermentation is criticised.

F. E. DAY.

Determination of the fat content of yeast. R. OTTO and A. HALTER (Chem.-Ztg., 1930, 54, 98—99).—The sample is dried at 105° and, after fine grinding, 5 g. are dissolved in 10 c.c. of hydrochloric acid (d 1.19) and 10 c.c. of water with gentle warming. The brown syrupy solution is boiled gently for 5 min., filter pulp is added, and the solution diluted with 25—30 times its volume of cold water and filtered. The filter is washed free from acid with cold water, dried, and the fat extracted in the Soxhlet apparatus in the usual way. Direct extraction of the yeast with ether gives low results.

A. R. POWELL.

Production of glycerin by fermentation. VII. **Velocity of fermentation in presence of sulphite.**

VIII. **Formation of β -butylene glycol and acetic acid during glycerin fermentation in presence of sulphite.** Y. TOMODA (J. Soc. Chem. Ind., Japan, 1929, 32, 229—230 B, 230—231 B).—VII. The velocity of fermentation in a synthetic medium is seriously affected by the sulphite concentration, but in molasses solution the effect is less marked. On the assumption that 3 mols. of sulphite combine with 1 mol. of zymase, producing 1 mol. of inactive zymase-sulphite complex, and that the velocity of fermentation depends solely on the uncombined active zymase, the relation between the velocity of fermentation and the concentration of the sulphite is expressed by $V_a = Bn/(1 + Ds^3)$ for the synthetic

medium, and by $V_m = B^1 \log(1+n) - \log(1+Ds^3)$ for molasses solution, where V_a or V_m represents the velocity of fermentation, n the initial number of yeast cells per unit of volume, s the initial concentration of sulphite, and B , B^1 , and D are constants. These equations are in agreement with the experimental results. The dissociation constant of the arbitrary zymase-sulphite complex, calculated from the relative velocities of fermentation, is 7.6×10^{-3} , which is similar to the value 4.6×10^{-3} obtained by Kerp (A., 1904, i, 713) for the acetone-bisulphite compound. In most cases the maximum concentration of sodium bisulphite tolerated by the fermentation is about 0.2%, and if the inhibiting action of sulphite is ascribed to the effect of bisulphite ions, the maximum allowable concentrations of sulphite calculated for different values of p_H of the medium are in good agreement with the experimental results (cf. A., 1928, 365, 923).

VIII. During the glycerin fermentation of sugar in presence of a relatively small proportion of sulphite and a large proportion of sugar, the formation of β -butylene glycol occurs after the exhaustion of the free sulphite in the medium, probably owing to dissociation of the acetaldehyde-bisulphite compound and subsequent condensation and reduction of the resulting acetaldehyde. A considerable amount of acetic acid is formed at the same time. The formation of glycerin after exhaustion of the free sulphite is to be attributed mainly to the induction of Neuberg's third form of fermentation and, to a less extent, to the regeneration of the aldehyde-fixing power of the sulphite consequent on the transformation of the dissociated acetaldehyde into β -butylene glycol. Formation of acetylmethylcarbinol during the glycerol fermentation of sugar is not observed, although in normal alcohol fermentation traces of both acetylmethylcarbinol and β -butylene glycol are formed, the amount of the latter being much less than in the glycerin fermentation (cf. A., 1928, 365, 923).

T. H. POPE.

Hop-drying investigations, their aims, and results already attained. A. H. BURGESS (J. Inst. Brew., 1930, 36, 57—59).—A *résumé* of work already abstracted (cf. B., 1928, 461; 1929, 533). C. RANKEN.

Brewing research and large-scale experiments. H. L. HIND (J. Inst. Brew., 1930, 36, 51—57).

Cause of odour of beer etc. SCHMALFUSS and BARTHMEYER.—See XIX.

See also A., Feb., 250, **Fermenting power of yeasts of first group of *Saccharomyces*** (TRAUTWEIN and WASSERMANN). **Acceleration of alcoholic fermentation of sucrose** (OWEN and DENSON). **Fermentation of sucrose in presence of sulphite** (POLAK).

PATENTS.

Manufacture of sparkling wine and similar non-alcoholic sparkling drinks. A. CHAUSSEPIED (F.P. 623,984, 2.11.26).—Fermented liquors made in closed containers are subjected to distillation in a vacuum and the alcohol and carbon dioxide are condensed separately, the latter being returned to the residual liquor in the still.

A. R. POWELL.

Wine vinegar. F. NOLDIN (F.P. 625,752, 6.12.26).—The mass to be fermented is agitated with compressed air in presence of filling materials. A. R. POWELL.

Production of bacterial enzyme preparations. A. BORDIN and J. EFFRONT (U.S.P. 1,744,742, 28.1.30. Appl., 28.6.23. Fr., 3.7.22).—See B.P. 221,860; B., 1924, 959.

XIX.—FOODS.

Gas storage of fruit. II. F. KIDD and C. WEST (J. Pomology, 1930, 8, 67—77. Cf. B., 1927, 425).—Commercial storage life of fruit is defined as the interval between dates of storage and of the development of 10% wastage in store. Wastage of apples at 1° was found to be entirely due to low-temperature breakdown in green, unripe fruit, and at 5° and 10° to fungal rotting. The optimum conditions for storing Bramley's Seedling apples were determined as 5° in an atmosphere of 10—15% of carbon dioxide and 10% of oxygen, and gave 50% greater life than storage under conditions of temperature control only. Further tests indicate that the loss of carbohydrates was approx. 1.3 times as fast in air as in an atmosphere with 10% of oxygen, and approx. 1.45 times as fast in the absence of carbon dioxide as with 10% of this gas. Loss of acid does not appear to be affected markedly by reduction in oxygen or increase in carbon dioxide up to 9%.

E. HOLMES.

Storage of pears in artificial atmospheres. S. A. TROUT (J. Pomology, 1930, 8, 78—91).—At moderate temperatures the storage life of pears may be extended as much as 50% by exclusion of oxygen, provided the storage atmosphere contains less than 10% of carbon dioxide. This treatment prevents the appearance of scald in susceptible varieties, and, by excluding oxygen, stops normal respiratory oxidations which produce acetaldehyde. The mechanism of core breakdown is discussed. (Cf. preceding abstract.) E. HOLMES.

Swelling capacity of dried [beet] slices and the effects of over-heating. F. MACH and R. HERRMANN (Landw. Versuchs-stat., 1929, 108, 349—370).—Processes for determining the "swelling coefficient" are described, and the effect of overheating at various temperatures on this value is obtained. In general, the swelling capacity decreases markedly when drying is carried out at 150° and upwards. Examination of the nutrient value of overheated slices showed the soluble protein content to diminish sharply at 150—300°. There is a corresponding increase in crude fibre and a decrease in "soluble" crude fibre within this temperature range. A. G. POLLARD.

Ensilaging of green fodder rich in protein with special reference to the hydrochloric acid process. K. VOLBEHR (Landw. Versuchs-stat., 1929, 108, 115—146).—Laboratory examination is described of the chemical and microbiological changes occurring in the Fingerling hydrochloric acid process for the preservation of green fodder. To inhibit unwanted enzyme and bacterial action the initial reaction of the mixture should not exceed p_H 2. The limiting value for the production of good silage is p_H 4.0. Butyric fermentation becomes active at p_H 5.0. Protein losses increase directly with

the storage period, with the proportion of protein in the original material, and inversely with the amount of hydrochloric acid used. The total nitrogen content of the product is not greatly affected by the conditions of storage. The use of 5% sodium chloride as a preservative yields excellent silage with similar protein loss to that of the hydrochloric acid method.

A. G. POLLARD.

Sensitiveness of test animals to French beans.

A. VAN RAALE (Chem. Weekblad, 1930, 27, 37).—Tins of French beans of which complaints had been received were found sterile and free from poisons by chemical examination. Rats and mice fed on these beans alone died within a few days, and the same result was obtained with fresh beans bottled in the laboratory.

S. I. LEVY.

Diacetyl as the constituent causing the odour of provisions and other materials.

H. SCHMALFUSS and H. BARTHMEYER (Biochem. Z., 1929, 216, 330—335).—The cause of the odour of tobacco smoke, butter, burnt coffee, beer, and honey is shown to be diacetyl, by isolation as the nickel glyoxime. Diacetyl was not obtained from tea, black bread, or bonbons.

P. W. CLUTTERBUCK.

Disposal of creamery wastes. C. E. SLAUGHTER (Mich. State Coll. Eng. Exp. Sta. Bull., 1928, No. 18, 27 pp.).—High acidity inhibits reduction of wastes. The acidity increases as the oxygen consumed is reduced. Septic action is facilitated by addition of lime to p_H 7.6. Chemical precipitation with ferrous sulphate, followed by addition of lime and then by secondary storage or oxidation on a sand filter, is recommended.

CHEMICAL ABSTRACTS.

Determination of water and fat. BULL. Soya-bean oil extraction.

MASHINO.—See XII.

See also A., Feb., 160, **Formation of pectin jellies by sugar** (SPENCER). 260, **Proteins of *Cajanus indicus*** (SUNDARAM and others).

PATENTS.

Production of butter. J. SIEDEL (B.P. 323,546, 6.10.28).—Butter is prepared from rich cream (at least 45% of fat) at 2—10° by sweeping or spreading it in a thin film over a suitable surface until the structure becomes granular with the formation of butter.

E. B. HUGHES.

Production of feeding-stuff. O. MARTENS (G.P. 459,172, 2.12.25).—Dairy residues, e.g., whey-albumin, lactose molasses, enriched with lactic acid and mixed with albuminous vegetable material, e.g., soya beans, are dried, worked up to a paste with water and feeding-stuff rich in carbohydrates, and the products are fermented with yeast.

L. A. COLES.

Production of feeding-stuff from cellulose. M. A. J. L. F. GIRAULT (F.P. 623,663, 25.10.26).—The cellulose in straw, sawdust, etc. is saccharified by mixing the material with sulphuric acid and heating the mixture, e.g., with steam, and the acid is neutralised by stirring with calcium carbonate. The product is used direct or after admixture with meal, molasses, etc. for the production of fodder.

L. A. COLES.

Conditioning and drying machines for wheat and other cereals. H. SIMON, LTD., and C. RAEBURN (B.P. 324,258, 13.2.29).

[Consumable] liquor cooling apparatus. T. ALLEN (B.P. 324,730, 8.11.28).

Dehydration (B.P. 323,740).—See I.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Methyl alcohol content in tobacco. II. J. O. GABEL and L. G. SCHMUKLOVSKA (Ukrain. Chem. J., 1929, 4, [Tech.], 241—248; cf. A., 1929, 1347).—The percentage of methyl alcohol in dry tobacco varies from 0.464 to 0.905 for cigarette, from 0.228 to 0.401 for cigar, from 0.074 to 0.158 for dark Bakun, and from 0.549 to 0.703 for light Bakun-Verkievsky tobaccos. Maxorka tobaccos vary widely in this respect; two varieties from the Zhabki-Romensky region gave 0.660 and 0.661%, and two from the Xmelev-Romensky region 0.090%; for other Maxorka tobaccos the mean percentage was 0.328. The determination of the methyl alcohol forms a valuable means of following the course of, and indicating the completion of, the fermentation of tobacco, and renders it unnecessary to determine the pectic acid. The proportion of methyl alcohol should not exceed 3.2% of the proportion of pectic acid present.

T. H. POPE.

Haziness of final chloroformic extractions in alkaloidal assaying. G. E. ÉWE (J. Amer. Pharm. Assoc., 1930, 19, 23—28).—The cloudiness of the final extracts obtained in the chloroform extraction of aqueous solutions of alkaloids containing ammonia and ammonium sulphate is due to an almost colloidal suspension of a trace of the aqueous liquid. The effect of the ammonium sulphate thus introduced into the alkaloid is practically negligible under the ordinary conditions of analysis, but in the presence of a great excess of the aqueous liquid lower results may be obtained due to liberation of ammonia by the alkaloid during the evaporation. Methods for clarifying the final extracts are described.

E. H. SHARPLES.

Therapeutic activity of neoarsphenamine. A. E. JURIST and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1930, 19, 35—40).—A commercial sample of neoarsphenamine is described. Compared with arsphenamine, it is less toxic and as uniform in its activity towards trypanosomes and only slightly less active. It also had a therapeutic index at least twice that of two other commercial products. In neoarsphenamine there is no relationship between either the toxicity and therapeutic efficiency or the solubility and biological properties. The chemical composition is not closely related to the solubility of the material, and the chemical factor is not the major controlling one in determining the trypanocidal activity.

E. H. SHARPLES.

Preparation of ethereal oils. A. W. K. DE JONG (Chem. Weekblad, 1930, 27, 82—87).—An account is given of the nature, occurrence, and extraction of ethereal oils in general, with a *résumé* of the possible mechanisms of syntheses in the plant.

S. I. LEVY.

Δ^8 -Decenaldehyde, the principal constituent of essential oil of *Achasma Walang Val*. P. VAN ROMBURGH (Proc. K. Akad. Wetensch. Amsterdam, 1929, 32, 1352—1354).—The oil was taken from the leaves, petioles, and rhizomes of *Achasma Walang Val*. It consists mainly of an aldehyde (Δ^8 -decenaldehyde). By repeated fractional distillation *in vacuo* a principal fraction was obtained as a colourless liquid, b.p. 104°/13 mm., 229—231°/760 mm. This showed typical aldehydic properties, and was unsaturated. It was oxidised by alkaline potassium permanganate, yielding *n*-octoic acid. The aldehyde was oxidised by passing oxygen through it until absorption had practically ceased; an unsaturated acid, $C_{10}H_{18}O_2$, of which the structure is still being investigated, was formed. A. J. MEE.

Cause of odour of tobacco etc. SCHMALFUSS and BARTHMEYER.—See XIX.

See also A., Feb., 192, Easily decomposed organic compounds of bismuth (PICON). 216, Rotenone, from *Derris* root (TAKEI and KOIDE). 217, Optically active pericarveol (SCHMIDT). 222, New antimalarials (BALDWIN). 223, Product from pyramidone (CHARONNAT and DELABY). Synthesis of 1-phenyl-2-methyl-3:4-cyclotrimethylene-5-pyrazolone (MANNICH). 224, New antimalarials (CHATTERJEE; BARGER and ROBINSON; ROBINSON). 227, Determination of nicotine (CHATTAWAY and PARKES). Alkaloids of *Anabasis aphylla* (ORÉKHOFF). Alkaloids of *Fritillaria verticillata* (FUKUDA). Yohimba alkaloids (HAHN and SCHUCH). 228—9, *Aconitum* alkaloids (MAJIMA and MORIO; MORIO). 229, Brucine (WAGENAAR; THOMS and GONEIN). *Strychnos* alkaloids (CORTESE). 230, Compounds of tryparsamide type (GIBSON and LEVIN). 233, Nucleic acid-protein compounds (HAMMARSTEN and others). 255, Hormone of the anterior pituitary lobe (ZONDEK and ASCHHEIM).

PATENTS.

Manufacture of thymol and menthol. SCHERING-KAHLBAUM A.-G. (B.P. 301,087, 23.11.28. Ger., 24.11.27).— α -Hydroxythymol is dehydrated and hydrogenated to thymol in one operation by heating at 120—180° with hydrogen in a closed vessel in presence of a nickel catalyst and kieselguhr etc. *r*-Menthol may also be obtained.

C. HOLLINS.

Manufacture of the *m*-aminobenzoyl derivative [ester] of dimethylaminopentanol [β -dimethylaminomethyl- β -hydroxybutane; local anæsthetics]. SOC. DES USINES CHIM. RHÔNE-POULENC, ASSECS. of ÉTABL. POULENC FRÈRES, E. FOURNEAU, and J. TRÉFOUEL (B.P. 301,845, 6.12.28. Fr., 6.12.27).— β -Dimethylaminomethyl-*n*- β -butyl alcohol is esterified with *m*-nitrobenzoyl chloride in benzene at 50° and the product is reduced with stannous chloride and hydrochloric acid. The hydrochloride, m.p. 189°, has high anæsthetic power.

C. HOLLINS.

Manufacture of phenylcarboxylic acid compounds [carboxyphenylaminoquinolines]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,738, 21.8.28).—A 2(or 4)-amino-4(or -2)-alkylquinoline is heated with an aminobenzoic acid or ester to give

therapeutically valuable compounds. 2-*o*-, *m*-, and *p*-Carboxyphenylamino-4-methylquinolines, m.p. 305—307°, 255—265°, and 203°, respectively, and 4-*p*-carboxyphenylaminoquinaldine, m.p. 327—328° [ethyl ester hydrochloride, m.p. 267° (decomp.)], are described.

C. HOLLINS.

Preparation of compounds of CC-disubstituted barbituric acids and 4-dialkylamino-1-phenyl-2:3-dimethyl-5-pyrazolones [pyramidone]. CHEM. WORKS, FORMERLY SANDOZ (B.P. 301,727, 6.11.28. Switz., 3.12.27).—The salt-like compounds of 5:5-disubstituted barbituric acids and pyramidone are obtained in purer condition by combining salts of the two ingredients in a saturated aqueous solution of pyramidone as medium.

C. HOLLINS.

Manufacture of difficultly soluble salts of organic bases and alkaloids. I. G. FARBENIND. A.-G. (B.P. 295,656, 15.8.28. Ger., 16.8.27).—Non-hygroscopic salts of alkaloids (quinine, strychnine) and of the bases described in B.P. 267,169 (B., 1927, 379), 274,058 (B., 1929, 264), and 236,087 (B., 1928, 347) are prepared by adding to a solution of the hydrochloride the sodium salt of a methylenedisalicylic acid, methylenebis-2:3-hydroxynaphthoic acid, or similar acid of the type $R \cdot CH_2 \cdot R'$ in which at least one of the aromatic residues *R* and *R'* carries one or more carboxylic or sulphonic groups.

C. HOLLINS.

Synthesising and transmuting antirachitic substances and the like, and the products thereof. C. M. RICHTER (B.P. 296,053, 10.8.28. U.S., 24.8.27).—Ultra-violet rays of about 3022 Å. and longer wavelength (*i.e.*, capable of passing through glass) are able to convert vitamin-*A*, contained in its preparations, into vitamin-*D* in about 40 hrs.

C. HOLLINS.

Manufacture of physiologically active substances from the anterior lobes of the hypophysis. I. G. FARBENIND. A.-G. (B.P. 291,018, 23.5.28. Ger., 23.5.27).—By extracting anterior lobe of the hypophysis separately in an organic solvent (acetone) and water (pH 2.5—9) there are obtained (a) an œstrus-retarding substance and (b) an œstrus-promoting substance; both are unstable above 60°.

C. HOLLINS.

Manufacture of hormones. I. G. FARBENIND. A.-G. (B.P. 295,680 and Addn. B.P. 323,474, 27.6.28. Ger., [A], 17.8.27, [B] 26.4.28).—The hormone of the anterior lobe of the hypophysis is obtained from the urine of pregnant women or other human or animal fluids, which may be concentrated and/or purified or freed from the ovarian hormone by previous extraction with ether, or by precipitation with a water-soluble organic liquid. The ovarian hormone can be recovered from the ether extract, and the crude precipitate obtained above is purified by fractional precipitation of its aqueous solution with (A) acetone, alcohol, etc., or (B) with either an equal quantity of alcohol and repeating the precipitation if necessary, or, when the initial precipitation of the crude material is carried out with acetone, with a quantity of alcohol equal to about 80% of the water added, the filtrate being worked up as in (A).

E. H. SHARPLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Metallic silver in undeveloped photographic layers. I. F. WEIGERT and F. LÜHR (Z. wiss. Phot., 1930, 27, 312—337; cf. B., 1928, 212, 348).—Further evidence is obtained for the existence of free metallic silver in undeveloped photographic emulsions. The last traces of silver thiosulphate can be removed from the plate by treating it a second time with the fixing solution after an intermediate washing with water. Any silver left after this treatment can be dissolved by means of persulphate solution, and is therefore not present as silver sulphide. The rôle of this free metallic silver in photographic phenomena is discussed.

O. J. WALKER.

Destruction and intensification of the image by development in bright light. LÜPPO-CRAMER (Z. wiss. Phot., 1930, 27, 305—311; cf. B., 1928, 693).—Curves are given which show the strong bleaching action of red light on plates which have been previously exposed and then desensitised. The weakening of the latent image is not due to the action of the desensitiser (Pina-cryptol Green), and is strongest in the weakly exposed portions. This effect is opposite in nature and probably related to the Sterry effect.

O. J. WALKER.

See also A., Feb., 174, **Law of blackening of photographic plates** (SEITZ and HARIG). **Photochemical oxidation and reduction** (STEIGMANN). **Photographic photometry** (VALENKOV).

PATENTS.

Production of light-sensitive layers by means of diazo compounds. KALLE & Co. A.-G. (B.P. 296,725, 25.8.28. Ger., 6.9.27).—For the sensitive layer a mixture of two or more diazo compounds which are not converted into phenols by alkalis (e.g., diazotised *o*-aminophenols) is used; preferably these are chosen so that one of them is not completely decomposed during the exposure to light. Examples are diazo compounds of *o*-amino-5-diethylaminophenol and 4-chloro-*o*-aminophenol-6-sulphonic acid (developed with ammonia for brown); dianisidine and 3-amino-5-sulphosalicylic acid (reddish brown); 3:3':6:6'-tetramethoxybenzidine and *o*-amino-5-diethylaminophenol (brown); *o*-amino-5-diethylaminophenol, 4-chloro-*o*-aminophenol-6-sulphonic acid, and 3-amino-5-sulphosalicylic acid (yellowish brown).

C. HOLLINS.

Preparation of light-sensitive layers by means of ferric salts. G. KÖGEL (B.P. 302,282, 7.12.28. Ger., 13.12.27).—A ferric salt, e.g., ferric ammonium citrate, is applied to paper (etc.) together with a ketone capable of forming a coloured ferrous compound. After drying, exposure to light under a negative produces at once a coloured positive which is washed with water and may be mordanted. Suitable ketones are isonitroso-acetylacetone, nitroso- β -naphthol, β -naphthaquinone-4-sulphonic acid.

C. HOLLINS.

Development of positive diazo[*type*] prints. F. VAN DER GRINTEN (B.P. 295,993, 11.8.28. Holl.,

22.8.27).—The exposed print is passed between rollers the lower of which is partly immersed in an alkaline developing solution and thus deposits a thin film of solution upon the print. The alkali used is preferably sodium or potassium carbonate, with the addition of phosphates or citrates as buffer salts. C. HOLLINS.

Photographic sensitised [metal] discs. D. A. MCCOWAN (B.P. 302,348, 20.11.28. Can., 15.12.27).

Transparencies [with stereoscopic effect]. J. MCCREATH (B.P. 324,691, 2.10.28).

XXII.—EXPLOSIVES; MATCHES.

Decomposition of nitrocelluloses by volatile solvents. J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 149—150).—Nitrocellulose explosives prepared with a volatile solvent show after ageing some 600 times as rapid an emission of nitrous vapours as explosives prepared without such solvents. H. F. GILLBE.

PATENTS.

Smokeless powders. IMPERIAL CHEM. INDUSTRIES, LTD., and O. W. STICKLAND (B.P. 324,044, 5.9.28).—In smokeless powders, made without volatile solvents, the nitrocellulose consists wholly or mainly of the insoluble variety, containing 13.2% N or more (preferably above 13.45% N). The higher viscosity of this insoluble guncotton is partly reduced by using an increased amount of gelatiniser, and this enables the paste to be pressed at lower temperatures and pressures. The finished powder is free from brittleness. W. J. WRIGHT.

Blasting cartridges and appliances. D. HODGE (B.P. 324,617, 18.9.27).

XXIII.—SANITATION; WATER PURIFICATION.

Chlorination of sewage and effluents. F. W. MOHLMAN, E. HURWITZ, and C. C. RUCHHOFF (Proc. XI. Texas Water Works' School, 1929, 135—142).—The effect of chlorination on the biological oxygen demand was studied (Chicago). Ammonia-, but not organic, nitrogen is reduced by chlorination, but this reduction did not account for the effect of chlorination in reducing biological oxygen demand, which is not attributed to the oxidising action of chlorine. Thus chlorination cannot supplant the biological process, but may increase the efficiency of overloaded plant.

CHEMICAL ABSTRACTS.

Thermophilic digestion of sewage solids. I. Preliminary. W. RUDOLFS and H. HEUKELEKIAN (Ind. Eng. Chem., 1930, 22, 96—99).—If sewage solids which have been previously inoculated with ripe digested sludge are anaerobically digested at 45—55°, the time required for complete digestion is reduced to about one half that necessary at the low-temperature optimum point (28°). The yield of gas is increased from 650 to 870 c.c. per g. of fresh organic matter, whilst the average composition remains the same (CH₄ 70%, CO₂ 22%). The increased yield of gas, together with the smaller capacity of tank required, may render it an economic

proposition to carry out the digestion of sewage solids at the higher temperature. C. JEPSON.

Effect of iron compounds on digestion, sedimentation, and [sewage] sludge conditioning. W. RUDOLFS (Proc. N.J. Sewage Works' Assoc., 1929, Mar.).—When ferrous sulphate (26 g. per litre of fresh solids) was added to a seeded mixture, the reduction of volatile matter was greater during the first part of the digestion but smaller than the control after 35 days, whilst after 60 days the results were identical. Hence the rate of gas production was accelerated by several days. CHEMICAL ABSTRACTS.

Effect of alkaline substances on sewage-sludge digestion. A. J. FISCHER, W. RUDOLFS, and P. J. A. ZELLER (N.J. Agric. Exp. Sta. Bull., 1929, No. 424).—Small quantities of lime accelerated digestion, but excess after neutralisation of initial acidity caused retardation. With sodium nitrate the digestion was more rapid and complete. Dolomite and calcium carbonate were also used. CHEMICAL ABSTRACTS.

Collection and utilisation of [sewage-sludge] gases. A. J. FISCHER (Proc. XI Texas Water Works' School, 1929, 148—150).—Average domestic sewage should, if thoroughly digested, produce 530 c.c. of gas per g. of dry solid; the controlling factors are discussed. The gas contains approx. CH_4 70, CO_2 25, N 5%, and has a heating value of approx. 700 B.Th.U. per cub. ft. CHEMICAL ABSTRACTS.

Significance of hydrogen sulphide in partly treated sewage. E. WHEDBEE (Proc. XI Texas Water Works' School, 1929, 142—144).—Of the bacteria present in raw sewage, 9% reduce sulphur compounds. Oxidation of the hydrogen sulphide so produced to sulphur and finally to sulphuric acid causes disintegration of concrete sewers. CHEMICAL ABSTRACTS.

Gravimetric determination of thallium in mouse poisons. W. LEPPER (Z. anal. Chem., 1930, 79, 321—324).—The method previously given (B., 1926, 390) has been modified as follows. The material (5 g.) is boiled with nitric and sulphuric acids until the organic matter is destroyed, any thallic salt formed is reduced with sulphurous acid, and after expulsion of the excess of the latter the solution is neutralised with ammonia. Acetic acid (5 c.c.) is then added and the thallium precipitated in an aliquot portion of the solution by the addition of potassium iodide and weighed as thallous iodide. The above method requires less time than the chromate process previously recommended, and also yields slightly higher figures for the thallium content of the material analysed. H. F. HARWOOD.

Trends in municipal zeolite water-softening. W. J. HUGHES and H. B. CRANE (J. Amer. Water Works' Assoc., 1930, 22, 68—81).—Clear waters, free from iron and with temporary hardness only, can be softened either by a lime-soda or zeolite treatment according to the relative cost of reagents and the size of plant required etc. A similar water with additional permanent hardness is most efficiently dealt with by a

combination of lime and zeolite treatment. If the water is turbid, filtration before softening with zeolite is essential, and if lime is used during the pre-softening process, the water must be stabilised with carbon dioxide to prevent incrustation of the zeolite grains. Zeolite softening plant is more efficient and economical if it is constructed as a rectangular, reinforced concrete tank instead of like a circular pressure-filter. By this means larger and deeper tanks are available as they can be constructed on the site and the increased depth permits longer periods to elapse between regenerations. A rectangular under-drainage system is more efficient than a circular one, and therefore better distribution through the bed of both water and brine (for regeneration) is obtained. If the direction of flow be upward there is no packing together of the material; the small amount of frictional energy required to keep the material in suspension is constant, and so a regular rate of flow through the bed can be maintained. C. JEPSON.

Uses of aeration in water purification. M. PIRNIE (J. New England Water Works' Assoc., 1929, 43, 395—398).—In order to be non-corrosive, waters having bicarbonate alkalinities of less than 100 p.p.m. should not contain more than 2 p.p.m. of free carbon dioxide, resulting in a p_{H} value of 8.1—8.3. Carbon dioxide, in excess of this amount, present in spring or well water or in the effluent from sand filters may be removed by fine-spray aeration and the residual gas may be neutralised by lime or caustic soda as desired. Fine-spray aeration is also a valuable aid to flocculation, resulting in considerable economy in the use of alum and soda. Operating results of three American plants are given. C. JEPSON.

Sewage effluent standards. V. M. EHLERS (Proc. XI Texas Water Works' School, 1929, 171—173).

Gas mask. KATZ AND GRICE.—See II. **Waterworks' corrosion problems.** VAN GIESEN.—See X. **Sulphur-oxidation of activated sludge.** AYYAR and NORRIS; AYYAR. **Sewage sludge as fertiliser.** FRAPS.—See XVI.

PATENTS.

Volumetric-displacement apparatus suitable for controlling the supply of gas for chlorinating water or similar purposes. B. BRAMWELL (U.S.P. 1,746,186, 4.2.30. Appl., 15.12.27. U.K., 16.3.27).—See B.P. 277,869; B., 1927, 894.

Apparatus for use in carrying out exchange reactions [particularly for water-softening]. E. B. HIGGINS, ASST. to UNITED WATER SOFTENERS, LTD. (U.S.P. 1,745,421, 4.2.30. Appl., 27.11.26. U.K., 1.10.24).—See B.P. 248,414; B., 1926, 422.

Forming plastic explosives into strings, e.g., for manufacture of dynamite cartridges. (Sir) G. C. MARKS. From FINSKA FORCIT-DYNAMIT AKTIEBOLAGET (B.P. 324,440, 10.11.28).

Carbonisation of oil, fat, etc. (U.S.P. 1,738,202).—See II.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 11 and 18, 1930*

I.—GENERAL; PLANT; MACHINERY.

Production of electrical energy in chemical plant without cost. H. WOLLENBERG (Chem.-Ztg., 1930, 54, 9).—Waste water from several coolers etc. positioned at a height is caused to operate a turbine on falling to the ground level. S. K. TWEEDY.

Removal of dust and sulphur acids from power-station waste gases. I. Methods of separation in actual use. II. Methods and apparatus for gas purification employed in the chemical and metallurgical industries. III. Recent patents and processes for cleaning chimney gases. J. B. C. KERSHAW (World Power, 1929, 12, 533–536; 1930, 13, 15–19, 164–165).—I. As an indication of the total amount of solid matter discharged into the atmosphere from modern power stations, it is observed that 8–11% of the fuel weight is the average collected in the grit catcher. Dry catchers associated with the fan, e.g., Sturtevant, Davidson, and "Pneumonex," and the Rowntree water-spray plant are described.

II. Sulphur compounds are removed from gases by some means of washing, and various type of washing towers are noted. In the metallurgical industries, dry methods, e.g., Halberg-Beth and Lodge-Cottrell, are generally used for removing dust.

III. Recent patents are described, including Baron's method (cf. B.P. 314,110; B., 1929, 741) now installed at an electricity works in London. C. A. KING.

Absorption of gases and impurities in air under pressure. A. BARTH (Chem.-Ztg., 1930, 54, 143–144).—The removal of such constituents as ammonium chloride vapours, silicon fluoride, hydrofluoric acid, etc. from the exit gases of chemical works by absorption in water is greatly facilitated by carrying out this operation under pressure. A pressure of only 50–100 mm. of water has a markedly favourable effect on the absorption, and a description is given of a plant of this type suitable for use in technical practice.

H. F. HARWOOD.

Moisture in technical gases. I. Principles of calculation. F. LÜTH (Arch. Eisenhüttenw., 1929–1930, 3, 397–405; Stahl u. Eisen, 1930, 50, 169–170).—Mathematical. Equations are derived and curves given for calculating the weight of water vapour in a given volume of air of known humidity at N.T.P. and for calculating the volume of dry gas from that of the wet gas. A. R. POWELL.

[Calculation of tanks for high temperature and pressure.] J. S. THORP (Chem. Met. Eng., 1930, 37, 107).—In the expression given by Wood (B., 1930, 123) the term 3D should be replaced by D/3.

Centrifuges. VENZKE.—See II. [Filters for] caustic sludge. LORENZ.—See V. **High-pressure compressors.** BLAU.—See VII.

PATENTS.

Furnaces for liquid, powdered, or gaseous fuels. SIEMENS-SCHUCKERTWERKE A.-G., Assocs. of K. A. MAYR (B.P. 298,161, 3.10.28. U.S., 4.10.27).—In a boiler furnace with water-walls constructed of spiral tubes it is difficult to find room for secondary air inlets through the walls. In this invention the upper closure of the radiating or final-combustion chamber is formed as a pre-combustion chamber lined with refractory material, through the walls of which the fuel is admitted in two or more streams which clash, and the air axially, tangentially, and radially. B. M. VENABLES.

[Apparatus for] combined heat and pressure treatment of materials, particularly solid or liquid carbonaceous matter. M. MCGUINNESS (B.P. 324,692, 2.10.28).—A pressure- and heat-resisting vessel suitable, e.g., for the hydrogenation of oil or coal, is constructed of several co-axial shells in the spaces between which the pressure rises in steps. The innermost vessel is surrounded by a heat insulator which may be the gaseous pressure-resisting medium, alone or with a material such as asbestos. Each shell is supported on the next through the medium of rollers, and all pipes and manholes are in the same end so that the shells may expand independently. B. M. VENABLES.

Operation of steam-heated dryers and apparatus therefor. GEWERKSCHAFT GUSTAV, and O. AOCKERBLOM (B.P. 324,635, 25.10.28).—In a steam-heated dryer means are provided by which the pressure or temperature of the steam, the rate of supply of material, and the speed of rotation of the dryer are directly and automatically varied by the moisture content of the material, but the quantity of steam is not directly varied by a throttle valve owing to the risk of uneven heating by incomplete filling with steam. The amount of steam flowing into the apparatus is a measure of the moisture in the in-going material, so the primary control device may be a Venturi throat in the steam-pipe. Alternatively, the control may be regulated by the moisture in the out-going material, in which case a hair hygrometer may form the sensitive device. It is preferable that there should be lag between the controls affecting the supply of material and steam pressure, the former being reduced before the latter is increased. The steam at variable pressure may be obtained from a "pass-out" or "back-pressure" turbine, or boiler steam may first be used in devices such as briquette presses or pumps. B. M. VENABLES.

Heat-exchanging apparatus. E. HABER (B.P. 324,940, 3.9.28).—One of the fluids is maintained in continuous circulation by means of a propelling device, and fresh medium, *e.g.*, combustibles, is added to the circuit and withdrawn after use through conduits which are situated in such a relation to restrictions in the circuit that circulation is aided. The apparatus is stated to be suitable for the combustion of powdered fuel.

B. M. VENABLES.

Distillation of normally solid substances. R. W. JAMES. From NAT. ANILINE & CHEM. CO., INC. (B.P. 324,840, 4.3.29).—The vapours leaving a vacuum still for distilling the material are cooled to a temperature above the m.p. of the product, and the liquid and uncondensed gases (air leakage) are withdrawn together through a wet vacuum pump which discharges them under atmospheric pressure into a separator. Alternatively, the liquid and gases pass into a separator from which the pump removes only the gases, a quantity of the product liquefied by heat, or of a solvent for the product, flowing around a separate circuit to act as the seal for the pump. The treatment of such substances as naphthalene, phthalic anhydride, anthracene, and β -naphthol is indicated.

L. A. COLES.

Vaporisers. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF P. P. ALEXANDER (B.P. 315,723, 16.7.29. U.S., 16.7.28).—A vaporiser suitable for producing a reducing atmosphere in the process of arc-welding and similar purposes comprises an electric heater surrounded by a spiral baffle upon which the liquid, *e.g.*, methyl alcohol, is dripped under control of a needle-valve.

B. M. VENABLES.

Conveying materials in bulk by means of compressed air. (SIR) G. C. MARKS. From G. POLYSIUS (B.P. 324,392, 25.10.28).—The material is fed into the conveying pipe by means of a number of pressure vessels which are alternately filled and emptied; during the emptying period the vessel is under air pressure, derived from the conveying pipe, from the air supply pipe, or from a separate source. When the vessel becomes empty there is a sudden drop in pressure, and this fluctuation is made use of to change over to another vessel.

B. M. VENABLES.

Pulverising mills. P. LEGRAND (B.P. 312,047, 25.3.29. Austr., 18.5.28).—A high-speed pulveriser is provided with grinding rings which surround all the hammers, and the fineness of grinding is regulated by axial adjustment of the rings. Several methods of effecting this adjustment are described.

B. M. VENABLES.

Process and apparatus [colloid mill] for preparation of semi-colloids and uniform colloids. H. E. POTTS. From H. PLAUSON (B.P. 324,977, 8.11.28).—The material is subjected to successive crushing and disintegration in the same apparatus, which comprises a number of gear wheels in mesh performing the crushing between their teeth and flinging the material against a surrounding serrated surface, which may be either stationary or consist of other gear wheels. The ground material may subsequently pass through a slowly rotating centrifugal separator which returns coarse particles to be re-ground.

B. M. VENABLES.

Grading and separation of granular materials. J. LORD (B.P. 324,469, 1.12.28).—In an apparatus where a substantially horizontal current of air passes across a number of collecting hoppers in succession and blows the particles into the different compartments according to their weight and surface area, arrangements such as the use of louvres are made to vary the direction of the air current at the line where the feed-stream enters, also between that point and the outlet. The exhaust air may return through an expansion chamber to the fan.

B. M. VENABLES.

Apparatus for mixing meal or powder with finely-divided liquids or gases. E. MAHLKUCH (G.P. 459,832, 20.6.26).—The powder is fed by gravity through a hopper into a mixing chamber where it meets a stream of compressed air. This, in turn, blows it in a finely-divided form into a spray of gas or liquid supplied from a pipe controlled by a valve operated by the weight of the solid so that when the hopper is empty the valve automatically closes.

A. R. POWELL.

[Vapour compression] evaporating plants. A.-G. BROWN, BOVERI & CO. (B.P. 315,717, 16.7.29. Ger., 16.7.28).—An evaporating plant having a heat pump is initially heated up, without the use of any external apparatus or source of heat, by operating the pump to compress air which is circulated between the heating coils and vapour pump.

B. M. VENABLES.

Separation of salts or other solids from solutions thereof. [Spray dryer.] R. H. WHEELER (B.P. 324,331, 17.10.28).—A solution which it is desired to evaporate completely with formation of granular solid particles is sprayed into a con- or counter-current of air. The size of drops, distance they have to fall, temperature, and quantity of air, are all regulable to obtain the desired effect. The size of drops may be pre-determined by the size of jets and/or the head under which the solution is supplied to them. The apparatus described comprises an inner, vertical, evaporating chamber surrounded by an annular chamber for gases only, and provision is made for admitting the gases to either end of the evaporating chamber in a whirling manner. The sprayer may, if desired, spray upwards and its position may be varied, or it may even be placed near the bottom of the chamber.

B. M. VENABLES.

Production of films. W. H. PEASE (B.P. 324,694, 22.10.28).—Material in the form of a very thin sheet is prepared by evaporation of a solution which has been spread in a thin layer on a conveyor belt, preferably a metallic band, which is itself heated and runs through casings which are heated, provided with means for removing air, and heat-insulated. At least one of the drums round which the belt runs is cooled and the finished film is stripped from the belt while on that drum.

B. M. VENABLES.

Filters. J. A. PICKARD (B.P. 324,924, 1.8.28. Cf. B.P. 312,944; B., 1929, 627).—A filter of the type in which the passages for the flow of liquid become narrower in the direction of travel is constructed of a number of superposed sheets of wire gauze through which the fluid passes perpendicularly; the diminishing apertures are obtained by varying the number of meshes and/or the

diameter of the wires, and a filter aid may be used or not.

B. M. VENABLES.

Filter for compressed air. BRAUTECHNIK GES.M.B.H. (G.P. 459,658, 15.4.26).—The air passes through a spiral space formed between the turns of a cooling-worm surrounding the filter drum, whereby moisture and oil are condensed, thence through a coke filter into a space below the filter drum, and finally upwards through adsorptive carbon or silica gel contained in the body of the drum between two perforated plates, the upper of which is covered with cotton wool to prevent the filter material being blown through.

A. R. POWELL.

Internal heating of vacuum filter drums. R. WOLF A.-G. (G.P. 459,044, 28.5.25).—Steam-heated chambers are provided below and around the individual cells of the drum and are interconnected with one another and with a common source of steam supply at one side of the drum.

A. R. POWELL.

Presses for extracting liquids from solid materials. P. A. H. VILLEZ (B.P. 312,906, 27.4.29. Fr., 2.6.28).—The material is fed through a tapering, flat nozzle to a pair of slow-speed rollers by means of a screw-conveyor. The last is provided with a device to prevent slip, comprising a disc with notches on its edge which intercalates with the threads of the screw. The diameter of the pressure rollers is 4–6 times that of the screw, so as to maintain the angle of nip between 70° and 80°, and the speed of the rollers is not greater than 2 r.p.m.

B. M. VENABLES.

Apparatus for straining fluids. F. BAILEY and F. H. JACKSON (B.P. 324,398, 26.10.28).—In an apparatus as described in B.P. 246,551, in which sectors of straining material are supported between the spokes of a wheel, the sectors are composed of accordion-pleated gauze or of corrugated perforated sheet metal, and are supported between radial bars which have V-notches corresponding to the corrugations. In the event of the strainers being double (coarse and fine), flap valves are provided on the inlet side through which trapped material is released when the sector concerned is subjected to cleansing by back-flush.

B. M. VENABLES.

Preparation of emulsions. R. AUERBACH and W. STEINHORST (B.P. 325,033, 28.11.28).—The liquid forming the continuous phase is put into rapid rotation, e.g., in a centrifugal pump, and the other liquid is admitted to it through a nozzle situated, e.g., on the axis of the pump at the centre of the vortex formed in the first liquid. The interior of the pump casing may be provided with disintegrating pins.

B. M. VENABLES.

Centrifugal separators. AKTIEB. SEPARATOR (B.P. 317,761, 19.8.29. Swed., 21.8.28).—A liquid-tight and detachable collecting dome for a centrifugal separator is attached to the body and to the outlet pipe in such a way that differential swelling of the packings may take place without affecting the tightness of the joints.

B. M. VENABLES.

Centrifugal machines or separators. RAMESOHL & SCHMIDT A.-G. (B.P. 310,544, 26.4.29. Ger., 27.4.28).—A construction of hinged lid, containing the collecting vessels, and detachable outlet pipes is described for a gas-tight centrifugal machine.

B. M. VENABLES.

Centrifugal separator bowls. AKTIEB. SEPARATOR (B.P. 316,181, 11.7.29. Swed., 24.7.28).—A centrifugal bowl having outlets for the continuous discharge of both lighter and heavier constituents, especially suitable for cases where the solid matter is present in the lighter constituent, is provided with guides over which the lighter constituents issuing from the separating chamber pass inwardly over the outside, then outwardly over the inside, the result being that the final discharge is at a radius greater than that of the actual collecting zone, thus giving a steep slope upon which solid particles are unlikely to accumulate.

B. M. VENABLES.

Cleaning of centrifugal bowls. AKTIEB. SEPARATOR (B.P. 310,418, 23.4.29. Swed., 25.4.28).—The solid matters are caused to deposit on a lining of flexible material which may be slit radially and provided with handles to permit easy removal.

B. M. VENABLES.

Method and apparatus for selectively discharging liquids according to their electric conductivity, particularly applicable for selecting pure water from water of condensation and returning it to boilers. PAPETERIES NAVARRE (B.P. 305,163, 7.12.28. Fr., 1.2.28).—The electrical conductivity of the condensate is used to determine whether successive batches may be used or must be run to waste. A closed vessel is filled and emptied through a two-way valve, the position of which is determined by a float in the vessel; the emptying branch is connected to another two-way valve, the position of which is determined by the *P.D.* between electrodes immersed in the liquid and controls whether the liquid will be used or wasted. In the event of failure of any part of the mechanism, safety is ensured by causing the first valve to stop in the mid-position, or the second valve in the waste position. Steam or other gas pressure may be used to force the water back into the boiler directly; if the valve is in the waste position this pressure is cut off.

B. M. VENABLES.

Compound for use as water softener. G. R. BRUTON (B.P. 325,099, 12.2.29).—A mixture of equal parts of soda ash, "Crex" (sodium carbonate and bicarbonate), and powdered borax is claimed.

H. ROYAL-DAWSON.

Exothermic catalytic gas reactions. H. HARTER (B.P. 325,028, 24.11.28).—In a reaction vessel, a heat-insulating cylinder is placed between the reaction chamber and the outer pressure-resisting cylinder. The gas for the reaction is passed through the space between the insulator and the outer wall, while a separate current of cooling gas is circulated, by a pump, through the space between the insulator and the reaction chamber, through a cooler, and back again, the circulation being controlled by the pump independently of the amount of gas subjected to the reaction. The circulating current may be split off from and rejoin the incoming gas, or may be entirely independent; even in the latter case it is preferably of the same composition and at the same pressure.

B. M. VENABLES.

Apparatus for conducting catalytic gas reactions under pressure. NORSK HYDRO-ELEKTRISK KVAELSTOF-A./S. (N.P. 43,725, 4.2.26).—The apparatus comprises a pressure vessel containing several concentric

contact chambers separated by empty annular chambers. The gases enter the vessel at the periphery and traverse alternately the contact chambers and the annular chambers.

A. R. POWELL.

Revivification of adsorptive materials. N. V. MONTAAN METAALHANDEL, Assees. of H. SIEGENS (G.P. 459,346, 24.11.22).—The material is heated by electrical resistance to expel adsorbed gases or vapours, with or without a treatment with another gas or vapour, *e.g.*, air or steam. The resistor may consist of the substance itself, or with the addition of a more conductive material, or after suitable chemical treatment to increase its conductivity. Alternatively, a resistor element may be embedded in it.

A. R. POWELL.

Measurement of the quantity of a gas or liquid passing through a tube. S. LÖFFLER (Aust.P. 109,123, 27.8.25. Ger., 28.8.24).—The movement of the liquid or gas through the tube lifts a conical valve from its seating, and this motion is communicated by a thread to an iron balance weight moving in an induction coil. An indicator device working on an electromagnetic principle records this motion.

A. R. POWELL.

Means for maintaining constant pressures, liquid level, specific gravities, etc. F. MOUFANG (B.P. 324,929, 1.11.28).—In a known system in which a jet of compressed air is more or less obstructed with consequent variation of pressure in the air-supply pipe, the pressure variations acting through relays to effect the required control, the obstructing valve is often affected by the pressure of the jet. In this invention the obstruction moves at right angles across the jet; it may, *e.g.*, be mounted on the pointer of a pressure gauge, on the arm produced of a float valve, or on the stem of a hydrometer.

B. M. VENABLES.

Vacuum measurement. A.-G. BROWN, BOVERI & Co. (Aust.P. 109,131, 21.5.26. Ger., 2.6.25).—The apparatus comprises a U-shaped, bifilar, bimetallic strip through which is passed a constant current. The temperature of the strip changes with the degree of vacuum, and the consequent variations in the shape of the strip are rendered visible by means of an indicator needle moving over an appropriate scale.

A. R. POWELL.

Indicating thermometer filled with two liquids. STEINLE & HARTUNG GES.M.B.H. (G.P. 458,926, 16.3.26).—A thermometer of the type in which the expansion caused by heat operates an indicator by means of an elastic membrane is completely filled with two immiscible liquids neither of which boils within the temperature range of the thermometer and one of which has a higher coefficient of expansion than the other.

A. R. POWELL.

Vessel for conducting chemical tests and measurements, *e.g.*, titrations. J. LINDNER (G.P. 459,627, 27.11.26).—The vessel of any suitable shape is provided with a pocket-like, external, auxiliary vessel connected to the main vessel by an outlet above the surface of the liquid therein. The auxiliary vessel serves to retain a portion of the liquid to be titrated until the end-point has been reached with the main volume, and is thus applicable for titrations requiring an outside indicator.

A. R. POWELL.

Evaporator and process of evaporation. P. B. SADTLER, ASSR. to SWENSON EVAPORATOR Co. (U.S.P. 1,735,979—80, 19.11.29. Appl., [A] 9.7.26, [B] 6.8.27).—See B.P. 321,698; B., 1930, 87.

Fractional distillation. R. B. CHILLAS, JUN., ASSR. to BARRETT Co. (Re-issue 17,595, 18.2.30, of U.S.P. 1,612,572, 28.12.26).—See B., 1927, 129.

Furnace wall construction. M. H. DETRICK Co. (B.P. 307,919, 26.2.29. U.S., 17.3.28).

Chemical fire extinguishers. T. H. BARTON (B.P. 325,046, 13.12.28).

Ammonia as lubricant (U.S.P. 1,739,957).—See VII. Filtering etc. of liquids (B.P. 325,004).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Composition of peat-forming plants and varieties of peat. I. G. STADNIKOV and A. BARYSCHEVA (Brennstoff-Chem., 1930, 11, 21—23).—Methods previously used for the analysis of peat are criticised. The composition of samples of peat taken at different depths of the Elektroperadatsche peat bog has been compared with that of the peat-forming plants (*Sphagnum parvifolium*, *Eriophorum vaginatum*). The samples were analysed by extracting successively with (a) benzene-alcohol mixture, (b) hot water, and (c) 1% sodium hydroxide; the last extract was subdivided into humic acids, which were precipitated by sulphuric acid, and acids soluble in ether and alcohol, respectively. The lignin content of the residue was determined by Willstätter's method and the cellulose calculated by difference. During the peat-forming process the sugars, pectins, and cellulose of the plant disappear; the lignin decreases in amount and shows also a progressive increase in its carbon content. At the same time insoluble salts of monocarboxylic and other more complex acids appear; these are regarded as intermediate products in the formation of the humic acids from lignin.

A. B. MANNING.

Formation of fusain from a comparatively recent angiosperm. W. P. EVANS (New Zealand J. Sci. Tech., 1929, 11, 262—269).—Samples of fusain, estimated to have been laid down at least 10,000 years ago, were probably formed by direct heat action of a pumice shower on the original wood, and modified by prolonged bacterial action under anaerobic conditions subsequently. A series of photographs is provided.

E. HOLMES.

Coking power and swelling of coal. I. H. A. J. PIETERS (Rec. trav. chim., 1930, 49, [iv], 289—306).—A knowledge of the coking properties of a coal may be obtained by determining its volatile matter, heating 1 g. of the coal at 850° for 2½ min. and measuring the volume of the coke button produced, determining its Campredon caking index, measuring its softening point when heated at the rate of 9°/min., determining its swelling power when heated (5°/min.) under pressure, or finding the temperature at which rapid evolution of gas begins and the rate at which gas is evolved when 2 g. of coal are heated at the rate of 4°/min. Examination of 28 samples of coking, non-coking, and gas coals by

the above methods shows that (i) a correlation may be made between the amount of volatile matter in the coal, its caking index, and the volume of the coke button, high volatile matter corresponding with high caking index and a large coke-button volume; (ii) for coals with a gas content of 18–25%, their coking and swelling properties are a function of their volatile matter content, but with coals having a gas content above 30% and with certain blends of coal this relationship no longer holds good; (iii) coking coals have lower softening points and commence to evolve gas at lower temperatures than low-volatile non-coking coals; (iv) coking coals begin to swell considerably and to soften at 360–420°. When the plasticity diminishes the swelling decreases and the volume of the coal becomes constant, but at 650–825° a definite shrinkage occurs, whereas a non-caking coal never swells but gradually shrinks during the whole period of its destructive distillation; (v) gas coals give low swelling values when tested in an open crucible, and when coking takes place under pressure these coals show only a temporary swelling and afterwards shrink; (vi) Campredon's method of evaluating the caking power of a coal gives uncertain results, and is too tedious; it is therefore not recommended as a routine test. Theoretical reasons are advanced for these conclusions and their application to blending problems is discussed. C. B. MARSON.

Relation between caking power and coking test of coal. I. Tests by Lessing's apparatus. II. Influence of weathering. S. IKI (J. Soc. Chem. Ind. Japan, 1930, 33, 9–11 B).—The gas and coke yields in the Lessing apparatus tend to increase with decrease of caking power. The tar yield rises and then falls again. The production of tar from the four coal constituents decreases in the order γ , β , α , ulmin, whilst that of gas increases. Yield of gas here refers to volume, which is in inverse ratio to the calorific value. Coals of various caking powers were exposed to temperatures from 100° to 250° for 10 hrs. and then tested. It was found that the volume of gas yield tended to increase owing to replacement of hydrocarbons by carbon monoxide. The tar yield decreased. Slight weathering of caking coal increased the swelling power. C. IRWIN.

Complete gasification of coal: tests of the suitability of coal for complete gasification. R. MEZGER and T. PAYER (Gas- u. Wasserfach, 1930, 73, 1–8).—The "double-gas" process for complete gasification provides a valuable adjunct to existing methods of gas production, mainly in respect of the rapidity with which the plant can be brought into operation and the ease with which the gas so produced may be blended with coal gas, producer gas, etc. to give a mixture of the desired calorific value. The mode of operation of the plant is described, and special reference made to difficulties experienced; these are mainly concerned with the properties of the coal used. A suitable coal should be of uniform size, free from fine material, non-swelling and non-caking, should give a high yield of gas and tar in proportion to the residual coke, and should yield its volatile constituents at the lowest possible temperature, a gas of high calorific value, and a tar capable of being readily cracked to a high-value gas. Methods

of examination of coal are described in detail with the aid of diagrams, and the results obtained from actual tests of various coals are tabulated. Assistance in evaluating a coal as to its suitability is given in recommendations for the limiting values obtained by the various tests, with special reference to swelling and caking, which greatly affect the working of the "double-gas" plant. D. G. MURDOCH.

Desulphurisation of gases either free from or containing ammonia by the process of the Ges. f. Kohlentechnik. W. GLUUD, W. KLEMP, and F. BRODKORB (Brennstoff-Chem., 1930, 11, 23–27; cf. B., 1927, 321; Ferbers, B., 1929, 5).—The plant at the Zeche "Viktoria," Lünen, is described. It has a daily throughput of 20,000 cub. m. of gas containing 10–11 g. of hydrogen sulphide per cub. m. The daily consumption of sodium carbonate is 144 kg., and of iron 23 kg.; the daily yield of crude sulphur is 255 kg., containing 55% S, 9% Fe (as hydroxide and sulphide), and 36% of water and other impurities. The material extracted from the new wooden grids by the alkaline solution caused undue foaming, which adversely affected the activity of the iron hydroxide suspension and interfered with the separation of the sulphur; it was therefore removed by pre-extraction. The activity of the suspension was determined in the laboratory by the rapidity of coloration on the addition of hydrogen sulphide and the subsequent rate of re-oxidation with compressed air. Luxmasse or Duisberger-masse (0.2–0.3% in 0.5–0.6% sodium carbonate solution) proved to be suitable materials for making up the suspension, the activity of which could be improved by the addition of a manganese suspension. The thiosulphate content of the suspension is preferably not allowed to rise above 5%. The expensive consumption of sodium carbonate can be avoided if the gas is desulphurised while still containing the ammonia. Objections to this procedure, e.g., the possible formation of a calcium sulphate sludge due to the increased concentration of ammonium sulphate in the liquor, or the re-addition of hydrogen sulphide to the purified gas from the wash oil in the benzol scrubbers, are shown to be without basis. A. B. MANNING.

Cracking of primary tar from a coal of Donetzki Basin under hydrogen pressure. V. N. IPATIEV, A. D. PETROV, and I. Z. IVANOV (J. Appl. Chem. Russia, 1929, 2, 429–435).—The cracking at 380–420° in presence of alumina and ferric oxide at 70 atm. initial hydrogen pressure gave a lower yield of low-boiling substances soluble in sulphuric acid and a higher yield of high-boiling fractions than in absence of hydrogen. By alteration of the conditions of cracking the aromatic content of the light products can be controlled.

CHEMICAL ABSTRACTS.

Cracking of gaseous saturated hydrocarbons. W. J. PIOTROWSKI and J. WINKLER (Przemysl Chem., 1930, 14, 49–64, 83–92).—Gaseous hydrocarbons possessing 2–5 carbon atoms undergo pyrolysis at all concentrations to give high yields (70%) of unsaturated hydrocarbons. All hydrocarbons tend on prolonged heating to undergo conversion into methane, carbon, and hydrogen, and for each concentration and

composition of gas submitted to pyrolysis there exist an optimum temperature and duration of heating at which maximum yields of olefines are obtained. These are 730° and 8 sec. for the propane-butane gas derived from gasoline, 740–765° and 10 sec. for Cross distillation gas containing methane, and 780° and 11 sec. for Boryslaw gasoline-free natural gas, possessing a high air and methane content. Carbon formed during pyrolysis catalyses the further disintegration of the unsaturated hydrocarbons formed, thus leading to enhanced soot formation. The process of pyrolysis consists below 730° in the formation of propylene from propane, and of propylene and butylene from butane. Between 730° and 800° propane yields chiefly ethylene, whilst butane yields ethylene and ethane. Above 800° acetylenic and aromatic hydrocarbons appear, together with carbon, hydrogen, and methane; the proportion of polynuclear aromatic hydrocarbons rises with temperature.

R. TRUSZKOWSKI.

Effect of branched alkyl groups on anti-knock properties of the lead triethylbutyl compounds. H. GILMAN, O. R. SWEENEY, and J. D. ROBINSON (Rec. trav. chim., 1930, 49, [iv], 205–207).—The preparation of the four lead triethylbutyl compounds and their examination for anti-knock efficiency by the bouncing-pin method are described. All four compounds are of about equal effectiveness, viz., 85% as efficient as is lead tetraethyl on a molecular basis (cf. A., 1929, 1092, 1472).

C. B. MARSON.

Influence of the cold-resistivity of neat's-foot oils on the greasiness of the compound oils prepared therefrom. P. CUYPERS (Chem.-Ztg., 1930, 54, 30–31).—The neat's-foot oils less resistant to cold (of higher m.p.) have a lower linoleic acid content (which confers improved chemical stability) and a higher stearic acid content than the similar oils which are more resistant to cold, and it is shown that the use of only the most cold-resistant and expensive neat's-foot oils for compounding with mineral oils is unnecessary; the cheaper type of oils, having a smaller cold-resistivity, are found to offer considerable advantages when so used, producing better lubrication properties and prolonged chemical stability in the compound oils.

S. K. TWEEDY.

Centrifuges for drying [solids] and separating [liquids]. VENZKE (Bremistoff-Chem., 1930, 11, 27–28).—Two types of centrifuge are described, suitable, respectively, for the continuous drying of small coal and the separation of heavy and light oils, or of oils from sludges.

A. B. MANNING.

Sulphite-cellulose waste liquor. SCHWALBE.—See V. **Fertilisation and the coal industry.** SCHULTE-OVERBECK.—See XVI.

PATENTS.

Coke ovens. P. E. VERPEAUX, and UNION CHIM. BELGE, SOC. ANON. (B.P. 324,584, 12.4.29).—The fuel gas is supplied to the vertical flues of a horizontal coke oven through chambers immediately underneath the flues, while air, in amount insufficient for the complete combustion of the gas, enters at the bottom of the flue from regenerators situated below the ovens. In the walls between the individual regenerators are air-expan-

sion chambers, connected on one side with pipes supplying air under pressure and on the other side with auxiliary flues which open at different levels in the heating flues, and thus supply secondary air to complete the combustion of the excess gas.

A. B. MANNING.

Coke ovens. W. DIAMOND (B.P. 324,195, 20.12.28).—To facilitate the withdrawal of charges which adhere to the oven walls, liners are provided within the ovens. These may be in the form of plates of wedge section (thin end downwards), and adapted to be raised by suitable hoisting gear, or they may consist of plates normally slightly inclined to the oven walls and adapted to be rocked back from the face of the charge.

A. B. MANNING.

Retorts for treating coal, peat, shale, etc. D. D. SHELTON (B.P. 324,348, 19.7.28).—A vertical retort comprises a central heating chamber down which pass the hot combustion gases from a furnace, and on two sides of which are narrow carbonising chambers. The outer walls of the latter are movable so that the width of the carbonising chambers can be varied as desired. The inner sides of the movable walls are provided with spaced louvres in between which are movable blades connected to an agitating mechanism. Means are provided for feeding the material continuously in at the top of the carbonising chambers and discharging the product through regulatable apertures on to hinged floors the free ends of which engage with the radial blades of internally cooled rotating drums in such a manner as to shake the carbonised material over the outer surface of the cooling drums into a hopper. The volatile products of distillation pass through the louvred wall to condensing chambers adjacent thereto.

A. B. MANNING.

Apparatus for recovery of valuable constituents in coal, peat, lignite, etc. C. C. LARSEN (B.P. 307,021, 25.2.29. Denm., 1.3.28).—The pulverised fuel is distilled in an internally heated retort, whence it is conveyed directly to the furnace of a steam-raising plant. After recovery of the tar etc. in a suitable condensing system, a part of the distillation gases is passed through a pipe coil within the furnace and recirculated through the retort, thus forming the heating medium for the distillation.

A. B. MANNING.

Apparatus for distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 324,212, 30.7.28).—An inclined rotary retort is divided longitudinally into a number of separate compartments. Helically twisted blades are provided at the ends of the retort for feeding the material from the charging chute into the compartments and for conveying the carbonised material into the discharge chute. Stationary gas-inlet and -outlet pipes are provided at the ends of the retort through which a gaseous heating medium may be brought into direct contact with the material.

A. B. MANNING.

Apparatus for separation from solid residues of oils obtained in the destructive hydrogenation of varieties of coal, tars, mineral oils, etc. under pressure. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,791, 11.9.28. Addn. to B.P. 312,657; B., 1929, 633).—The residues are mixed with solvents

and the mixture is filtered in an apparatus to and from which the solvent, the mixture, the extracted products, and the extracted residues are continuously supplied and removed. The solvents used are preferably oils of boiling range 150—300°, derived from the destructive hydrogenation process itself. The residues from the filter may be subjected to a subsequent low-temperature carbonisation. A. B. MANNING.

Fractional dry distillation of organic materials. J. MÜLLER (G.P. 457,500, 28.1.25).—The material, *e.g.*, coal, peat, wood, is fed in small quantities into the apparatus wherein it is heated at once to the distillation temperature. The distillation gases are separated into their different fractions by being passed through filters which are heated by the gases themselves in successive zones in such a manner that the temperature of the filter at the gas outlet lies directly above the initial temperature of condensation of the succeeding stage. The filters are connected to a series of openings serving as gas outlets and leading to the corresponding condensers. One or more filters may be arranged below the distillation chamber and connected directly thereto. A. B. MANNING.

Dry distillation of brown coal. A. G. PULT (F.P. 634,300, 12.5.27. It., 25.5.26).—The plant consists of a furnace containing one or more retorts the heads of which are especially long and so arranged that compounds of the aliphatic series of which brown-coal tar consists are converted therein into aromatic compounds. A. B. MANNING.

Gasification and carbonisation of earthy moist fuels, *e.g.*, raw brown coal. C. ARNEMANN (G.P. 458,879, 12.2.22).—The coarser fuel is separated from the dust, and is fed directly into the generator. The dust is burnt in a combustion chamber in the lower part of the generator, and the hot combustion gases are used for drying, carbonising, and gasifying the coarser fuel in the generator shaft. A. B. MANNING.

Manufacture of water-soluble products from lignite and similar fossil materials. I. G. FARBERIND. A.-G. (B.P. 323,781, 10.10.28. Addn. to B.P. 284,670; B., 1929, 505).—Lignite degradation products, soluble in dilute acid, are treated with chlorine until the medium becomes acid, if not already so, and the chlorine treatment is continued with the gradual addition of alkali in such manner that the reaction medium remains just acid to litmus, and only becomes more strongly acid towards the end of the process. H. S. GARLICK.

Production of low-temperature coke. C. B. WATSON and R. H. CARR (B.P. 323,829, 20.10.28).—Bituminous coal is charged under pressure, *e.g.*, by means of a reciprocating ram, into the bottom of a cylindrical chamber, wherein it travels upwards countercurrent to a gaseous heating medium, *e.g.*, superheated steam. A central vertical shaft carries a conveyor blade which controls the upward motion of the coal. The coke is withdrawn in a compact form, being propelled through a tangential conduit near the top of the chamber by means of a spiral conveyor. A number of arms having their outer ends connected by means of a ring are attached to the upper part of the shaft. The arms and the ring carry depending fingers which by their rotation

break up the surface of the fuel so as to render it readily pervious to the heating medium. The volatile products of distillation are withdrawn through a circular, perforated manifold at the bottom of the chamber, and pass to a suitable condensing system. A. B. MANNING.

Preparation or revivification of active carbon. METALLBANK U. METALLURG. GES. A.-G., and H. VON HALBERN (G.P. 459,347, 14.8.25).—The carbon is subjected to alternate short reduction and oxidation treatments in a divided electrolytic cell, in which the electrolyte contains a substance which in the anode compartment becomes oxidised to an active oxidising agent and in the cathode compartment is reduced to a powerful reducing agent. A. R. POWELL.

Composite fuel. J. A. GREENE, and BINDPHAST PRODUCTS, LTD. (B.P. 324,081, 18.10.28).—A bituminous or other combustible substance, *e.g.*, small coal, is mixed with a relatively small quantity of calcined dolomite, and with sufficient of an aqueous solution of chlorine or hydrochloric acid to form a plastic coherent mass, which is then briquetted. Alum may be added to the mixture to make it dry more rapidly, and clay to produce a slower-burning fuel. A. B. MANNING.

Determination of moisture in coal and similar substances. H. HEINICKE (G.P. 458,927, 29.1.26).—The coal is packed round an electrically-heated wire within a suitable containing vessel; the moisture content of the coal is then determined from the change in the resistance of the wire. A. B. MANNING.

Gas-making retorts. C. L. SINCLAIR and C. M. CROFT (B.P. 323,909, 31.12.28).—In retorts having projector-type coal-charging machines, a shield is provided within each retort which normally fits against the lid, but which can be moved forward during the charging operation, *e.g.*, by means of a rod passing through a hole in the lid and screwing into a spigot on the shield, so as to act as a barrier to prevent coal reaching the mouthpiece, while permitting gas to pass freely to the ascension pipe. A. B. MANNING.

Apparatus for production of gas from wood and dry purification thereof. E. M. RAUJOUAN (F.P. 633,237, 23.4.27).—The plant comprises a generator with a container for the wood situated above the hearth, a cooling arrangement attached thereto and consisting of two horizontal collecting chambers connected by four narrow pipes, a purifier with a lower chamber for the separation of the solid gas impurities, a condenser, a liquid seal, and a valve. A. B. MANNING.

Generation of hydrogen. R. BATTIG (B.P. 294,113, 14.7.28. Ger., 16.7.27).—The methane fraction obtained by the liquefaction of coke-oven gas is converted by interaction with steam and/or oxygen into carbon dioxide and hydrogen. The carbon dioxide is removed by washing the gas mixture with water under pressure, and the hydrogen is purified by fractional liquefaction, *e.g.*, by the use of liquid nitrogen. The gas may finally be washed with liquid nitrogen in such a manner as to give a mixture of hydrogen and nitrogen suitable for the synthetic manufacture of ammonia. A. B. MANNING.

Hydrogen and hydrogen-nitrogen mixtures. NORSK HYDRO-ELEKTRISK KVAELSTOF-A./S. (F.P. 627,665, 18.1.27. Norw., 13.2. and 9.12.26).—Steam, alone or mixed with air, is passed through carbon raised electrically to incandescence, and hydrogen, alone or mixed with nitrogen, respectively, is obtained from the water-gas produced by removal of the carbon dioxide etc. Alternatively, the water-gas is employed to reduce ferric oxide and, after removal of the excess steam and carbon dioxide, is repressed through the incandescent carbon together with the requisite quantity of steam; the reduced iron is used to decompose further quantities of steam, the first, impure hydrogen thus obtained being added to the circulating reducing gas and the pure hydrogen subsequently obtained being used for the preparation of ammonia. S. K. TWEEDY.

Production of a mixture of hydrogen and carbon monoxide by decomposition of hydrocarbon gases or vapours in the presence of steam. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 323,864, 16.11.28).—Coke-oven gases etc. are mixed with a limited supply of steam, *e.g.*, 5–16% by vol. for a gas containing 30% CH_4 , and passed through a bed of preheated refractory material. The heat of the gases produced is used to preheat air, and the heat required to bring about interaction of the steam and the hydrocarbons in the gases is obtained by the combustion of the deposited carbon in the preheated air, each reaction period being followed by a reheating period.

A. B. MANNING.

Manufacture and utilisation of water-gas. FABR. NAT. DE PROD. CHIM. ET D'EXPLOSIFS SOC. ANON. (B.P. 317,460, 19.10.28. Belg., 17.8.28).—The residue from the carbonisation of a mixture of coal with about 15% of iron oxide, obtained as described in B.P. 296,443 (B., 1930, 7), is used in a water-gas generator. The gas thus produced has a higher calorific value than ordinary water-gas and is particularly suitable for the synthesis of light petrols, methyl alcohol, etc. A. B. MANNING.

Separation of hydrogen sulphide and hydrogen cyanide from gases. SOC. ANON. HOLLANDO-BELGE POUR LA FABR. DU COKE (B.P. 314,860, 11.6.29. Ger., 3.7.28. Cf. B.P. 195,061; B., 1923, 929 A).—The gases are washed with an alkali carbonate solution, from which the hydrogen sulphide and hydrogen cyanide are subsequently expelled by means of carbon dioxide. The gases from the expelling apparatus are passed through a purifying box in which the hydrogen sulphide is separated in any manner which does not fix the carbon dioxide, *e.g.*, by hydrated iron oxide, and are again used for expelling a further amount of hydrogen sulphide from the wash liquor. Preferably only the upper layers of the wash liquor are treated with these re-circulated gases, the lower layers being freed from hydrogen sulphide by means of carbon dioxide obtained by heating the bicarbonate liquor. A. B. MANNING.

Gas purification (removal of benzol). SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 636,162, 12.10.26).—The benzol is absorbed in activated carbon. Only very highly active carbon is used and the plant is constructed to work automatically. A. B. MANNING.

Shaft furnace for production of low-temperature tar. ALLGEM. VERGASUNGS-GES. M.B.H. (G.P. 459,307, 5.6.21. Addn. to G.P. 435,210; B., 1927, 291).—The design of the shaft furnace described in the prior patent is modified by reducing the area of cross-section of the upper part to about one third that of the lower.

A. B. MANNING.

Extraction of oil from solid fuels, shales, torbanites, etc. J. L. STREVEVS (B.P. 323,773, 6.10.28).—Carbonaceous materials are ground in oil, an oil-soluble colloid is added, and the resultant mixture is pumped at 300–350° and 40 atm. into tubular digesters where the temperature is raised to about 400°. The digested mixture is then pumped through pressure-cracking conduits at normal cracking temperatures into soaking tubes, and the cracked mixture released through pressure-reducing valves into an evaporator from which the lighter volatile fractions are removed. The residual mixture is filtered and the hot oil returned to the system to be mixed with solid fuel again; the solid matter left on the filters may be used as a dustless powdered fuel or briquetted. Catalysts may be added to the mixture at any suitable stage in order to modify the character of the reactions. H. S. GARLICK.

Heating of [petroleum] oil. J. PRIMROSE, ASSR. to FOSTER WHEELER CORP. (U.S.P. 1,738,263, 3.12.29. Appl., 2.7.23).—In the operation of an oil still, fuel is burnt in an enclosed space and air is supplied in substantial excess of the amount required for the combustion of the fuel, to lower the temperature and increase the volume of the combustion gases. Heat is absorbed by subjecting circulating oil to direct radiation, and by convection from the products of combustion and excess air as these pass into a second enclosed space, where the remaining heat is utilised in heating further circulating oil and preheating substantially all the excess air supplied to the first enclosed space. H. S. GARLICK.

Preparation of aqueous dispersions of petroleum products. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 302,346, 13.11.28. Holl., 15.12.27).—Natural emulsifying agents such as naphthenic acids and acid resins are contained in many mineral oils. When the residues from these oils are to be emulsified the emulsifying agent is retained in the oil by the addition of sufficient alkali before distillation. The residue is then dispersed directly in water or acidified to set free the organic acids and then emulsified in dilute soda solution, preferably by the aid of a colloid mill. The naphthenic acids recovered from some distillates may be used as emulsifying agents. T. A. SMITH.

Continuous treatment of hydrocarbons with liquid sulphur dioxide. ALLGEM. GES. F. CHEM. IND. M.B.H. (G.P. 457,580, 16.11.26).—The refined product as well as the extract is subjected to a continuous process of evaporation in stages under successively different pressures. A. B. MANNING.

Treatment of heavier hydrocarbons. C. A. JENSEN. From JENKINS PETROLEUM PROCESS CO. (B.P. 324,112, 18.10.28).—In a cracking plant in which oil and contact material are circulated through heating tubes, cracked distillate and residue are con-

tinuously removed. The exit for residue is kept clear from deposited carbon by occasionally pumping cracking stock through it into the plant. Alternative exits are provided so that one may be used for the removal of residue while the other is being washed clear.

T. A. SMITH.

Apparatus for decomposition of hydrocarbons by explosion. A. FECHTER and P. BURGER (B.P. 295,938, 20.8.28. Ger., 20.8.27).—Mixtures of hydrocarbons and air are exploded in a cylinder fitted with three mechanically operated valves. One valve serves for the admission of the mixture, another for the admission of hydrogen to sweep out the soot and other products of explosion, and the third, which acts as a spring-loaded safety valve, serves as an exit for the products of combustion. The valves are operated by cams and the apparatus is water-cooled. The conditions of pressure and gas mixture are easily controlled and the plant is very flexible.

T. A. SMITH.

Conversion of heavier [hydrocarbon] oils into lighter oils. R. J. BLACK, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,737,634, 3.12.29. Appl., 3.8.23).—Heavy hydrocarbons are cracked under pressure, the resultant vapours being condensed and the permanent gases separated at a lower pressure, recompressed, and injected into relatively cool hydrocarbon oil. The resulting oil containing absorbed and entrained gases is introduced into the main body of the oil maintained at cracking temperature.

H. S. GARLICK.

Gasification of heavy fuel oils. B. SAINT-FORT (F.P. 633,391, 13.8.26).—The oil is heated to a temperature below that at which its lightest constituents volatilise, *e.g.*, 70°, then atomised and heated in air, *e.g.*, in an ordinary carburettor, and finally further heated to a temperature below that at which decomposition or coking of the oil occurs.

A. B. MANNING.

Low-temperature cracking of residual and fuel oils. W. McI. CRANSTON (B.P. 324,443, 12.11.28).—Powdered bituminous coal and oil are heated in a retort fitted with a nickel-coated stirrer and to which steam may be admitted. The operation is carried out at 180–260° and under 200 lb./in.² pressure. The vapours formed are passed through fine nickel gauze to a condenser and the condensable portion is separated. When distillation ceases the contents of the retort, which are fluid, are discharged into externally heated vertical retorts and coked. The lower portions of the retorts are maintained at 450° and the upper at 550°. The vapours from the coking retorts are taken to a condenser and the condensable portions recovered. The coke residue is dry and suitable for use as a smokeless fuel.

T. A. SMITH.

Purification of hydrocarbon oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,805, 11.10.28).—Crude liquid hydrocarbons from any source are intimately mixed with chemical refining agents (solutions of alkali, sulphuric acid, or metaphosphoric acid) and subjected to distillation under a high vacuum, *e.g.*, 2 mm. Hg, while passing in a current of gaseous agents containing atomised liquids of lower b.p. range than those of the hydrocarbons under treatment.

H. S. GARLICK.

Refining of hydrocarbon oils. R. H. GARDNER and H. G. HODGE, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,740,584, 24.12.29. Appl., 13.7.27).—Light petroleum distillates, *e.g.*, benzine, gasoline, and cracked distillates, after treatment with sulphuric acid, removal of acid sludge, and freeing from traces of remaining acid, are heated in a still and the vapours led to a reflux tower where they come into countercurrent contact with fresh oil to be treated, containing a dissolved oil-soluble soap of a metal of the sulphide group, *e.g.*, copper resinate. The reflux condensate and unvaporised fresh oil from the reflux tower are conveyed to the still and the vapours from the reflux tower are led to a condenser.

H. S. GARLICK.

Refining of hydrocarbon oils. H. L. PELZER, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,740,625, 24.12.29. Appl., 11.6.27).—In the process of cracking hydrocarbon oils by distilling under pressure while circulating the charge of oil downwardly through a bed of finely-divided absorbent material, layers of coarser average particle size are maintained on either side of the main body of absorbent material.

H. S. GARLICK.

Treating hydrocarbon oils. H. WADE. From STANDARD OIL Co. (B.P. 323,784, 10.7.28).—Emulsifiable constituents of naphthenic petroleum oils or distillates that have not been treated with sulphuric acid may be removed by agitating with an alcohol which may be diluted with water and separating the alcoholic layer with naphthenic materials in solution therefrom. Prior to such washing, the distillate may be pretreated with aqueous alkali solution.

H. S. GARLICK.

Removal of cloud-forming materials from hydrocarbon oil. C. L. SUHR and W. S. ZEHRUNG, Assrs. to PENNZOIL Co. (U.S.P. 1,737,737, 3.12.29. Appl., 8.9.27).—From 3 to 10% of crystalline wax is added to heavy distillates of residuum oils, which are then chilled and the wax is separated by filtering.

H. S. GARLICK.

Apparatus for separation of hydrocarbons. E. R. COX and H. S. COLE, JUN., Assrs. to CALIFORNIA PETROLEUM CORP. (U.S.P. 1,738,870, 10.12.29. Appl., 1.12.27).—A plate or tray for an absorber, evaporator, or fractionating column for separating hydrocarbons or other liquids having fractions of different volatility comprises (a) a central dividing wall with an equalising passage therethrough; (b) two distributing walls with outwardly and downwardly sloping crests, situated on opposite sides and at opposite ends and forming diagonally disposed intake reservoirs; and (c) two liquid-level maintaining walls, each on opposite sides and at opposite ends, forming diagonally disposed discharge reservoirs with absorption reservoirs intermediate the distributing and liquid-level maintaining walls, with perforate bottom sections.

H. S. GARLICK.

Elimination of impurities in insulating oils. C. J. RODMAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,739,092, 10.12.29. Appl., 21.10.24).—In oil-immersed electrical devices, *e.g.*, circuit interrupters and transformers, a dehydrated inert gas is bubbled through the insulating oil to preserve its dielectric qualities by driving off moisture and volatile

solvents, and by coagulating and precipitating sludge and decomposition products. II. S. GARLICK.

Production of substances such as benzene, fuel oil, lubricating oil, and the like from solid fuels. A. POTT and H. BROCHE (B.P. 293,808, 12.7.28. Ger., 12.7.27).—Coal, lignite, peat, etc. are treated in one or more stages with solvents such as tetralin or tar fractions at temperatures up to 400°. Under the conditions a large proportion of the coal (up to 70%) goes into solution. The extract after removal of solvent is more readily hydrogenated than the original coal.

T. A. SMITH.

Manufacture of products of a high stage of oxidation from hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,189, 14.12.28).—Paraffin hydrocarbons, waxes, etc. are treated with gas mixtures containing oxygen until only a part of the initial material is oxidised and the reaction is stopped before discoloration occurs. The oxidation products are continuously removed while an equal quantity of the initial material is introduced, and the oxidation of the non-oxidised portion and fresh material is then continued.

H. S. GARLICK.

Purification of paraffin wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,083, 15.8.28).—Crude paraffin wax containing considerable quantities of oil is treated with a solvent which dissolves the oil but not the wax, *e.g.*, acetone, benzene, and the wax is separated from the mixture by means of a continuous suction filter, and preferably washed with a solvent of the same character.

H. S. GARLICK.

Carburetion of methyl alcohol [for fuel]. E. P. DUMANOIS (F.P. 633,443, 27.8.26).—Anhydrous methyl alcohol is mixed with ethyl alcohol and hydrocarbon fuels, *e.g.*, mixtures of benzene and naphthalene, in order to produce a fuel of higher calorific value.

A. B. MANNING.

Soluble oil containing ether derivatives of polyhydroxy-alcohols. D. R. MERRILL, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,739,686, 17.12.29. Appl., 4.1.28).—A soluble oil consists of approx. 5 pts. of a neutral oil, preferably a mineral oil, 1 pt. of water-soluble soap, preferably an alkali naphthenate the water content of which has been reduced to give a final concentration in the mixture of less than 8%, and 0.2 pt. of a blending agent consisting of a hydroxy-ether at least partially soluble in both the oil and water, *e.g.*, the monoethyl ether of ethylene glycol.

H. S. GARLICK.

Motor fuel. DEUTS. HYDRIERWERKE A.-G. (G.P. 457,581, 20.1.25).—The pre-ignition of fuels which tend to detonate is prevented by the addition of at least 5% of hydrogenated phenols.

A. B. MANNING.

Production of motor fuels. S. SCHANZER (WERNICKE & BEYER) (B.P. 301,076, 9.11.28. Ger., 25.11.27).—Quinol is dissolved in the usual mixture of alcohol and mineral oil.

H. ROYAL-DAWSON.

Production of fuel for industrial and domestic purposes. H. DEBAUCHE (U.S.P. 1,746,767, 11.2.30. Appl., 6.7.27. U.K., 29.11.26).—See B.P. 270,976; B., 1927, 548.

Combustion [with the aid of catalysts]. J. W. GRUETTER (U.S.P. 1,747,771, 18.2.30. Appl., 23.8.26).—See B.P. 276,300; B., 1928, 778.

Production of paraffin hydrocarbons with more than one carbon atom. F. FISCHER and H. TROPSCH (U.S.P. 1,746,464, 11.2.30. Appl., 1.4.26. Ger., 21.7.25).—See B.P. 255,818; B., 1927, 695.

Conversion or purification of hydrocarbon fluids. H. ROSTIN (U.S.P. 1,732,111, 15.10.29. Appl., 29.2.28. Ger., 1.3.27).—See B.P. 286,255; B., 1930, 179.

Treatment of hydrocarbons with electric arcs. P. GRELIN and O. EISENHUT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,746,934, 11.2.30. Appl., 24.1.27. Ger., 29.1.26).—See B.P. 294,494; B., 1928, 738.

Utilisation of residue [mineral] oils containing water. A. H. MALLERY, Assr. to MALLERY PROCESS CORP. (U.S.P. 1,747,375, 18.2.30. Appl., 5.4.26).—See B.P. 268,814; B., 1928, 884.

Burners for pulverulent fuel. S. A. WARD (B.P. 325,057, 22.12.28).

Heat-treatment etc. of carbonaceous matter (B.P. 324,692). **Heat-exchanging apparatus** (B.P. 324,940).—See I. **Treatment of ammonium thiocyanate** (B.P. 309,565). **Lead acetate from pyro-ligneous acid** (F.P. 632,866). **Hydrogen from methane** (B.P. 294,580).—See VII.

III.—ORGANIC INTERMEDIATES.

Retene and its derivatives. ANON. (Bull. Inst. Pin, 1929, Special No., 108—110; Chem. Zentr., 1929, ii, 1794).—The possible use of retene as a primary material for the preparation of dyes is considered. By heating colophony or resin oil with sulphur at 210—220°, heating the product in a vacuum, and extracting the distillate with alcohol, a 41% yield of retene (m.p. 98—99°; picrate, m.p. 126—127°; retene-quinone, m.p. 196—197° [*p*-nitrophenylhydrazones, m.p. 219°]; dinitroretenequinone, m.p. 229—230°) was obtained.

A. A. ELDRIDGE.

Lead triethylbutyl compounds. GILMAN and others.—See II. **Purification of absolute alcohol.** FRITZWEILER and DIETRICH. **Influence of salts on distillation of alcoholic mixtures.** MARILLER. **Alcoholometry.** MARILLER.—See XVIII.

PATENTS.

Manufacture of olefines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 322,524, 7.9.28).—Neutral or acid alkyl sulphates are added to salts of di- or tri-basic acids (tricalcium phosphate, sodium chloride or sulphate, disodium hydrogen phosphate) at 250—300° at ordinary or reduced pressure. Diethyl or ethyl hydrogen sulphate gives a 90% yield of ethylene, propyl hydrogen sulphate 99% of pure propylene.

C. HOLLINS.

Manufacture of polymerisation products of γ -butylene [isobutylene] and of trimethylethylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,102, 1.12.28).—*iso*Butylene and *iso*amylene (trimethylethylene) are absorbed from gases in 50—60% sulphuric acid and the solution is heated for $\frac{1}{2}$ hr. at 100°.

Dimerides, with 20% of other polymerides, separate out, and the acid, cooled by heat-exchange, is used to absorb fresh gases. C. HOLLINS.

Concentration of lower aliphatic acids. BRIT. CELANESE, LTD. (B.P. 301,734, 1.12.28. U.S., 3.12.27).—Methylene dichloride, or its mixture with ether, is used as solvent for extraction of acetic acid (etc.) from dilute solutions. C. HOLLINS.

Production of esters. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 295,275 and Addn. B.P. 320,113, 5. and 6.7.28. Ger., [A] 8.8.27).—A continuous process is described for the esterification of dilute acetic acid (etc.) with, for example, methyl alcohol, (A) in the presence of acid catalysts producing p_H 1.5—3, (B) in the absence of catalysts. In both cases the mixture of alcohol and dilute acid is heated at 8—10 atm. pressure and 150—250°, and the ester formed is continuously removed from the reaction mixture and rectified in an upper column. C. HOLLINS.

Manufacture of monoalkyl ethers of ethylene glycol. IMPERIAL CHEM. INDUSTRIES, LTD., W. GIBSON, and J. B. PAYMAN (B.P. 322,037, 27.9.28).—The reaction between ethylene oxide and alcohol proceeds more smoothly in presence of normal sulphates of zinc, nickel, or chromium. C. HOLLINS.

Production of crotonaldehyde. HOLZVERKOHLUNGS-IND. A.-G., and J. SEIB (B.P. 320,210, 13.9.28).—In the conversion of aldol into crotonaldehyde, acetates and other organic salts in the aldol are first neutralised with hydrochloric or sulphuric acid. Higher yields of crotonaldehyde are obtained, and acetaldehyde containing methyl formate (and acetic acid) may be used without purification for adolisation. C. HOLLINS.

Manufacture of sulphonic acids [wetting-out, cleansing, and emulsifying agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,005, 1.9.28).—In the process of B.P. 263,873 (B., 1928, 665) the olefines are replaced by crude cracked gases. C. HOLLINS.

Manufacture of wetting agents, emulsifying agents, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,429, 27.7.28).—Oxidised paraffin wax is neutralised with aqueous alkali and emulsified with the addition of a water-soluble salt (sodium chloride or sulphate, magnesium chloride); if an alkaline-reacting salt (sodium silicate) be used, the preliminary neutralisation may be omitted. The products have strong wetting action. C. HOLLINS.

Manufacture of divinyl and homologues thereof. I. G. FARBENIND. A.-G. (B.P. 301,898, 14.11.28. Ger., 9.12.27. Addn. to B.P. 291,748; B., 1929, 886).—Other acid salts, e.g., potassium dihydrogen phosphate, aluminium sulphate, are used in place of the dehydrated alums of the prior patent. C. HOLLINS.

Production of cyanamide. G. BARSKY and P. W. GRIFFITH, ASSTS. to AMER. CYANAMID Co. (U.S.P. 1,741,674, 31.12.29. Appl., 8.2.24).—Decomposition of cyanamide during its preparation, e.g., from metal cyanamides, and during the concentration of solutions containing it, is minimised by maintaining the solutions at p_H 2—7. L. A. COLES.

Production of thiocarbamide. E. MERCK, Assees. of H. MAYEN and O. WOLFFES (G.P. 452,025, 14.11.24).—An aqueous cyanamide solution, prepared, e.g., by extracting calcium cyanamide with water at 15° and precipitating the calcium with carbon dioxide, is saturated below 60° (preferably at 15°) with hydrogen sulphide in the presence of water-soluble bases, preferably ammonia, and the solution is concentrated to effect crystallisation of the thiocarbamide. L. A. COLES.

Preparation of finely-divided nitroguanidine and like bodies. NOBEL INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 322,427, 1.6.28).—Nitroguanidine in a form suitable for incorporation in nitrocellulose powders is obtained by discharging into a large amount of ice a solution of 6 pts. of nitroguanidine in 10 pts. of water at 140—150°. C. HOLLINS.

Manufacture of *N*-methyl-*p*-aminophenol. CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER A.-G. (B.P. 306,939, 6.2.29. Ger., 29.2.28).—Anhydroformaldehyde-*p*-aminophenol or a mixture of *p*-aminophenol and formaldehyde is reduced with aluminium and mercury in presence of alkali. C. HOLLINS.

Manufacture of *N*-[hydr]oxyethyl derivatives of 2-amino-1-oxybenzene [*o*-aminophenol]. I. G. FARBENIND. A.-G. (B.P. 296,309, 27.8.28. Ger., 26.8.27. Addn. to B.P. 292,950; B., 1929, 935).—*o*-Aminophenol is treated in presence of a solvent or diluent (water) with ethylene oxide; if desired, a catalyst (chalk, sulphuric acid) may be added. *N*-β-Hydroxyethyl-*o*-aminophenol, m.p. 80—81°, is obtained. C. HOLLINS.

Manufacture of water-soluble condensation products and technical applications thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 320,056, 28.6.28).—A sulphonated phenol or naphthol is condensed with a halogenated aralkyl halide, preferably in presence of sulphuric acid, to give a soluble product which acts as a reserve on wool or silk in mixed cotton fabrics or may be used as a tanning agent. Examples are: phenolsulphonic acid with *o*-chlorobenzyl chloride or trichlorobenzyl chloride (b.p. 270—273°); β-naphthol-sulphonic acid with technical (*o*- and *p*-)chlorobenzyl chloride. C. HOLLINS.

Manufacture of azine derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,209, 27.8.28).—Azinesulphonic acids are converted into hydroxyazines by heating with 5—10% sodium hydroxide, preferably under pressure at 245—250°. The hydrolysis may be combined with the synthesis of the azinesulphonic acids; e.g., β-naphthylamine-8-sulphonic acid is oxidised with alkaline hypochlorite and then heated under pressure without isolation to give dihydroxydinaphthazine. C. HOLLINS.

Manufacture of 2-[4-alkylbenzanthrone]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,253, 7.9.28).—The 4-acetonylbenzanthrone obtained according to B.P. 319,593 (B., 1930, 136) from benzanthrone and ketones of the type $R \cdot CH_2 \cdot CO \cdot R'$, are converted into 4-alkylbenzanthrone by hydrolysis with alkali, the acyl group $R'CO$ being removed. Examples are: 4-methylbenzanthrone, m.p. 198—200°, from the

condensation product of benzanthrone with acetone or acetophenone; 4-*n*-propylbenzanthrone, m.p. 130—131°, from the condensation product of benzanthrone with di-*n*-propyl ketone. C. HOLLINS.

Manufacture of anthraquinone- and benzanthrone-carboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 321,916, 16.7.28).—Methyl-anthraquinones or -benzanthrone are heated at 150—200° in aqueous alkaline suspension with oxygen or air at 30—50 atm. The preparation of anthraquinone-2-carboxylic acid, m.p. 292°, 1-aminoanthraquinone-2-carboxylic acid, m.p. 284°, and benzanthrone-4-carboxylic acid, m.p. 295°, is described. [Stat. ref.]

C. HOLLINS.

Preparation of aromatic diacidyl compounds. O. WULFF, R. SEDLMAYR, and W. ECKERT, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,747,535, 18.2.30. Appl., 18.10.27. Ger., 23.10.26).—See B.P. 279,506; B., 1928, 440.

Production of menthol. W. SCHOELLER, H. JORDAN, and R. CLERC, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,737,272, 26.11.29. Appl., 29.6.28. Ger., 15.7.27).—See B.P. 293,874; B., 1930, 218.

Distillation of solid substances (B.P. 324,840).—See I. Utilisation of water-gas (B.P. 317,460).—See II. Anthraquinone dye intermediates (B.P. 322,184).—See IV. Tanning agents (G.P. 456,931).—See XV.

IV.—DYESTUFFS.

PATENTS.

Leuco-compounds of vat dyes. E. I. DU PONT DE NEMOURS & Co. (B.P. 295,716, 8.8.28. U.S., 19.8.27).—The sodium salts of sulphuric esters of leuco-vat dyes (Indigosols, Soledon colours) are converted into hydroxyalkylamine salts by acidifying with acetic acid, filtering off the acid ester, and treating it, e.g., with "triethanolamine." The hydroxyalkylamine may be added to the soluble leuco-ester printing-paste. C. HOLLINS.

Manufacture and use of [vat] colouring matters [from phthalic anhydride, ammonia or amines, and a metal]. A. G. DANDRIDGE, H. A. E. DRESCHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 322,169, 16.5.28 and 24.1.29).—Phthalic anhydride and iron filings are heated with ammonia gas at 140—240°; on removal of phthalimide with alkali a blue vat dye is obtained, which is also suitable for dyeing acetate silk. The iron may be replaced by nickel or copper, or by sulphides, chlorides, or sulphates of the metals, and the ammonia by primary amines. Phthalimide, phthalamide, and phthalamic acid may be used. The products may be sulphonated to give wool dyes. C. HOLLINS.

Manufacture of a vat dye [of the perylene series]. F. BENSA (B.P. 305,136, 28.1.29. Austr., 31.1.28).—Di-β-naphthol is chlorinated in boiling acetic acid, and the product, recrystallised from aniline, is heated at 140—150° with concentrated sulphuric acid to give a heliotrope vat dye. C. HOLLINS.

Production of vat dyes of the anthraquinone series and intermediate products therefor. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,184,

23.7.28).—Imide chlorides of 2:6-diaroyldiaminoanthraquinones are treated with sulphur or alkaline sulphides. Under mild conditions there is first formed the intermediate imide thiol $C_{14}H_6O_2(N:CR \cdot SH)_2$, which on dehydrogenation, e.g., with concentrated sulphuric acid or alkaline ferricyanide, gives the final product, an anthraquinonebisthiazole. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone [pyrazolanthrone] series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 322,008, 3.9.28).—A pyrazolanthrone, carrying in position 2 ($N:N=1:2$) an alkyl group and in position 3 a halogen, sulphonic, or thiol group, is vatted and re-oxidised to give a red vat dye, identical with the product of alkaline fusion of pyrazolanthrone followed by alkylation; the group in position 3 is eliminated. C. HOLLINS.

Manufacture of vat dyes of the *N*-dihydro-1:2:2':1'-anthraquinoneazine series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,281, 10.10.28).—The formaldehyde-indanthrone products of B.P. 20,527 of 1904 (B., 1905, 840) are oxidised, e.g., with chromic acid, caustic alkali, or sulphuric acid and manganese dioxide; or the condensation and oxidation are performed in one operation. Greenish-blue vat dyes result. C. HOLLINS.

Manufacture of blue [azine] dyes of the anthracene series which contain halogens. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,277, 5.10.28).—Indanthrone is halogenated by treatment with hydrogen halides (or sodium halides) in 60% sulphuric acid suspension. The pure indanthrone obtained by oxidation of the crude product with sulphuric acid and manganese dioxide may be treated with halogen acid without isolation. C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 302,599, 17.12.28. Ger., 17.12.27).—Light-fast ice-colours and pigments are obtained by coupling a 2:3-hydroxynaphthoic arylamide, especially an *o*- or *p*-anisidine, with a dihalogenated *o*-toluidine having a halogen atom in position 4, one or both of the halogens being bromine, e.g., 4-chloro-5-bromo-*o*-toluidine, m.p. 102—103°, or 4:5-dibromo-*o*-toluidine. C. HOLLINS.

Production of conversion products of azo dyes [for dyeing leather]. J. R. GEIGY SOC. ANON. (J. R. GEIGY A.-G.) (B.P. 306,447, 30.11.28. Ger., 20.2.28).—Mono- or dis-azo couplings of resorcinol or its derivatives, or of 2-amino-*p*-cresol, α-naphthol, or 1:5-dihydroxynaphthalene, are treated with air in dilute ammonia at 70—80° to increase their affinity for leather; the products no longer dye silk and wool. Thirteen examples are tabulated. C. HOLLINS.

Manufacture of azo dyes and chromed derivatives thereof. COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ETABL. KUHLMANN (B.P. 304,298, 22.5.28. Fr., 19.1.28).—An *o*-amino-phenol or -naphthol is diazotised and coupled with a pyrazolone derived from an *N*-arylsulphonylarylenediamine. The products may be prechromed. Pyrazolones containing the following 1-aryl groups are mentioned: 3-*p*-toluenesulphonamidophenyl, 4-*p*-toluenesulphonamido-*o*-tolyl, and *m*-benzenesulphonamidophenyl. C. HOLLINS.

Manufacture of [prechromed azo] dyes. Soc. CHEM. IND. IN BASLE (B.P. 295,944, 20.8.28. Switz., 19.8.27. Addn. to B.P. 289,094; B., 1930, 277).—In the process of the prior patent an acyl-J-acid is used as coupling component.

C. HOLLINS.

Manufacture and application of dyes containing metal. Soc. CHEM. IND. IN BASLE (B.P. 298,494, 8.10.28. Switz., 8.10.27).—Cyanuric chloride is used to link up nuclei of prechromed or precoppered azo dyes with similar or different nuclei. Amongst the 34 examples are products made by condensing cyanuric chloride with: precoppered *o*-aminophenol-*p*-sulphonic acid \rightarrow J-acid (blue-red); prechromed 1:2:4-aminonaphtholsulphonic acid \rightarrow α -naphthol and aniline (blue-grey); precoppered *o*-aminophenol-*p*-sulphonic acid \rightarrow γ -acid and 4-chloro-*m*-phenylenediamine (blue-red); 2:4:8-naphthylaminedisulphonic acid \rightarrow oresidine, precoppered *o*-aminophenol-*p*-sulphonic acid \rightarrow J-acid and aniline (brown); prechromed 1:2:4-aminonaphtholsulphonic acid \rightarrow α -naphthol, *p*-aminoacetanilide, and aniline, hydrolysed and phosgenated (blue); 2:4:8-naphthylaminedisulphonic acid \rightarrow cresidine, nitrated diazo compound from 1:2:4-aminonaphtholsulphonic acid \rightarrow 5:8-dichloro- α -naphthol, reduced, and aniline (green). The products dye wool, cotton, and silk.

C. HOLLINS.

Manufacture of dyes [chromium compounds of triarylmethane and azo dyes]. Soc. CHEM. IND. IN BASLE (B.P. 296,310, 27.8.28. Switz., 27.8.27).—A prechromed triarylmethane dye and a prechromed azo dye are made to interact in the dye-bath or on the fibre. The dyeings are unexpectedly fast to light. Examples are: 1:2:4-aminonaphtholsulphonic acid \rightarrow α -naphthol and Chrome Azurol S or Erichrome Azurol B.

C. HOLLINS.

Manufacture of sulphur dyes. I. G. FARBERIND. A.-G. (B.P. 297,123, 14.9.28. Ger., 15.9.27. Addn. to B.P. 286,005 and 285,382; B., 1928, 740, 672).—The indophenols or leucoindophenols of the prior patents are heated with polysulphide. The product from hexahydrocarbazole gives a bluish-violet, that from octahydro- α -naphthaquinoline a yellowish-green, and that from octahydroacridine a deep green.

C. HOLLINS.

Manufacture of hair dyes. A. JINNO (B.P. 322,972, 8.3.29).—*p*-Phenylenediamine or other diamine is mixed with tannic acid (4 pts.) and starch, boiled with water, and pasted with hydrogen peroxide. The tannate of the base does not injure the skin and is oxidised slowly by the hydrogen peroxide. The starch facilitates removal of the dye from the skin.

C. HOLLINS.

Manufacture of [yellow] vat dyes. A. LÜTTRINGHAUS, P. NAWIASKY, and A. KRAUSE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,747,520—1, 18.2.30. Appl., [A] 3.10.27, [B] 27.10.28. Ger., [A, B] 25.10.26).—See B.P. 296,386; B., 1928, 848.

Yellow monoazo dye. H. WAGNER and E. FISCHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,179, 1.10.29. Appl., 1.5.28. Ger., 10.5.27).—See B.P. 290,253; B., 1930, 95.

[Manufacture of] azo dyes. H. CLINGESTEIN, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,747,539,

18.2.30. Appl., 22.10.28. Ger., 22.9.26).—See B.P. 319,407; B., 1930, 95.

Preparation of water-soluble [chrome] dyes of the triarylmethane series. B. FRANCKE and H. MOEHRKE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,747,541, 18.2.30. Appl., 31.12.26. Ger., 2.1.26).—See B.P. 263,879; B., 1927, 742.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Heat power economy of a paper and straw-cellulose factory. V. S. INDENBAUM and N. M. OGNEV (Izvestia Teplotekh. Inst. Moscow, 1929, No. 8, 31—47).—Details are given of the amounts of steam consumed in the various sections of a factory comprising a paper department with three machines turning out 30—35 tons of paper, a straw-boiling department of six cookers producing about 12 tons of air-dry cellulose, and a rag-boiling plant of six boilers with the maximum total output about 6 tons of rags, all these quantities being per 24 hrs. The wood pulp used was partly made on the spot and partly imported.

T. II. POPE.

Regenerative soda-oven at straw-cellulose factory. V. S. INDENBAUM, G. N. BEZRADECKI, and N. V. SCHISCHAKOV (Izvestia Teplotekh. Inst. Moscow, 1929, No. 8, 48—54).—The heat balance of the regeneration of spent soda from a straw-cellulose factory is worked out in detail from experimental results.

T. H. POPE.

Regeneration of caustic sludge in sulphate- and soda-cellulose works. R. LORENZ (Papier-Fabr., 1930, 28, 97—103).—Methods of using the waste sludge from cellulose works are described and discussed. Since it consists chiefly of calcium carbonate and oxide it is very suitable for the preparation of chalk manure for acid soils. The carbonate may be isolated from it in a very fine state of division as whiting, which may be used as filling material for thin printing- and cigarette-papers, as weighting material for rubber, or in the manufacture of glass and powders. These uses depend on the cost of preparing the material, which may be high, and on the demand, which may be small, so that alternatively the sludge may be ignited, recausticised, and used again in the cellulose works instead of fresh lime. Plant for the preparation of whiting and for chalk-burning, and drum filters for treating the sludge are illustrated and described.

B. P. RIDGE.

Manufacture of sulphite-wood cellulose and waste liquors therefrom. C. G. SCHWALBE (Chem.-Ztg., 1930, 54, 4—5, 21—22).—A general account of the methods of manufacturing wood celluloses and the utilisation of the sulphite waste liquors as fuel material.

S. K. TWEEDY.

Composition of copper alkali cellulose. V. I. SHARKOV (J. Appl. Chem., Russia, 1929, 2, 437—443).—Dry cotton was soaked, with occasional shaking, in various mixtures of potassium hydroxide and ammoniacal cupric hydroxide solutions for 1.5 hrs. The copper content of the cellulose increases with increase in concentration of copper in the solution and depends on the alkali hydroxide concentration. Formation of the compound $\text{Na}_2(\text{C}_{12}\text{H}_{16}\text{O}_{10}\text{Cu})$ was not observed. Part of the copper is probably adsorbed as cuprammonium

hydroxide. Copper alkali cellulose forms with carbon disulphide a copper xanthate, sparingly soluble in ammonia and insoluble in water or in solutions of sodium or potassium hydroxide. In ammonia there is obtained a thick red liquid which quickly forms a gel showing distinct syneresis.

CHEMICAL ABSTRACTS.

Artificial silk containing nitrogen. H. STADLINGER (Chem.-Ztg., 1930, 54, 5—6).—The patent literature pertaining to the production of artificial silk containing nitrogen is reviewed. Bergmann's process (B., 1928, 239) is considered advantageous.

S. K. TWEEDY.

Viscose. TANEMURA and others.—See VII. **Bakers' yeast from sulphite liquor.** HÄGGLUND.—See XVIII. **Writing on documents etc.** MEZGER.—See XXI.

PATENTS.

Preparation of esparto fibre etc. A. RUIZ (F.P. 635,747, 10.6.27).—The separated and beaten esparto fibre is carded and treated with a solution of sodium hydroxide, either cold or warm (4—6 kg. NaOH per 100 kg. of fibre).

F. R. ENNOS.

Degumming of ramie and like fibres. W. GATER (B.P. 324,121, 20.10.28).—The fibre is soaked or fermented for 5—10 days in soft water at 13—24°, washed, immersed in and afterwards boiled in soap solution, and dried.

F. R. ENNOS.

Saturation of fibrous articles. H. PARKER, Assr. to BROWN Co. (U.S.P. 1,738,794, 10.12.29. Appl., 6.3.25).—The articles such as tubes or pipes for underground conduits are immersed in the heated saturant, e.g., pitch, and, after subjecting the whole alternately to higher and lower pressures and permitting the escape of the gases from the pores of the material when pressure is reduced, the saturant is withdrawn and the articles are allowed to cool.

F. R. ENNOS.

Treatment of textile materials containing carbonisable fibres, and products obtained thereby. BRIT. CELANESE, LTD. (B.P. 310,936, 3.5.29. U.S., 3.5.28. Addn. to B.P. 274,074; B., 1928, 811).—By means of the carbonising process described in the prior patent a fabric presenting lace-like, gauze-like, or other open-work effects may be produced from fabrics composed of mixtures of yarns of organic derivatives of cellulose and of cotton, flax, or reconstituted cellulose; in the same way, spun yarns consisting of staple fibre formed of organic derivatives of cellulose are made from mixed spun yarns of the staple fibre with cotton, flax, or reconstituted cellulose.

F. R. ENNOS.

Manufacture of artificial leather and the like [from fibrous materials]. LONZA-WERKE ELEKTROCHEM. FABR. G.M.B.H. (B.P. 299,023, 19.10.28. Ger., 19.10.27).—Vegetable, animal, mineral, or artificial fibres are thoroughly mixed in a kneading machine with a viscous solution of cellulose or one or more cellulose derivatives (cuprammonium cellulose solution), and the homogenised mass is passed through slotted apertures into one or more coagulating baths. After washing, the product is impregnated with softening agents, treated with a hygroscopic agent, and dried.

F. R. ENNOS.

Manufacture of iridescent products. M. C. MEYER, Assr. to J. H. MEYER BROS., INC. (U.S.P. 1,740,615, 24.12.29. Appl., 3.10.25).—A textile material is coated with a substance having an oil base, and, after drying and polishing the surface produced by passing through heat rollers under pressure, a basic halogen salt of bismuth is applied thereto and covered with a film of lacquer the solvent of which is immiscible with the coating; the surface of the applied film is finally rubbed with the basic halogen salt.

F. R. ENNOS.

Manufacture of an indurated porous object. J. J. KESSLER (U.S.P. 1,739,597, 17.12.29. Appl., 24.9.28).—Objects such as wood, paper, fibrous materials, etc. are saturated with a drying and polymerising oil prepared by the action of formaldehyde on tar fractions of b.p. 150—250° (cf. U.S.P. 1,616,321; B., 1927, 304), and finally dried.

F. R. ENNOS.

Production of a liquid- and moisture-proof transparent and flexible material. WOLFF & Co. KOMM.-GES. AUF AKT., R. WEINGAND, and F. W. SPIECKER (B.P. 318,166, 22.2.29. Ger., 29.8.28. Addn. to B.P. 309,017; B., 1930, 11).—A modification of the prior patent in which nitrocelluloses having a nitrogen content above 12% are employed as the cellulose ester in the lacquer, whilst the solvent for the wax mixture consists of a liquid which is a solvent for the nitrocelluloses, or of a mixture of liquids at least one constituent of which will dissolve the ester.

F. R. ENNOS.

Production of spinning fibres. W. MISCHKE (B.P. 312,321, 23.5.29. Ger., 24.5.28).—A staple fibre, capable of being spun on machines suitable for relatively short staple cotton, is produced by cutting artificial silk filaments of 2—4 deniers into lengths of 20—40 cm., and passing them in a very moist and preferably loosened state to a drying process.

F. R. ENNOS.

Manufacture of textile fibres. ALSA SOC. ANON. (B.P. 299,710, 27.10.28. Ger., 29.10.27).—In order to blow out and increase the gas content of artificial, hollow textile fibres, they are immersed, before being dried, in a liquid in which gases are then caused to develop by chemical or physical means. Examples are ice-cold water saturated with air or carbon dioxide.

F. R. ENNOS.

Apparatus for dry-spinning artificial textile fibres, with recovery of the volatile solvents. SOIE DE CHÂTILLON (B.P. 309,158, 17.9.28. Ital., 7.4.28).—The heated spinning cell has a cooling device consisting of a pipe which is adjacent and substantially parallel to the path of travel of the filaments and through which flows a cooling liquid; the condensed solvent vapours are collected in a trap at the bottom of the cell.

F. R. ENNOS.

Apparatus for heating air in dry-spinning of artificial threads. ACETA GES.M.B.H. (B.P. 308,350, 13.3.29. Ger., 22.3.28).—The spinning cell is heated by a fluid or by a resistance coil in an external casing surrounded, wholly or partially, by a second externally insulated, concentric casing, which heats the air passing through it on its way to the spinning cell.

F. R. ENNOS.

Manufacture of artificial filaments etc. H. DREYFUS (B.P. 324,061, 9.7.28).—A solution of cellulose

acetate is extruded into a closed and heated spinning cell which is maintained under reduced pressure, and the solidified filament is led outside the cell for winding and twisting.

F. R. ENNOS.

Manufacture of artificial threads. I. G. FARBENIND. A.-G. (B.P. 297,118, 11.9.28. Ger., 15.9.27).—A permeable spool which is elastic or variable in diameter is inserted in the cake of spun thread in the centrifugal box, wherein without rotation the subsequent processes of purification, treatment, and drying are carried out without risk of tangling the thread.

F. R. ENNOS.

Spinning pots for artificial silk. R. ATTWATER (B.P. 324,214, 9.1.29).—The pots are moulded from tubes and discs formed of layers of fabric impregnated with a synthetic resin.

F. R. ENNOS.

Treatment of materials made of or containing cellulose esters or ethers. H. DREYFUS (B.P. 324,662, 29.9.28).—The filaments of cellulose derivatives are treated while under tension with concentrated mineral acids for a short time (0.5 sec. or longer, depending on the nature, concentration, and temperature of the acid), whereby the tensile strength is increased.

F. R. ENNOS.

Manufacture or treatment of threads or filaments of organic cellulose derivatives. H. DREYFUS (B.P. 323,790, 27.7.28).—After reaching a set elastic condition the filaments, continuously with their production, are stretched in the presence of softening or swelling agents to yield a permanent extension of 100% or more.

F. R. ENNOS.

Manufacture of artificial silk. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 322,730, 9.8.28).—Artificial silk which is fast to boiling is produced by dry-spinning a water-insoluble cellulose ether from which substantially all the constituents of low viscosity which swell in water have previously been removed (e.g., in accordance with the process described in B.P. 321,651 (B., 1930, 99).

F. R. ENNOS.

Artificial silk. SONDERMANN & Co. (MECHANISCHE WEBEREI SESSMAR SONDERMANN & Co., G.M.B.H.) (B.P. 314,787 and 318,211, 28.9.28. Ger., [A] 2.7.28, [B] 31.8.28. Addns. to B.P. 297,778; B., 1930, 238).—Modifications of the prior process are described in which the sizing treatment is applied (A) in one operation with the warping, copping, winding, etc., and (B) to the filaments derived directly from the wound form in which they are obtained after spinning, and without previous rewinding.

F. R. ENNOS.

Manufacture of filaments, threads, bands, etc. from viscose. COURTAULDS, LTD., H. J. HEGAN, and J. H. TAYLOR (B.P. 323,830, 22.10.28).—A product of reduced lustre is produced by mixing the viscose with a high-boiling liquid petroleum or petroleum jelly and a blown animal or vegetable oil or blown oleic acid.

F. R. ENNOS.

Treatment of artificial silk. G. B. ELLIS. From SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 323,846, 30.10.28).—Filaments of cellulose esters or ethers are rendered plastic by being heated to a suitable temperature and stretched to a considerably greater extent than the breaking elongation in the dry state

(if desired, more than 100%), whereby threads of extremely fine denier with improved mechanical properties are produced.

F. R. ENNOS.

Treatment of films, sheets, bands, filaments, or other articles or products composed of regenerated cellulose, cellulose hydrate, or cellulose ethers which are not resistant to water. WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 324,677, 25.9.28. Ger., 12.10.27).—A water-immiscible plasticising agent suitable for use with cellulose ester plastics, e.g., amyl phthalate, is incorporated with the regenerated cellulose etc. with the aid of a water-miscible swelling agent (an organic acid, alcohol, etc.). (Cf. B.P. 298,605; B., 1930, 237.)

F. R. ENNOS.

Diminishing swelling in cellulose and cellulose products. I. G. FARBENIND. A.-G. (B.P. 297,463, 18.9.28. Ger., 23.9.27).—Paper or vulcanised fibre, but not textile materials, are saturated with an aqueous neutral or weakly alkaline solution of an artificial resin or its components (urea or thiourea and formaldehyde), and the whole is heated, in some cases with an intermediate treatment with formaldehyde, to render the condensation product insoluble.

F. R. ENNOS.

Solution for repairing [stopping the formation of "ladders" in] silk and artificial silk goods. E. A. BARRETT and J. G. FLETCHER. From L. BACHMANN (B.P. 324,641, 29.10.28).—The solution consists of 90% of glacial acetic acid or acetone, 5% of dinitrocellulose, and 5% of alcohol denatured with toluene.

F. R. ENNOS.

Manufacture of cellulose butyric esters. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 323,869, 20.11.28 and 23.7.29).—After pretreatment with butyric acid, cellulose is esterified with butyric anhydride in the presence of a sulphonic acid of an aliphatic hydrocarbon (methane) and other suitable catalysts if desired, preferably with addition of a diluent (benzene) which is a solvent for the ester formed.

F. R. ENNOS.

Production of artificial leather [from cellulose esters or ethers]. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS, Assees. of W. PLINATUS (B.P. 301,759, 3.12.28. Ger., 3.12.27).—The cellulose derivatives are kneaded, preferably *in vacuo*, with softening and gelatinising agents (multivalent alcohols, vegetable or animal oils, sulphonated if desired) to a semi-solid state, and rolled out to a thin layer which is placed on a base of fabric, paper, etc.; the whole is then inserted between two plates capable of being heated or cooled, which are drawn between two pressure rollers so that a sliding pressure is exerted on the material.

F. R. ENNOS.

Plastic [cellulose-cement] compositions. L. G. HARRIS (B.P. 324,652, 26.10.28).—Fibrous cellulose material (waste paper or rags) is disintegrated, swelled with water, and partially hydrolysed with soda or acid. After addition of sodium silicate or magnesium chloride, the material is mixed with cement in the proportion of 1 pt. of cement to 3 pts. of dry cellulose, moulded, and allowed to set.

F. R. ENNOS.

Mouldable material [from kelp]. (SIR) G. C. MARKS. From PLASTIC, INC. (B.P. 324,638, 26.10.28).

—Kelp is macerated with a solution of copper sulphate and then ground with a fibrous substance (asbestos, or jute flocks with or without a refractory material) to which ammonia is subsequently added; the plastic mass formed dries at ordinary temperatures to a tough material.

F. R. ENNOS.

Chemical pulping process. B. JOHNSON, Assr. to HAMMERMILL PAPER Co. (U.S.P. 1,737,590, 3.12.29. Appl., 16.2.27).—Fibrous vegetable material is cooked in a mixture of equal volumes of fresh liquor and waste cooking liquor from a previous operation, the latter having been freed from lignin compounds by precipitation with calcium oxide while retaining its dissolved carbohydrates.

F. R. ENNOS.

Retting, decomposition, and degumming of vegetable materials to obtain long fibres or paper material. P. KÖPPEL and E. WUENSCH (G.P. 423,397, 7.3.22, and 458,909, 19.4.22).—(A) Wood, straw, flax, etc. are heated with magnesia or an alkaline-earth oxide or hydroxide, preferably under pressure in the presence of water. (B) Clay, kiesguhr, adsorptive carbon, colloidal barium sulphate, or colloidal phosphates are used in place of lime or magnesia.

A. R. POWELL.

Manufacture of straw paper. E. B. WESTON and W. G. CLARK, Assrs. to WESTON PAPER & MANUFACTURING Co. (U.S.P. 1,740,132, 17.12.29. Appl., 15.2.29).—Separate batches of straw are cooked with lime and caustic soda, respectively, and the pulp formed by mixing the cooked batches is formed into paper.

F. R. ENNOS.

Manufacture of carbon paper. G. WAGNER GES. (B.P. 307,435 and Addn. B.P. 324,859, [A] 7.3.29; [B] 5.4.29. Ger., [A] 7.3.28, [B] 8.10.28).—(A) A mixture of the pigment (dye, wax, and oil) with a solution of natural or artificial caoutchouc, gutta-percha, balata, etc. in a volatile solvent, together with softening materials and vulcanising agents if desired, is applied to the paper and the solvent allowed to evaporate. (B) The paper is pretreated with the caoutchouc solution containing softening and vulcanising agents, and, after evaporation of the solvent, a layer of pigment is applied, followed by another layer of caoutchouc.

F. R. ENNOS.

Manufacture of oil- and grease-proof liner paper or board. A. L. CLAPP (U.S.P. 1,741,556, 31.12.29. Appl., 16.7.26. Renewed 3.6.29).—The material comprises an outer layer of paper and an inner layer containing asbestos and cellulose fibres, preferably in equal pts. by wt., and lime or similar material, the inner layer, after drying, being treated with sufficient sodium silicate solution to penetrate to the outer layer.

L. A. COLES.

Manufacture of imitation fur products. G. ROMANE (B.P. 312,184, 21.5.29. Fr., 21.5.28).

Drying apparatus for loose [textile] material. B. SCHILDE MASCHINENBAU-A.-G. (B.P. 305,979, 1.11.28. Ger., 13.2.28).

Spinning machinery for artificial silk. HARBEN'S (VISCOSE SILK MANUFACTURING), LTD., and L. C. GOODWIN (B.P. 325,051, 17.12.28).

Manufacture of seamless flexible tubes from cellulose solutions, particularly viscose. WOLFF &

Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 304,717, 7.12.28. Ger., 25.1.28).

Separation of sodium sulphate from sulphuric acid (B.P. 298,639). **Alkali lyes** (B.P. 303,482).—See VII. **Rubber-coated fabric** (B.P. 324,664).—See XIV. **Cellulose waste-liquors in agriculture** (F.P. 611,545 and Addn. F.P. 32,516).—See XVI. **Sutures** (U.S.P. 1,741,893).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Bleaching of [sensitive vegetable and animal] fibres and fabrics. H. GOLDARBEITER (B.P. 304,680, 31.10.28. Austr., 24.1.28).—In bleaching furs and feathers by means of alkaline solutions of peroxides, particularly hydrogen peroxide, in the presence of catalysts, the treatment is carried out in a succession of steps. The furs are treated first with a solution of the catalyst, *e.g.*, a cobalt salt, then with a solution of hydrogen peroxide, and are finally exposed to alkaline vapours, *e.g.*, ammonia; deleterious action on the furs is thereby avoided.

A. J. HALL.

Manufacture of [azo] dyes on the fibre. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,737, 21.8.28).—Diazonium fluosulphonates, which are stable, readily soluble in water, and have low inflammability, are used for developing ice-colours.

C. HOLLINS.

Dyeing or otherwise colouring of cellulose derivatives. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 322,463, 1.8.28. Cf. B.P. 321,401; B., 1930, 54).—Acetate silk etc. is dyed directly with benzo- or naphtha-quinone mono- and di-arylimides or with arylamino-derivatives of the quinones. Examples are: 2:5-dianilino-*p*-benzoquinone (reddish-grey), 2:5-di-(*p*-aminoanilino)-*p*-benzoquinone (brown), 2:5-bis-(*p*-dimethylaminoanilino)-*p*-benzoquinone (purple-brown), 3:6-dichloro-2:5-dianilino-*p*-benzoquinone (yellow), 2-*p*-anisidino- α -naphthaquinone (heliotrope), 1-imino-4-*p*-tolylimino- β -naphthol (orange), 1-imino-4-*p*-aminoanilino- β -naphthol (puce).

C. HOLLINS.

Production of fast-coloured discharge effects. DURAND & HUGUENIN SOC. ANON., Assees. of I. G. FARBENIND. A.-G. (B.P. 302,291, 13.12.28. Ger., 13.12.27).—Dyeings dischargeable by oxidation are printed with an oxidising discharge paste containing a soluble ester of a leuco-vat dye (Indigosols); *e.g.*, 6:6-diethoxyindigo leuco-ester is printed with chlorate on goods dyed with indigo, the fabric is dried, steamed, treated with a 1% solution of water-glass, and washed.

C. HOLLINS.

Production of fast dyeings and prints by means of [water-soluble esters of] vat dyes. DURAND & HUGUENIN SOC. ANON. (B.P. 298,088, 1.10.28. Ger., 1.10.27).—More uniform and intense dyeings and prints are obtained by the processes of B.P. 203,681, 212,546, 220,964, 281,336, and 306,800 (B., 1924, 978; 1925, 627, 879; 1929, 280, 320) if glycol or monoethyl glycol is added to the dyebath or printing paste.

A. J. HALL.

Treating [ageing or reducing printed or dyed] fabrics. E. S. CHAPIN and A. H. JACOBY, Assrs. to

DELTEX Co. (U.S.P. 1,738,946, 10.12.29. Appl., 15.12.27. Renewed 14.5.29).—An ageing machine similar to that described in B.P. 312,423 (R., 1929, 751) is described, wherein heat liberated in the fabric during the early stages of its travel through the chamber is rapidly dissipated.

A. J. HALL.

Apparatus for examination of dyed materials in artificial light. IMPERIAL CHEM. INDUSTRIES, LTD., H. BLACKSHAW, and L. G. LAWRIE (B.P. 325,112, 18.2. and 27.3.29).—Apparatus of simple construction is described, by means of which coloured materials may be compared, or artificial sources of light compared with each other or with daylight.

A. J. HALL.

Process and agent for carroting furs. H. T. LEAVENWORTH (U.S.P. 1,738,189, 3.12.29. Appl., 4.4.28).—Furs are carroted by means of solutions containing nitric acid (in suitable excess) and chloric or perchloric acid (obtained from the barium salt), instead of the poisonous and usually employed mercuric nitrate. The furs then retain their natural sheen and felt more easily.

A. J. HALL.

Enhancing the efficacy of treatment liquids in the textile industry. H. T. BÖHME A.-G. (B.P. 301,824, 2.11.28. Ger., 6.12.27).—A small amount of a glycol ether, particularly ethers of ethylene glycol, is added to the treatment liquor; greater dispersion of the treatment agents in the liquor (*e.g.*, a direct dye) is thus obtained without giving it an objectionable odour.

A. J. HALL.

Waterproof cellulosic material. P. H. KING (B.P. 324,911, 2.11. and 6.12.28).—Waterproof cellulosic material, especially photographic film, is prepared from solutions of cellulose esters to which has been added 1–5% of bone oil and/or whale oil, or clarified neat's-foot oil, mutton fat, fish oil, or bleached beeswax; suitable plasticising agents such as camphor, phenyl or tolyl phosphate may also be added. Gelatin coatings adhere satisfactorily to such films.

A. J. HALL.

Production of waterproof materials. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 323,785, 7.8.28. Addn. to B.P. 323,501; B., 1930, 281).—The material consisting of an organic derivative of cellulose is brought to a swollen condition and treated with an emulsion of wax in soap solution, the latter being precipitated thereon by means of a metallic compound which is applied before or after the emulsion or is incorporated with the spinning solution.

F. R. ENNOS.

Preparation of waterproof materials such as tissues, yarns, etc. F. FUCHS (Austr.P. 109,167, 9.11.26).—The material is first treated with aluminium acetate solution and then impregnated with an emulsion formed by heating rubber with colophony to 200° and saponifying the product with alkali.

F. R. ENNOS.

Protection of wool, fur, etc. from attack by moth. I. G. FARBERIND A.-G. (B.P. 299,055, 18.10.28. Ger., 20.10.27).—The material is treated with a cold aqueous solution of a non-halogenated, non-sulphonated, aromatic hydroxycarboxylic acid or a substitution product thereof, together with an organic or inorganic

acid or salt if desired. Examples are hydroxybenzoic acid or its methyl ester and resorcylic acid.

F. R. ENNOS.

Production of fast dyeings and printings with vat dyes. G. DE NIEDERHÄUSERN, ASSR. to DURAND & HUGUENIN SOC. ANON. (U.S.P. 1,747,107, 11.2.30. Appl., 17.11.27. Ger., 29.11.26).—See B.P. 281,336; B., 1929, 280.

Treatment of fibrous plant tissues. D. R. NANJ, ASSR. to D. M. STEWART (U.S.P. 1,746,842, 11.2.30. Appl., 9.3.28. U.K., 29.4.27).—See B.P. 296,334; B., 1928, 809.

Treatment of dye effluents. P. MAHLER, ASSR. to DARCO SALES CORP. (U.S.P. 1,747,175, 18.2.30. Appl., 3.12.25).—See B.P. 262,382; B., 1927, 510.

[Apparatus for] parti-colour dyeing of yarns, threads, and woven fabrics. S. HOWARD, A. BAILEY, and MULTI-COLOR DYERS (1920), LTD. (B.P. 325,104, 15.2.29).

Decatising of textile fabrics. H. and W. KRANTZ (H. KRANTZ SÖHNE) (B.P. 325,052, 17.12.28. Ger., 20.10.28).

Sizing machines for artificial silk. P. ASHWORTH (B.P. 324,896, 1.11.28).

Wetting agents etc. (B.P. 322,005 and 322,429). **Water-soluble condensation products** (B.P. 320,056).—See III. **Use of vat dyes** (B.P. 322,169). **Dyes containing metal** (B.P. 298,494).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

The chamber [sulphuric acid] process. XIX. Equilibrium in the gaseous phase. M. MATSUI (J. Soc. Chem. Ind., Japan, 1930, 33, 13–16 B).—The equilibrium constant of the reaction $2\text{NO}_2 = 2\text{NO} + \text{O}_2$ over a range of temperatures is recalculated from data obtained by Bodenstein and Lindner, and compared with results from older data worked out by an equation of Lewis. The heat of reaction of the same equation is calculated from the author's equations and shown to agree well with the calorimetric values of Thomsen and Berthelot. The equation $\text{SO}_2 + \text{NO}_2 = \text{SO}_3 + \text{NO}$, water being absent in the gaseous phase, is dealt with as a combination of the reactions of sulphur dioxide and nitric oxide with oxygen. The degree of conversion of sulphur dioxide over the range 0–300° is calculated. It falls from 99.9% at 0° to 94.8% at 300°.

C. IRWIN.

High-pressure compressors for ammonia synthesis. I–III. E. BLAU (Chem.-Ztg., 1930, 54, 2–3, 29–30, 66–67).—The general requirements of such compressors are discussed, and specific examples of German-made compressors actually in use are described in detail.

S. K. TWEEDY.

Hygroscopicity of ammonium nitrate and its mixtures. S. I. VOLFKOVICH and R. E. REMEN (Trans. Sci. Inst. Fertilisers, Moscow, 1927, No. 46, 5–52).—Ammonium nitrate does not absorb moisture at 69–84% humidity between –10° and +20°. The various crystalline modifications of ammonium nitrate differ

in hygroscopicity, the most stable being the γ -modification. Admixture with sodium chloride or carnallite increases, and with potassium chloride, potassium dihydrogen phosphate, or ammonium sulphate decreases, the absorption of water. In mixtures of solid salt with soluble substances a reaction takes place; the hygroscopicity is at first additive, but later diminishes. Between 18° and 140° ammonia is not lost from mixtures of ammonium nitrate with calcium salts.

CHEMICAL ABSTRACTS.

Lixiviation of Chili saltpetre at various temperatures. G. LEIMBACH (Caliche, 1929, 11, 290—293).—The general methods available for the separation of the various components of Chili saltpetre are described, and isotherms and abridged tables are given for the system $\text{NaNO}_3\text{--NaCl--Na}_2\text{SO}_4\text{--H}_2\text{O}$. H. F. GILLBE.

Conversion of technical barium sulphide into barium chloride and sulphur or sulphur chloride. F. STROKOV (Trans. State Inst. Appl. Chem. Moscow, 1928, 11, 167—185).—Dry barium sulphide is boiled with sulphur monochloride (10—20% in excess), barium chloride and sulphur being formed. A larger excess of sulphur monochloride may be employed.

CHEMICAL ABSTRACTS.

Viscose. I. Analysis of barium thiocarbonate. K. TANEMURA and T. KOHNO. **II. Influence of sodium sulphite on the reaction between carbon disulphide and caustic soda.** K. TANEMURA, T. KOHNO, and S. MIYOSHI (J. Soc. Chem. Ind. Japan, 1930, 33, 3—4B, 4—7B).—I. Barium thiocarbonate, which can be prepared in a purer state than the sodium salt, was determined by (1) reaction with zinc sulphate and determination of the loss of weight through evaporation at $50\text{--}60^\circ$; (2) neutralisation with acetic acid to phenolphthalein and titration with iodine; (3) neutralisation with acetic acid, addition of formaldehyde, and titration with acetic acid. The results are in fair agreement on the assumption that the reaction in the second method is $\text{BaCS}_3 + 2\text{I} = \text{BaI}_2\text{CS}_2 + \text{S}$. This reaction is accordingly considered to be established.

II. If caustic soda is in excess only sodium sulphide is formed. Excess of carbon disulphide gives as final products chiefly sodium thiocarbonate and carbonate. Sodium sulphide is an intermediate product, later disappearing with continued agitation. Sodium sulphite was found to increase the yield of sodium sulphide at the expense of sodium thiocarbonate. Sodium thiocarbonate in 1% solution is only slowly hydrolysed by caustic soda when stored in stoppered bottles at the ordinary temperature, the concentration being reduced by about one half in a month.

C. IRWIN.

[Preparation of hydrogen by] ignition of powdered zinc, cadmium, and iron with calcium hydroxide. T. GEUTHER (Chem.-Ztg., 1930, 54, 133).—An historical account of the process is given, and the possible application of the gas in autogenous welding is indicated. If zinc dust containing cadmium be heated with slaked lime the latter metal remains unattacked, and is subsequently obtained in the form of a fused regulus.

H. F. HARWOOD.

Heating bath for digestions in Kjeldahl's method of determining nitrogen. E. STAUDT (Chem.-Ztg.,

1930, 54, 9).—The bath consists of an alloy of equal parts by weight of lead and tin, having m.p. 210° . The heating is uniform, and the time of digestion shortened. The surface of the bath oxidises slightly.

S. K. TWEEDY.

Carbonyl chloride. A. E. KRETOV (J. Appl. Chem., Russia, 1929, 2, 483—501).—Up to -10° , carbonyl chloride dissolves less than 0.1% of carbon monoxide or dioxide, the solubility of chlorine being 3.9—4.3% at -10° , and of hydrogen chloride 1.6—2.2% at 0° and 4% at -12° . Carbonyl chloride corrodes impure, but not pure, iron. The commercial product contains (minimally) 0.08—0.20% FeCl_3 , 0.2—1.2% HCl , and 0.1—0.3% of organic substances, and has d 1.428—1.431. Analytical methods are described. Kling and Schmütz' method is recommended, not being affected by the presence of chlorine up to 6.5%. Hydrogen chloride cannot be quantitatively separated from carbonyl chloride by washing with water.

CHEMICAL ABSTRACTS.

Absorption of impurities from air. BARTH.—See I. **Sodium selenosulphate.** A. and L. LUMIÈRE and SEYEWETZ.—See XXI.

PATENTS.

Manufacture of sulphuric acid. H. PETERSEN (G.P. 459,978, 17.12.24).—The acid is made by the chamber process in a series of towers, the gas mixture being so regulated that the acid leaving all the towers except the last is saturated with nitrous gases while the last tower serves as a concentrating and denitrating tower. The nitrous gases are mixed with the requisite quantity of water in the form of a fine spray before being subdivided between the various towers. A. R. POWELL.

Manufacture of sulphuric acid and artificial Portland cement. H. MEURIS (B.P. 324,199, 28.12.28. Belg., 6.10.28).—Mixtures comprising clay or shale, calcium sulphate, up to 2.2 pts. of carbon, and up to 18.3 pts. of calcium carbonate or 21.3 pts. of ferrous carbonate per 100 pts. of calcium sulphate, preferably compressed into cylindrical briquettes, are heated under conditions such that the carbon, which reacts with the carbon dioxide liberated from the carbonate to form carbon monoxide, is burnt away completely before the material enters the clinkering zone. The sulphur dioxide is used for the manufacture of sulphuric acid.

L. A. COLES.

Purification (de-arsenification) of sulphuric acid. J. RILEY & SONS, LTD., W. WILDE, W. M. COATES, and W. H. BENTLEY (B.P. 324,844, 7.3.29).—When sulphuric acid is freed from arsenic by treatment with hydrogen sulphide followed by flotation with hydrocarbons, such as paraffin oil or molten paraffin wax or naphthalene, the acid is liable to become contaminated owing to its solvent action on the hydrocarbons. This disadvantage is avoided if chlorinated hydrocarbons are employed as flotation agents, particularly those of the aromatic series, the aliphatic chlorinated hydrocarbons being less efficient. Oily dichlorobenzene is recommended because of its comparative cheapness, its resistance to concentrated sulphuric acid, its high efficiency, and the ease of its recovery.

W. J. WRIGHT.

Separation of sodium sulphate from sulphuric acid. VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 298,639, 10.10.28. Ger., 13.10.27).—The acid, *e.g.*, spent spinning bath from the manufacture of artificial silk, is evaporated to that concentration (70–85 wt.-% of acid) at which it possesses the least solubility coefficient for sodium sulphate at the ordinary temperature, and is then cooled to 40° or below, crystallisation being promoted, if necessary, by seeding, since the solutions are very liable to supercool. The crystals may be removed by centrifuging or filtration under pressure. The solubilities of sodium sulphate in sulphuric acid of 30–98 wt.-% for temperatures between 0° and 80° are recorded. S. K. TWEEDY.

Rendering suitable for re-use sulphuric acid used for treatment of titanium ores containing chromium. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 309,597, 10.4.29. Ger., 14.4.28).—The used acid is concentrated to at least 92% and then kept at about 300° for 1 hr. to effect removal of chromium oxide.

H. ROYAL-DAWSON.

Nitric acid concentration. R. H. MCKEE, ASSR. to SOUTHERN ELECTRO-CHEM. CO. (U.S.P. 1,740,549, 24.12.29. Appl., 22.8.25).—In concentrating nitric acid from acids containing a large amount of organic matter, such as nitroglycerin waste acids, the acids mixed with a dehydrating agent such as sulphuric acid are passed in countercurrent to steam over a relatively long path. The temperature of the exit gases is controlled within the limits 35–79.4°, the temperature being increased or decreased in inverse proportion to the amount of organic matter present. The nitric acid vapours are condensed, and the nitrogen oxides absorbed in hyponitrite towers, whence the acid is fed to the concentration towers. Nearly all of the nitric acid in the waste acids may thus be recovered as acid of 95% concentration. W. J. WRIGHT.

Production of hydrochloric acid and magnesia from magnesium chloride. SALZBERGWERK NEUSTASSFURT (G.P. 459,809, 29.9.26).—Magnesium chloride or oxychloride is heated in steam or the vapour of dilute hydrochloric acid in a shaft furnace provided with a conveyor mechanism which removes the charge when almost, but not completely, decomposed, to a rotating cylindrical furnace in which the final decomposition is carried out. A. R. POWELL.

Preparation of phosphoric acid from crude phosphates. K. NIEDENZU (G.P. 459,254, 17.10.25).—A mixture of the phosphate with sand, coal, and a small quantity of alumina or ferric oxide is finely ground and heated at 900–1600° in a rotating furnace in an oxidising atmosphere and the phosphorus pentoxide evolved is collected in water. A. R. POWELL.

Manufacture of chromic acid. M. J. UDY (U.S.P. 1,739,107, 10.12.29. Appl., 5.11.25).—A tervalent chromium compound, *e.g.*, chromic hydroxide, is electrolytically oxidised at 95–100° in an electrolyte containing 15–30% of chromic acid between an insoluble (*e.g.*, lead) anode and a preferably hollow cathode (*e.g.*, of aluminium or iron) immersed in the electrolyte. The electrolyte is kept substantially uniform in composition

between the electrodes by supplying tervalent chromium solution equivalent to the chromic acid solution drawn off. S. K. TWEEDY.

Use of ammonia as a lubricant. F. A. ERNST and C. H. YOUNG, ASSRS. to A. B. LAMB (U.S.P. 1,739,957, 17.12.29. Appl., 24.6.26).—In the synthetic production of ammonia, the gases from the converter are passed to a condenser to cool the gases and to liquefy the ammonia, and the gases and ammonia are conducted to the suction side of the circulating pump where the ammonia is used to lubricate the working parts. The ammonia is caused to expand, thereby cooling the pump, and is then passed through cooling apparatus to a receiver, whence the gases are recirculated through the system. W. J. WRIGHT.

Cleaning of alkali lyes. R. WINTERNITZ (B.P. 303,482, 3.1.29. Ger., 4.1.28).—The organic contaminating substances are separated by the addition of an alcohol (*e.g.*, methyl or ethyl alcohol), any colour being also removed, if necessary, by the addition of lead salts. The process is advantageously applicable to the waste lyes of the viscose silk industry. S. K. TWEEDY.

Production of ammonia carbonate. SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE ET PROD. CHIM. (F.P. 578,333, 14.5.23).—Gases with a low content of ammonia and carbon dioxide are passed through a chamber into which a fine spray of water is admitted. A. R. POWELL.

Treatment of salt masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,916, 5.11.28).—Crude, soluble salt masses, *e.g.*, caliche or reduced sodium or barium sulphate melts, are lixiviated by means of steam introduced from above. Water heated above 100° may be used and the pressure released in the treatment vessel, or the charge may be heated to above 100° before water or a mother-liquor is sprayed thereon. Suitable apparatus is described. F. G. CLARKE.

Obtaining ammonium sulphate and sulphur from ammonium thiocyanate. C. J. HANSEN, ASSEE. of H. KOPPERS A.-G. (B.P. 309,565, 24.9.28. Ger., 14.4.28).—Ammonium thiocyanate liquors obtained, *e.g.*, in the purification of coal gas are heated, preferably above 140° and under raised pressure, in the presence of sulphurous acid and/or liquors containing ammonium polythionates, thiosulphates, etc. obtained by a thionate scrubbing process, the proportions being such that 2 mols. of free or combined sulphur dioxide are available per mol. of thiocyanate. L. A. COLES.

Leaching of raw phosphate. KÜNSTDUNGER-PATENT-VERWERTUNGS A.-G., ASSEES. of F. G. LILJENROTH (B.P. 301,864, and Addn. B.P. 309,023, [A] 30.10.28, [B] 7.3.29. Swed., [A] 7.12.27, [B] 3.4.28).—Raw phosphate is leached with acid or alkali (*e.g.*, potassium or ammonium) sulphate solution (A) at temperatures of at least 80°, or (B) advantageously from 95° to the b.p. of the solution, when the calcium sulphate is converted into the crystalline form and thus readily separated. The lime may also be allowed to dissolve and be precipitated as sulphate at the said temperature, or it may be precipitated during or after leaching in ordinary form and then subjected to the elevated temperature. An alkali salt (phosphate, sulphate,

nitrate, or chloride of ammonium or potassium) may advantageously be present in the liquor at any stage in order to facilitate the separation of the calcium sulphate. S. K. TWEEDY.

Refining of crude borax. C. F. RITCHIE and W. A. GALE, ASSRS. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,739,091, 10.12.29. Appl., 8.10.26).—Sodium metaborate forms the sparingly soluble double salts, $\text{Na}_3\text{XO}_4 \cdot \text{NaBO}_2 \cdot 18\text{H}_2\text{O}$, where $\text{X} = \text{As}$ or P . The crude borax is extracted at 22° or above with natural brine substantially saturated with respect to borax; the arsenate and phosphate present pass into solution as the above double salts together with other soluble impurities. S. K. TWEEDY.

Production of potash alum from leucite. G. A. BLANC (F.P. 578,472, 23.5.23).—The powdered mineral is digested with sulphuric acid at 70–80° and the filtered solution is cooled rapidly to obtain potash alum in a finely-crystallised form free from silica and iron. The mother-liquor is agitated with a small quantity of barium or calcium carbonate, whereby its iron content is adsorbed by the barium or calcium sulphate formed. After filtration the liquor is returned to the digesting pans. A. R. POWELL.

Preparation of alumina and potassium sulphate. G. S. TILLEY, M. E. DAM, and E. S. DAM (B.P. 325,017, 13.11.28).—Potash alum is heated at a temperature (80–85°) below that at which it would melt in its water of crystallisation, and when more than half the latter has been removed the temperature is raised sufficiently high (800–1000°) to drive off the sulphur trioxide combined with the alumina without dissociating the potassium sulphate. The latter is then removed by lixiviation. F. G. CLARKE.

Rotating, externally-heated drum furnace for production of sulphate from salt and acid or hydrogen sulphate. F. SCHUCH (G.P. 457,897, 18.10.25).—The furnace is provided with an after-gasification chamber which widens in a stepwise manner, has an enlarged reaction surface, and may be separated from the preliminary chamber by means of a perforated partition. A transport device for supplying salt, having, if desired, a scraping device rigidly attached thereto, may extend from the exterior into the after-chamber. S. K. TWEEDY.

Apparatus for the manufacture of calcium cyanamide. G. HILGER (G.P. 458,028, 18.7.26).—The apparatus comprises a series of reaction chambers separated from one another by passages and arranged regularly around a central supply-pipe for nitrogen, which can be passed to each chamber in turn by means of a rotating pipe. The walls of each chamber carry a number of pipes through which steam, air, or other gas is passed to regulate the exothermic reaction, and means are provided for passing the hot gases from the pipes of one chamber through those of the next to preheat the charge therein. A. R. POWELL.

Continuous production of calcium cyanamide. H. WITTEK (G.P. 458,029, 15.5.26).—A shaft furnace is lined with a heating surface of granular material (e.g., of coke or sand) which is as gas-imperious as

possible, and calcium carbide is uninterruptedly charged into the central space, the cyanamide formed being withdrawn at the other end. S. K. TWEEDY.

Drying of powdered gypsum in cylindrical drums. EISENGIESSEREI U. MASCHINENFABR. MOSEBACH & SOHN (G.P. 459,460, 11.12.26).—The material is separated by sieving into fine, almost dry powder and damp agglomerated aggregates, the former being dried by a direct current of air in a rotating cylinder and the latter with a countercurrent of air at below 200°.

A. R. POWELL.

Manufacture of lead sulphate. A. P. CASSOU (F.P. 635,134, 30.5.27).—Dilute sulphuric acid containing lead or alkali sulphide or an insoluble lead salt is electrolysed at 40° with alternating current between lead electrodes. A. R. POWELL.

Pure lead hydroxide from crude lead sulphate. R. DALOZE (F.P. 636,163, 12.10.26).—Impure lead sulphate, e.g., lead-chamber residue, is digested with calcium acetate solution and the resulting lead acetate solution is treated with milk of lime to precipitate lead hydroxide and regenerate the calcium acetate for use again. A. R. POWELL.

Production of white, crystalline lead acetate from pyroligneous acid. M. KLAR (F.P. 632,866, 15.4.27).—A solution containing about 20% of lead acetate, obtained by blowing air through a mixture of lead shavings and pyroligneous acid or by passing the acid in the presence of a countercurrent of air down a tower packed with lead shavings, is treated with about 2% of active charcoal, filtered, evaporated to 50–60% concentration, again treated with active charcoal, and filtered; lead acetate crystallises on cooling the solution and the mother-liquor is returned to the process. L. A. COLES.

Manufacture of shaped articles of metallic oxides. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 324,028, 10.9.28).—Porous articles of metals, produced by sintering metal powders, preferably prepared by the thermal decomposition of the carbonyls with or without pressing, are oxidised by air, carbon dioxide, etc. at a temperature below the m.p. of the metals and their oxides. The articles may contain in compact form metals which are more difficultly oxidisable than those from which the porous articles are produced, the oxidation conditions being regulated to leave such metals unoxidised. W. G. CAREY.

Production of high-percentage manganese dioxide. I. G. FARBENIND. A.-G. (B.P. 311,271, 22.4.29. Ger., 8.5.28).—Manganese carbonate (and/or bicarbonate) or manganese carbonate previously roasted in air at 250–450° is oxidised with excess of air enriched with oxygen or with pure oxygen by heating at 250–600° at ordinary or increased pressure. The conversion is effected preferably in a tube, the oxidising gas passing continuously in countercurrent to be used again after purification from detrimental gases. W. G. CAREY.

Production of solid carbon dioxide. W. HESSLING (B.P. 298,910, 15.10.28. Switz., 15.10.27).—Carbon dioxide is passed successively through the low- and high-pressure cylinders of a compressor, through a

condenser, and thence into a chamber in which it expands to a pressure slightly below that of the triple point; the solid collecting in this chamber falls into a chamber below maintained at about 1 atm. The gases liberated by evaporation in the upper and lower chambers are conveyed, respectively, to the high- and low-pressure cylinders of the compressor.

L. A. COLES.

Production of [pure] hydrogen [from methane or gases containing it]. GASVERARBEITUNGSGES. M.B.H. (B.P. 294,580, 25.7.28. Ger., 26.7.27).—Hydrogen obtained by the catalytic or thermal decomposition of methane is scrubbed, after removal of carbon dioxide, with liquid nitrogen, preferably under pressure, and is then passed over heated iron, nickel, cobalt, or copper to remove oxygen compounds and dried. The temperature of the liquid nitrogen may be so regulated that 1 vol. vaporises per 3 vols. of hydrogen, thereby yielding a mixture suitable for the synthesis of ammonia. [Stat. ref.]

L. A. COLES.

Cyanogen chloride-hydrocyanic acid mixture [as fumigant]. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,738,280, 3.12.29. Appl., 29.5.25).—The f.p. curve for mixtures of liquid hydrogen cyanide and cyanogen chloride is recorded, and it is proposed to use as a fumigant a mixture containing from 17% up to 61.4% (eutectic) of the latter compound, freezing between -18° and -45.5° (eutectic).

S. K. TWEEDY.

Recovery of oxides of nitrogen. J. Y. JOHNSON. From L. BERGFELD (B.P. 324,220, 16.1.29).—The gases are absorbed by solutions containing compounds of sexavalent chromium (potassium chromate or dichromate) or of manganese having a valency higher than 2 (manganic sulphate, calcium permanganate, etc.), and, if desired, neutral salts, *e.g.*, sodium or potassium nitrate, or acids, *e.g.*, phosphoric or arsenic acid, and are expelled by heating the solutions and the solids left on evaporating them to dryness, the residues being oxidised for re-use.

L. A. COLES.

Manufacture of nitric oxide [and chlorides or oxychlorides] from nitrosyl chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,190, 14.12.28).—Mixtures of nitrosyl chloride with carbon monoxide or gases containing it are heated in the presence of oxides, carbonates, or sulphides of metals (*e.g.*, aluminium, zinc, tin, magnesium, lead, antimony, chromium, manganese, iron, titanium, zirconium) which react with carbon monoxide and chlorine to form the metal chlorides, or of carbon, or of compounds of carbon or silicon.

L. A. COLES.

Recovery of sulphur from solutions of alkali polysulphides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,908, 31.12.28).—The solutions are heated at $150-374^{\circ}$ under their own vapour pressure. Part of the polysulphide sulphur (*e.g.*, 40–80%) separates as liquid sulphur and is run off; the process is continuous.

S. K. TWEEDY.

Production of pure sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,919, 7.1.29).—The sulphur is separated in stages from ammonium

polysulphide solutions at temperatures above its m.p. When the polysulphide solutions are progressively decomposed (cf. B.P. 269,546; B., 1928, 91) very pure sulphur is obtained at first; towards the end the metal sulphides etc. which discolour the pure sulphur obtained by the prior process separate out. Apparatus is described for carrying out the process.

S. K. TWEEDY.

Production of sulphur trioxide by combustion of sulphur. L. F. POLLAIN (F.P. 578,153, 6.9.24).—The gases evolved from the combustion of sulphur in air are passed together with more air through a hot tube arranged immediately above the burning sulphur, whereby conversion of sulphur dioxide into trioxide is stated to occur.

A. R. POWELL.

Separation of phosphorus from phosphorus vapours. VER. F. CHEM. U. MET. PROD. (G.P. 457,762, 29.10.24).—The vapours are subjected to single or repeated electrostatic influence. Particles of phosphorus mist contained in the vapours are first enlarged by being brought into intimate contact with a surface of different (lower) temperature.

S. K. TWEEDY.

Manufacture of phosphorus chlorides. E. URBAIN (B.P. 312,685, 4.5.29. Fr., 31.5.28).—Ferro-phosphorus is treated with dry chlorine to the maximum chlorination, and before a red heat is reached a further quantity of ferrophosphorus is added, which causes the decomposition of the molecular compound, $\text{FeCl}_3 \cdot \text{PCl}_5$, into ferrous chloride and phosphorus trichloride. These are easily separated by distillation. The ferrous chloride also reacts with the phosphorus pentachloride with formation of the trichloride. In another process ferrophosphorus is treated with a limited quantity of dry chlorine, and by suitably raising the temperature the pentachloride is caused to react with excess of ferrophosphorus, and the phosphorus trichloride is distilled.

W. J. WRIGHT.

Manufacture of boron trifluoride. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,016, 12.10.28).—A mixture of a fluoride and boric acid or a borate is heated with sulphuric acid in the presence of a dehydrating agent such as sulphur trioxide or excess sulphuric acid, or, alternatively, boric acid is treated with fluoro-sulphonic acid and, if desired, sulphuric acid.

L. A. COLES.

Preserving [in paper bags] solid carbon dioxide during storage and transportation thereof. J. W. MARTIN (B.P. 300,985, 21.11.28. U.S., 22.11.27).

Salts from solutions (B.P. 324,331). Water softener (B.P. 325,099).—See I. Hydrogen (B.P. 294,113). Hydrogen and hydrogen-nitrogen (F.P. 627,665). Hydrogen-carbon monoxide mixture (B.P. 323,864). Hydrogen sulphide and hydrogen cyanide from gases (B.P. 314,860).—See II. Acid-resisting alloy (B.P. 324,009). Flux for tinning etc. (B.P. 324,611).—See X. Electrolysis of chlorides (U.S.P. 1,741,290). Electrolysis of halogen salts (U.S.P. 1,740,291). Removal of chlorine ions from solutions (U.S.P. 1,740,165). Ozone (B.P. 315,349).—See XI. Colloidal white lead (B.P. 297,116). Chrome-green (U.S.P. 1,738,780).—See XIII. Insecticide (U.S.P. 1,737,132).—See XVI.

VIII.—GLASS; CERAMICS.

Problems of glass technology. E. ZSCHIMMER (*Z. angew. Chem.*, 1930, 43, 122—124).—A summary of the development of the theory of glass technology since 1900 and a formulation of outstanding problems.

F. G. TRYHORN.

Determination of the alkalis in vitreous enamels. R. B. SCHAAL (*J. Amer. Ceram. Soc.*, 1930, 13, 113—125).—Details are given of three methods of separating the other constituents of enamels from the alkalis: the acid dissolution method, used when the enamel contains elements which attack platinum; an adaptation of the J. L. Smith method; and the Jannasch-Heidenreich method. The last two are used for enamels insoluble in hydrochloric acid or for those which do not attack platinum at high temperatures. In all methods the sodium and potassium are obtained as the mixed chlorides. Two gravimetric methods are given for the determination of potassium in the mixed salts: the chloroplatinate method, with an adaptation for the presence of sulphates, and the perchloric acid method.

F. SALT.

Effect of furnace gases on glass enamels. E. E. GEISINGER and K. BERLINGHOF (*J. Amer. Ceram. Soc.*, 1930, 13, 126—142).—The effect of a number of gases and of air under pressure and at 26 in. vacuum on four ground-coat enamels was studied, the physical condition, gloss, and bonding with the steel being noted. Enamels fired in nitrogen and carbon dioxide excelled in gloss and gave the best results. These were followed, in order of merit, by enamels fired in air at normal pressure, air at 22 lb. pressure, and oxygen. Carbon monoxide and methane also gave good results. Hence it follows that the products of complete combustion of fuels are not harmful to enamels; water vapour, however, is an important exception. Sulphur dioxide is also harmful.

F. SALT.

Behaviour of soluble salts during the burning of clay: a contribution to the problem of efflorescence. II. **Behaviour of salt mixtures.** A. SIMON and W. VETTER (*Z. anorg. Chem.*, 1930, 186, 225—250).—The efflorescences which are frequently seen on bricks consist usually of hydrated magnesium and calcium sulphates, the magnesium salt being formed by double decomposition between magnesium oxide or silicate and calcium sulphate when the bricks are exposed to rain and sunshine. Experiments have been made on brick clay and pure kaolinite to which a sulphate has been added to discover a method of removing the sulphur trioxide during the burning operation. Addition of sodium, calcium, or magnesium chloride to the damp clay before moulding and drying causes most of the sulphate present to disappear at 1000°. Practically the whole of the chlorine is eliminated at 750°, at which temperature only traces of sulphate are decomposed; on further heating, however, rapid decomposition of the sulphate occurs, probably owing to the secondary action of an impurity in the clay the nature of which has been modified by the volatilisation of the chlorine. Fluorides have a similar effect, but are not quite so active as chlorides. Tests in a commercial brick kiln showed that, even in an atmosphere containing sulphur dioxide,

addition of sodium chloride to brick clay caused practically complete expulsion of sulphur trioxide at the usual firing temperature of 900—1000°.

A. R. POWELL.

Adaptability of various methods to the colour determination of whiteware bodies. W. W. MEYER (*J. Amer. Ceram. Soc.*, 1930, 13, 98—112).—Colours are defined in terms of hue, value, and chroma. A number of instruments and methods of measuring colour are critically examined, and it is noted that in most cases insufficient importance is attached to the source of illumination. The theory of the subject is discussed in some detail. A modification of the Pfund colorimeter is suggested as the best means of measuring the colour of bodies nearly white in colour.

F. SALT.

Cold crushing strength of firebricks. H. K. MITRA (*J. Amer. Ceram. Soc.*, 1930, 13, 85—97).—Six varieties of firebricks were investigated, the determinations being made in three directions, viz., flat, on edge, and endwise. The moduli of rupture and porosity were also determined, and the latter was correlated with the other two properties. No simple mathematical relation was found between porosity and cold crushing strength or modulus of rupture; between the crushing strengths in the three different directions on the same make of brick; nor between modulus of rupture and cold crushing strength. To determine the cold crushing strength, it is recommended that firebricks be tested on end. A capping material consisting of a mixture of sand and molten sulphur was found more suitable for the crushing tests than materials hitherto used for this purpose.

F. SALT.

Caustic sludge. LORENZ.—See V.

PATENTS.

Clouding of enamels and glasses and clouding agents therefor. I. KREIDL (B.P. 297,737, 21.9.28. Austr., 26.9.27).—Clouding is produced by the formation in the enamel or glass of fine gas bubbles from decomposed compounds or complexes, which contain, or leave as residue, a small insoluble portion in the enamel, insufficient of itself to produce mechanical clouding. The clouding agent may be a suitable colloidal compound, or a compound or complex containing a large volatile (preferably organic) portion. Fatty acid compounds of aluminium, tin, titanium, etc. are mentioned.

F. SALT.

Firing of tiles. J. P. JOHNSON (U.S.P. 1,739,151, 10.12.29. Appl., 5.3.28).—To prevent warping and cracking, tiles are placed on edge in saggars, spaced from each other, the lower edges resting on the bottom of the sagger. The tiles are covered, and the spaces between them filled, with a material sufficiently refractory to resist the firing heat.

F. SALT.

Sparking plugs. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,316, 20.8.28).—To prevent loss of the insulating property at high temperatures, the insulation of such plugs is effected with highly refractory materials melting above 1600°, such as alumina (other than that fused above 1600°), Marquardt's porcelain, and the refractory rare earths. The refractory material is provided with a glaze of similar composition.

F. SALT.

Purification of kaolin and clays. Y. M. PIRAUD (F.P. 633,745, 3.5.27).—The material is suspended in preferably ten times its weight of pure water, and neutral or alkaline protective colloids such as gum arabic, pectin, soap solution containing amines or ammonia, etc. are added. The kaolin forms a true colloidal suspension which can be decanted off after 1 hr. or so and coagulated, *e.g.*, by addition of acid. Traces of metallic salts (*e.g.*, of copper, silver, nickel) must be avoided.

S. K. TWEEDY.

Drying of green clay goods prior to baking. J. PROCTER (B.P. 324,380, 26.9.28).—A brick or pottery kiln consisting of a group of chambers, all of which are adapted to receive green clay ware for drying and firing, has a separate suction main and a separate discharge or pressure main connected, respectively, to the suction and discharge sides of a fan or blower. Each of these mains may be connected to each chamber of the group, so that hot gas may be withdrawn from any one or more of the chambers and passed to others.

F. SALT.

Drying of ceramic ware. W. J. MILLER (U.S.P. 1,737,259, 26.11.29. Appl., 6.8.27).—Improved drying apparatus comprises a drying tunnel or chamber and apparatus arranged over air-conditioning apparatus in zones. Temperature is regulated and controlled in each zone, or portion of a zone, by means of heating or refrigerating elements. The air-conditioning apparatus provides means for humidifying or dehumidifying the spent air and/or atmospheric air, and also for raising and lowering the temperature of such air.

F. SALT.

Manufacture of tiles, bricks, and other pottery ware. J. W. MELLOR, and CERAMIC PATENT HOLDINGS, LTD. (B.P. 323,379, 11.1.29).—Bricks, tiles, etc. are made by the "semi-dry-press" method from the waste material remaining from the washing of china clay to which a suitable fluxing agent is added to reduce the firing temperature below that of the ordinary biscuit oven. The flux may be a borate of an alkaline earth (including magnesia), an alkali silicate, an alkaline-earth silicate, or mixtures of these, or a suitable furnace slag.

F. SALT.

Manufacture of refractories. R. L. FRINK (U.S.P. 1,736,909, 26.11.29. Appl., 18.9.26. Renewed 24.10.28).—Highly refractory material, such as natural or artificial sillimanite or mullite, is ground and suitably graded as to grain size and is mixed with about 10% of a frit comprising silica, boric acid, and alkali, which is also powdered and classified. The mixture is heated in a rotary or other suitable kiln, which can be regulated so that the mass can be discharged in sufficiently fluid state to fill moulds of desired form. Pressure can then be applied, or other means adopted, to complete the shaping operation.

F. SALT.

Production of highly refractory bodies. A. SPRENGER (B.P. 324,312, 17.7.28).—A mixture of the oxides of aluminium, magnesium, chromium, and silicon is fused, without the addition of a reducing agent, the mixture being in such proportions that the alumina may combine with the silica, or in part with the silica and partly with the magnesia, to form at least simple double oxides. The melted mass may be cast in moulds, or it

may be allowed to cool and then broken up and moulded with a suitable bond. The heating and cooling may be so conducted as to give 50% of vitreous substance in the finished product.

F. SALT.

Production of heat-absorbing borosilicate glass and batches therefor. W. H. RISING, ASSR. to CORNING GLASS WORKS (U.S.P. 1,737,685, 3.12.29. Appl., 24.5.28).—See B.P. 312,311; B., 1930, 145.

Manufacture of [solid] bodies of great density [from refractory materials]. M. PIRANI, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,747,133, 11.2.30. Appl., 11.5.28. Ger., 29.6.27).—See B.P. 292,997; B., 1929, 395.

Glass-melting apparatus. W. A. MORTON (B.P. 325,238, 12.11.28).

Rolling and annealing of glass plates. N. V. Mij. TOT BEHEER EN EXPLOIT. VAN OCTROOIEN (B.P. 325,185, 17.6.29. Ger., 12.10.28).

Glass furnaces (U.S.P. 1,741,469 and 1,741,977).—See XI.

IX.—BUILDING MATERIALS.

Cement past and present. D. B. BUTLER (Cement, 1930, 3, 273—292).—A survey of the manufacture of cement with illustrated descriptions of modern plant and methods. It is stated that the average quality of British cements considerably exceeds the requirements of the British Standard Specification of 1925.

C. A. KING.

Laitance of cement mortars and concrete. II. S. NAGAI and K. YOSHIZAWA (J. Soc. Chem. Ind., Japan, 1930, 33, 19—21 B; cf. B., 1930, 191).—An increase of water in a 1 : 2 cement-sand mortar caused an increase in the carbonate and sulphate content of the laitance, and similarly with neat cement. The use of such additions as gypsum or calcium chloride or oxychloride resulted in the added acid radical being found in the laitance.

C. IRWIN.

Calcium alloys. MEYER and GORALCZYK.—See X.

PATENTS.

Burning of lime and the like with powdered fuel. A. B. HELBIG (G.P. 458,374, 9.12.25).—The operation is carried out in the usual lime kiln, but in order to avoid dead-burning of the lime the charge is withdrawn from the kiln through the firing openings before all the carbon dioxide is removed, and the remainder is then expelled by passing air through the hot material in the cooling chamber.

A. R. POWELL.

Manufacture of slaked lime free from impurities. MASCHINENFABR. GREVENBROICH (G.P. 458,873, 10.8.26).—Burnt limestone is slaked with the minimum of water and the fine slaked lime is removed by screening. The coarse residue containing sand, unburnt lime, and dead-burnt lime is then rotated in a damp condition in a heated drum until all the lime present is hydrated to a fine dry powder, which is again separated by sieving.

A. R. POWELL.

Utilisation of the steam produced in the manufacture of cement by the wet method. A. EIGER (B.P. 324,798, 22.1.29).—The raw slurry is heated by waste gases to 80—100° and then dried by means of

superheated steam (400°) either in a drying zone of a rotary kiln or in a separate chamber. Water vapour from these processes is superheated again to about 400° by gases leaving the sintering zone of the kiln, and is used in part for power purposes. C. A. KING.

[Porous] cement, concrete, gypsum, and the like. M. PLATSCH (B.P. 299,854, 1.11.28. Ger., 2.11.27).—A granular substance, which liberates gas in the presence of moisture, *e.g.*, calcium hydride or carbide, is coated with an innocuous, difficultly soluble substance, *e.g.*, gelatin or fused water-glass, and is then incorporated in mouldable masses of cement, clay, gypsum, etc. The soluble coating may combine with the plastic mass and strengthen it. F. G. CLARKE.

Manufacture of artificial marble. S. KIN (B.P. 324,038, 15.10.28).—A mixture of white cement with water, alone or with powdered glass and/or white sand, is worked into glazed or enamelled moulds, marbled with Indian ink or pigment, backed with a sand-cement mixture, and pressed at 40 lb./in.² The blocks are stacked in the dark for a fortnight at 16° at a humidity of 100%, and are then exposed to dry heat at 38° for 24 hrs. W. G. CAREY.

Manufacture of bricks, blocks, etc. from loam or earth. BINDPHAST PRODUCTS, LTD., and J. A. GREENE (B.P. 324,110, 18.10.28).—Loam or earth is mixed with 15–25% of a solution of equal parts by wt. of calcined magnesia and magnesium chloride of 33% concentration. The hardness is improved by the addition of 5% of zinc oxide, alum, or sodium silicate. W. G. CAREY.

Manufacture of a durable coating composition [for wood, masonry, etc.]. G. EHRENTAUF (B.P. 324,970, 5.11.28).—A pulverulent clay is mixed, in the cold, with sufficient water-glass to give a product of paint-like consistency. Colouring materials may be added. [Stat. ref.] F. G. CLARKE.

Solutions for impregnating vegetable material [*e.g.*, wood] to protect it against parasitic attack. F. MOLL (G.P. 453,192, 7.10.24).—The solutions contain, in addition to the active material, substances capable of retarding the precipitation of the active material or the formation of insoluble compounds in the vegetable tissue, *e.g.*, 0.25% of sodium chloride (or 0.2% of hydrogen chloride), or 0.1% of hydrogen chloride is added to 1% mercuric chloride solution, or to a 0.4% solution of the aniline or sodium derivatives of dinitrophenols, respectively. (Cf. B., 1927, 878.) L. A. COLES.

Constructing and repairing roads. C. J. HOGSHOLT (B.P. 324,654, 29.10.28).—Coralliferous limestone coated with tar or asphalt emulsions forms the foundation of a road; the surface coating consists of hard material, *e.g.*, gravel, stone chips. A surplus of tar may be used to assist in binding the covering layer. C. A. KING.

Building cement or plaster. C. E. KRAUS (U.S.P. 1,747,551, 18.2.30. Appl., 18.6.25).—See B.P. 253,929; B., 1928, 15.

[Tapping or abrading] means for testing the durability and hardness of tiles and other flooring material and the like. J. LYONS & Co., LTD., L. H. LAMPITT, and P. L. BILHAM (B.P. 325,358, 21.1.29.)

Cellulose-cement compositions (B.P. 324,652).—See V. Artificial Portland cement (B.P. 324,199).—See VII. Cement furnace (U.S.P. 1,741,977).—See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Reactions in the lower part of the [iron] blast furnace. A. WAGNER (Stahl u. Eisen, 1930, 50, 122–126).—The relatively low extraction of manganese and phosphorus from minette ores in the usual blast-furnace practice is attributed to the relatively small area of penetration of the blast in front of the tuyères. Much better extractions of these elements in the pig iron produced are obtained by using a tuyère shaped like two truncated cones joined at their narrow ends. Large-scale experiments have shown that for an equal smallest diameter this type of tuyère will pass 15% more blast into the furnace than the ordinary tapering tuyère used up to the present. This extra wind increases the reducing zone behind the initial oxidising zone in front of the tuyères, and thus increases the rate of reduction of phosphorus and manganese compounds.

A. R. POWELL.

Temperature measurements in the [iron] blast furnace. P. RHEINLÄNDER (Arch. Eisenhüttenw., 1929—1930, 3, 487–503; Stahl u. Eisen, 1930, 50, 205–206).—Observations made during the operation of three blast furnaces of the temperature at different parts are recorded and discussed. Total-radiation pyrometers were used for measuring the temperature of the tuyère zone at four points; the mean temperature in front of the tuyères varied from about 1700° to 1800°, whereas the central core at this point was only at 1000–1200°, the smallest temperature fall from outside to centre being recorded with the smallest furnace. Variations in the blast had very little effect on these temperatures unless the temperature of the blast was changed, but local temperature variations caused appreciable differences in the composition of the metal produced; the steadier were the conditions of operation the poorer was the metal in sulphur and manganese. Most of these local variations in the temperature were found to be due to insufficiently preheated parts of the charge falling into the tuyère zone.

A. R. POWELL.

Heating of open-hearth furnaces and casting ladles. G. BULLE (Stahl u. Eisen, 1930, 50, 193–205).—Data with regard to the heat consumption during the operation of open-hearth steel furnaces has been obtained as a result of a questionnaire sent to numerous German steelworks, and the information is recorded in detail in a series of tables and briefly discussed. According to the size of the furnace, 200–1000 × 10⁶ kg.-cal. are required to bring a new furnace into full operation. Similar data are given for the heating of casting ladles.

A. R. POWELL.

Influence of the age of the furnace on the time of melting and the energy consumption of electric steel furnaces. S. KRIZ and H. KRAL (Stahl u. Eisen, 1930, 50, 221–222).—The time required for melting the charge and the energy consumed are, respectively, 35 and 25% above the average for the first melt after relining an electric steel furnace, the average life of which

is 29 melts. The minimum time and energy consumption, 96 and 94% of the average, respectively, are obtained with the fifth and sixth melts, after which there is a gradual increase until the lining is destroyed. The time and energy required in the refining operation are below the average for the first half of the life of the lining, and reach a minimum at the fourth melt. The experiments were made with a 5.5-ton arc furnace and summarise the experience of over 2000 melts.

A. R. POWELL.

Two-chamber, regenerative, direct gas-fired, steel-heating furnace. H. FREY (Stahl u. Eisen, 1930, 50, 223—229).—A new type of Siemens furnace for preheating steel billets for hot-rolling is described with reference to scale drawings, and numerous details of its operation are recorded in a series of tables. The air from the two regenerative chambers is divided, one part being fed to tuyère-like gas burners, the flames from which pass through the furnace in the same direction as the charge, and the second part being used for the combustion of cold gas in the shaft in front of the regenerative chambers. Three modifications of the furnace are described, in which provision is made for the efficient cooling of the crown of furnaces fired with blast-furnace gas and in which water-cooled slide rails with an efficiency of 85% are assembled in the regenerative furnaces.

A. R. POWELL.

Alteration of cast-iron [water-pipes by corrosion] in the earth of a town. A. ROMWALTER (Gas- u. Wasserfach, 1929, 73, 178—182).—After about 30 years' use in the water system of a Hungarian town large cast-iron pipes were found at certain places to be completely converted into a light, porous, dark-grey mass, of apparent d 1.49, which could be cut with a knife and had an unctuous feel like graphite. Analysis gave the following composition: 44.5% Fe, 12.4% C, 9.4% Si, 5.07% P, 2.47% of moisture, 9.23% of combined water, and 16.93% O (diff.). Only a very small portion of the iron was in the metallic form, the remainder being present as a ferrous salt; most of the carbon was in the form of graphite, the remainder being present apparently as humic acid, and part, at least, of the phosphorus was there as phosphite. The surrounding soil was of a loamy character with a high content of organic acids, but almost free from chlorides. Several theories in explanation of the composition of the material are discussed, but sufficient evidence in favour of any particular one could not be obtained. The cause of the corrosion is considered to be the acid nature of the soil, which contained much colloidal silicic acid, but oxidation by the air in the water inside the pipes cannot have played any great part in the action.

A. R. POWELL.

Corrosion of underground steel structures and its prevention. R. E. WILSON (J. West. Soc. Eng., 1929, 34, 578—595).—An explanation and discussion.

CHEMICAL ABSTRACTS.

Direct indication of metal corrosion by current-density measurements and its practical significance. F. TÖDT (Z. Ver. deut. Zucker-Ind., 1929, 79, 1—16).—From theoretical considerations confirmed by experiments it is shown that if the corroding metal is

joined to a more noble metal, *e.g.*, platinum, to form a galvanic cell in the corroding medium, then, if the rate of diffusion of oxygen to the surface of the latter metal is the same as that to an equal surface of the corroding metal and the course of the anodic reactions is unchanged by increasing the current density, the current flowing between the two metals is a measure of the amount of the less noble metal lost by corrosion from a surface equal to that of the more noble metal; this amount may be calculated directly from Faraday's law. Tests with several common metals under varying conditions met with in practice have shown that the above conditions are generally fulfilled; naturally any circumstances which alter the rate of corrosion, *e.g.*, film formation, are reflected in a change in the current density at the electrodes, and, conversely, a change in the current density indicates changes in the mechanism of the corrosion, *e.g.*, film formation or breakdown, passivity, etc. Some examples of the use of the method in testing the corrosive action of sugar syrups are given.

A. R. POWELL.

Continuous indication of the content of dissolved oxygen [of salt solutions] and of the protective action of sodium hydroxide solution [on the rusting of iron] by current measurements. F. TÖDT (Z. Ver. deut. Zucker-Ind., 1929, 79, 680—695).—The current density at the electrodes of various metal couples in salt and alkali solutions has been measured at various temperatures. In potassium chloride, sodium hydroxide, and sodium carbonate solutions the current density increases with rise of temperature to a maximum at 100°, but after a short time (1—10 min.) at this temperature it decreases almost to zero; the same effect is produced by addition of sufficient sodium sulphite. These results are attributed to the removal of oxygen from the solution (*cf.* preceding abstract). The protective action of alkaline solutions ($p_H > 10$) on iron is due to the formation of passive films, and addition of chlorides causes breakdown of these films, but they may be reformed by increasing the p_H of the solution; all these phenomena may be followed more closely by measurements of the current in the iron-platinum cell than by the weight-increment method.

A. R. POWELL.

Properties of cast silicon steel. E. H. SCHULZ and F. BONSMANN (Stahl u. Eisen, 1920, 50, 161—168).—The mechanical properties of cast silicon steels containing 0.14—0.18% C, 0.7—1.2% Mn, and 0.7—1.3% Si have been compared with those of a steel having the same tensile strength and containing 0.39% C, 0.45% Si, 0.81% Mn, and 0.11% Cu. The silicon steels had a higher elongation, reduction in area, and impact strength than the plain steel; thus, for equal yield points, the silicon steels had a 60% greater elongation and an 80% greater impact strength. The silicon steels behaved similarly to the plain steel at higher and lower temperatures, but in all cases their ductility was greater. The resistance to fatigue (repeated impact or alternate bending) of silicon steels was also greater than that of the plain steel, but its resistance to corrosion was less; addition of 0.3% Cu to the silicon steels, however, made them more resistant to corrosion than the plain steel without affecting their satisfactory mechanical properties. The effect of various heat treatments on the

mechanical properties of the steels tested is shown graphically and photomicrographs of the structure are reproduced.

A. R. POWELL.

Determination of oxygen in iron alloys by reduction in hydrogen. H. PETERSEN (Arch. Eisenhüttenw., 1929—1930, 3, 459—472; Stahl u. Eisen, 1930, 50, 170—171).—In the usual method of determining oxygen in steel by heating in hydrogen and collecting the water formed, low results are obtained owing to part of the oxygen being removed from the steel as carbon monoxide. This error is the greater the higher is the temperature used, and is appreciable even when the metal contains only 0.09% C. Experiments have been made with various catalysts under varying conditions to effect complete conversion of the oxygen into water by the Sabatier-Senderens reaction: $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$; complete conversion was obtained by passing the gas mixture, containing less than 4% CO or CO_2 , over broken porcelain impregnated with metallic nickel containing 10% of thoria and heated at 250°. By the use of a high temperature (about 1250°) in the reduction, all the oxygen, even that present as silica, is removed from the steel provided that sufficient carbon is present, hydrogen alone taking little part in the reduction of the more refractory oxides. The results obtained by the modified hydrogen reduction method are reasonably accurate, but the method is not so convenient or reliable as the hot-extraction method.

A. R. POWELL.

Determination of small amounts of titanium in alloy steels. H. MATHESIUS (Chem.-Ztg., 1930, 54, 134—135).—The following procedure is recommended for use in cases where chromium, aluminium, and copper are also present. The filings (5—10 g.) are dissolved in hydrochloric acid, nitric acid is added, and the solution filtered. After extraction of most of the iron with ether the copper present is removed with hydrogen sulphide, the excess of the latter boiled out, and the liquid boiled with sodium hydroxide and peroxide. The precipitate is dissolved in sulphuric acid, tartaric acid and ammonia are added, and the residual iron and manganese removed with hydrogen sulphide. After filtration, the tartaric acid in the filtrate is destroyed by evaporation with nitric and sulphuric acids, and the titanium precipitated with ammonia and weighed. This method affords a value only for the titanium alloyed with the iron; should a determination of the titanium also present as nitride, carbide, and oxide be required, the solution of the steel in nitric and hydrochloric acids must be evaporated with 15—30 c.c. of 1:1 sulphuric acid to strong fuming. Then 100 c.c. of concentrated hydrochloric acid are added, and the solution is boiled and filtered; the filtrate will contain the whole of the titanium originally present in the steel, and is further treated as described above.

H. F. HARWOOD.

Special zinc-, silicon-, and antimony-aluminium bronzes. A. SÉVAULT (Compt. rend., 1930, 190, 431—434).—Zinc (1—10%) is without influence on the transformation points of aluminium bronzes (10.08—12.8% Al) and dissolves in the α - or γ -constituent, or in both, with the production in the latter case of an alloy, the hardness of which increases after tempering, but returns to its original state after recovery at 450°. With more

than 14.5% Zn and less than 8% Al a special constituent is formed which is permanent after successive temperings, and which in conjunction with the α - and γ -constituents produces an alloy softened by high-temperature tempering. A special constituent is formed when the silicon content of aluminium bronzes is greater than 1.85%, and when the Al/Si ratio is greater than 4.6. In its presence the hardness of the ($\alpha + \gamma$)-alloys decreases with high-temperature tempering, and increases in its absence. Antimony below 4.5% and an Al/Sb ratio greater than 3.3 produces no special constituent, and free antimony is found when this ratio is below 6. The ($\alpha + \gamma$)-antimony alloys have maximum hardness after tempering at 650°.

J. GRANT.

Analysis of chromite and determination of chromium. I. MAJDEL (Arh. Hemiju, 1930, 4, 8—13).—The ore (1 g.) is fused with six times its weight of sodium peroxide in a nickel crucible, the heating being prolonged for 2 min. after fusion has taken place. The melt is extracted with water, the whole boiled for 15 min., filtered, and the filtrate made up to 500 c.c. Chromium is determined volumetrically in one aliquot portion of the solution, and in another aluminium is determined by evaporation to dryness with hydrochloric acid and alcohol, filtration from separated silica, and precipitation of the aluminium and chromium hydroxides by ammonia. Iron may be determined in the residue obtained from the solution of the melt by dissolving this in hydrochloric acid, removing silica by evaporation, and precipitating the ferric hydroxide twice by ammonia. Silica is best determined in a separate portion after fusion with sodium peroxide in an iron crucible. The author considers the usual method of separation of iron and aluminium from a chromate by treatment with ammonia or ammonium carbonate to be untrustworthy. Even after three precipitations up to 30% of the total chromium may be left in the precipitate, whilst appreciable amounts of aluminium pass into the filtrate.

H. F. HARWOOD.

New technical alloys of calcium. J. MEYER and R. GORALCZYK (Z. angew. Chem., 1930, 43, 149—154).—A number of alloys of calcium with magnesium, aluminium, and zinc are described. They are greyish-white powders impermanent in air and moisture. Alloys with high calcium contents ignite spontaneously when warmed in moist air. The most stable alloys are those with aluminium. On treatment with water the calcium is dissolved, leaving the alloying metal. The reaction with water is not vigorous enough to allow of the use of the alloys for dehydrating alcohol. Physical data and particulars of the volume of hydrogen evolved by the dissolution of 1 g. of alloy in dilute hydrochloric acid are given. The velocity of evolution is of more practical importance in connexion with porous light cement and for reduction purposes. The rate of dissolution is accelerated by the presence of chlorides and decreased by nitrates. The influence of the cation is only slight. Quantitative data for reaction velocities with water and acids of the different alloys are given. The alloy 65Ca:35Zn is considered suitable for admixture with cement, for which purpose a too rapid reaction is not desirable. The reducing effect of these alloys on various

compounds was determined by measuring the hydrogen evolved from water to which 1 g. of the substance to be reduced had been added. In most cases the reducing action was only slight (much less than with sodium), and was not much increased by using the alloys as amalgams. Calcium-zinc alloys are more effective than calcium-magnesium alloys owing to the lower reaction velocity with water. C. IRWIN.

Variations in hardness of metals and alloys resulting from cold-working. GUICHARD, CLAUSMANN, and BILLON (Compt. rend., 1930, 190, 112—114).—The increase in hardness caused by the cold-working of ten metals and alloys used for coinage has been investigated by cold-rolling sheets of the completely annealed metals and determining the relation between the deformation (calculated as $D = 100[E - e]/E$, where E is the initial thickness and e that reached at any stage) and the Brinell hardness. The results are expressed by curves which show that after a preliminary rapid increase in hardness a limit is practically attained when $D=80$. Results are independent of whether the thickness is diminished by cold-rolling, cold-hammering, or by both processes combined. The hardest metals examined were pure nickel and copper-aluminium bronze (8.8% Al), followed by 900-fine gold and bronze (Cu 94, Sn 4.6, Zn 1.4%), and then in order by copper-nickel alloy (25% Ni), silver-copper alloys, copper, and silver. Coins show hardnesses far below the limit for their constituent alloys. C. A. SILBERRAD.

Coinage metal: forgery detectable without analysis. M. CHIKASHIGE and S. UENO (World Eng. Congress, Tokyo, 1929, 2 pp.).—Ternary alloys containing 63—71% Ag, 28—36% Zn, and 0.5—5.5% Cu have a homogeneous β -structure at high temperatures and a duplex ($\alpha + \beta$)-structure at the ordinary temperature. The hardness of quenched alloys with a β -structure is about double that of the same alloy with a duplex structure. The quenching temperature varies from 340° for the 64:35:1 silver-zinc-copper alloy to 650° for the 70:28:2 alloy. Forgery of alloys of this type used for coinage could, it is suggested, be detected by heat-treatment and hardness determinations at temperatures above and below the transformation point. A. R. POWELL.

Working of electrolytic copper. K. BERNHOEFT and W. WUNDER (Z. Metallk., 1930, 22, 27—29).—Good castings are essential for obtaining sheets and wire free from flaws; the metal must be pure, the mould surface smooth, and the casting temperature and mould temperature suitably adjusted. Even with correct casting conditions only about 95% of the ingots yield good sheet or wire. Minute hair-cracks in the ingots cannot always be detected before rolling, but in the finished sheet they produce blue spots or feathering. These defects are particularly noticeable when the hair-cracks are transverse to the direction of rolling. During heating prior to rolling air penetrates the cracks and cupric oxide is formed; this becomes reduced to cuprous oxide by the pressure of the rolls at the high temperature, and these areas then contain more cuprous oxide than corresponds with the eutectic, so that the metal becomes

brittle in these parts and hence feathers or opens out with severe work. A. R. POWELL.

Electrolytic refining of copper, using complex salt of cuprous chloride. N. KAMEYAMA and T. NODA (J. Soc. Chem. Ind., Japan, 1930, 33, 11—12 B).—Arsenious acid added to the electrolyte, up to 2.4% of the copper in solution, yielded a cathode copper containing not more than 0.002% As. Using an anode copper containing 0.08% As, the arsenic found in the cathode was equally low. The greater part of the arsenic was deposited as anode slime. C. IRWIN.

Early use of the metals. T. A. RICKARD (Inst. Metals, Apr., 1930. Advance copy. 43 pp.).—Historical, mainly devoted to non-ferrous metals.

Treatment of waste gases. KERSHAW.—See I. **Ignition of powdered zinc etc. with calcium hydroxide.** GEUTHER.—See VII. **Furnace gases and glass enamels.** GEISINGER and BERLINGHOF.—See VIII.

PATENTS.

Smelting of ores to metal, matte, and slag. H. SKAPPEL (B.P. 324,902, 3.8.28).—The ore is smelted with such additions of sodium, zinc, manganese, or alkaline-earth sulphides, or materials which produce these sulphides, that the resulting matte has a mean heat of formation of more than 25 kg.-cal./g.-atom of contained sulphur. The fluxes added are so adjusted that the final slag contains a maximum of 15% FeO, whereas the speiss formed, in cases where cobalt, nickel, arsenic, and antimony are present, contains an excess of iron and is therefore relatively free from lead. A. R. POWELL.

Roasting of pyritic slimes. METALLBANK U. METALLURG. GES. A.-G., Assees. of H. KLENCKE (G.P. 458,604, 3.1.26).—Fine pyritic material, e.g., that obtained in flotation practice, is mixed with coarser roasted material and the mixture is roasted and sintered by drawing compressed air through the mass. A. R. POWELL.

Core and mould for cast iron etc. C. AUER (Aust. P. 109,181, 17.11.25).—A pasty mass of gypsum, sand, sulphur, and water is compressed into the desired shape under a pressure which removes all the superfluous water, leaving a damp mould or core ready for the casting operation without further drying. A. R. POWELL.

Treatment [annealing] of metal sheets. E. and E. E. GRIFFITHS (B.P. 324,679, 31.7.28 and 21.5.29).—One or each of the side walls of an annealing chamber carries a travelling trolley from which carrying arms extend into the kiln. Metal sheets are suspended from the arms by, e.g., curving the sheet at the top to form a hook. The return path of the arms may be arranged to be on the outside of the kiln. C. A. KING.

Annealing. H. M. ROBERTSON (B.P. 324,627, 26.10.28).—The products of combustion are withdrawn from near the bottom of an annealing chamber and returned without any admixture of air to the upper part of the chamber. C. A. KING.

Manufacture of iron and steel. VEREIN. STAHLWERKE A.-G. (B.P. 295,022, 26.7.28. Ger., 6.8.27).—

Molten steel or iron is deoxidised successively with a series of reducing agents, each of which is more powerful than the preceding. Thus the series may be ferro-manganese, -silicon, -aluminium, -titanium, -beryllium, but any one or more of these may be omitted or replaced by a similarly acting substance. In this way the metal is obtained almost free from oxide inclusions or dissolved oxygen and does not exhibit the phenomena of ageing and blue-brittleness.

A. R. POWELL.

Production of chrome-iron sponge. H. G. FLODIN (B.P. 300,637, 26.10.28. Swed., 17.11.27).—Iron ore, together with sufficient carbon to reduce the iron oxide to metal, is briquetted with chromium ore, with sufficient ferrosilicon to reduce the chromium oxide to metal. The briquettes are heated at 800–1100° until all the iron oxide is reduced to metal and the carbon completely eliminated, then at 1150–1300° to effect reduction of the chromium oxide, the resulting chromium alloying with the iron previously formed to produce a sintered alloy which is suitable for addition directly to molten iron in the manufacture of chromium steels.

A. R. POWELL.

Welding flux [for iron-chromium alloys]. W. B. MILLER, Assr. to OXWELD ACETYLENE CO. (U.S.P. 1,741,031, 24.12.29. Appl., 27.1.26).—The flux consists essentially of a borosilicate glass containing 5–50% SiO₂, 20–65% B₂O₃, and 15–30% of a base. A preferred composition is that obtained by fusing 3.5 pts. of anhydrous borax with 1 pt. of silica.

A. R. POWELL.

Acid-resisting alloy. H. J. HENBREY and C. J. M. THORNHILL (B.P. 324,009, 11.10.28).—White alloys containing 20–75% Fe, 5–20% Cr, 5–20% Cu, 10–50% Ni, 1–10% Ag, and up to 2% Mn are claimed.

C. A. KING.

Flotation process [for copper sulphide ores]. S. P. LOWE and H. T. KOENIG, Assrs. to R. H. CHANNING, JUN. (U.S.P. 1,741,030, 24.12.29. Appl., 19.1.27).—Addition of a cinchona alkaloid to the alkaline pulp prevents interference of colloidal slimes in the froth flotation of copper sulphide ores. The process requires a smaller amount of oil, and gives a higher grade of concentrate and a poorer tailing than the ordinary method.

A. R. POWELL.

Flotation process [for complex ores]. H. T. KOENIG, O. A. FISCHER, E. F. HAFEEY, and A. B. CLAMPITT, Assrs. to R. H. CHANNING, JUN. (U.S.P. 1,741,028, 24.12.29. Appl., 21.9.27).—In the selective flotation of copper-zinc-iron sulphide ores, small quantities of a solution of gum arabic or other gum are added to modify the action of the various reagents. Thus the copper is floated with pine oil, xanthate, and 1.5 lb. of gum arabic/ton, and the zinc with the same reagents after addition of copper sulphate, both operations being conducted in the presence of lime (1 lb. per ton of ore). After the zinc and copper have been removed, micaceous material may be floated away from the iron pyrites by the same reagents, using more gum and more lime.

A. R. POWELL.

[Copper-nickel] alloys. C. PHILIPPOSIAN (B.P. 325,131, 4.3.29. Switz., 3.11.28).—The alloys contain 65–75% Cu, 18–22% Ni, 5–15% Zn, 0.01–0.2%

Al, 0.01–0.2% Mg, 0.01–0.08% Si, 0.01–0.02% Mn, and 0.01–0.1% Cd. The preferred composition is 73.009% Cu, 19.704% Ni, 6.891% Zn, 0.098% Al, 0.098% Mg, 0.049% Cd, 0.029% Si, and 0.029% Mn.

A. R. POWELL.

Reduction of zinciferous materials. C. W. LE PLASTRIER (Austral.P. 9107, 29.8.27).—A porous charge of zinc-bearing material and reducing agent is smelted in an externally heated reducing chamber connected with a condenser and having an additional opening below the level of the escape outlet for the gases from the condenser, so as to allow ingress or egress of gas from the chamber to control the draught in the stack to which the exhaust gases pass. This opening, in the case of vertical retorts, may be the discharge opening for removing residues from the retorts.

A. R. POWELL.

Elimination of the iron contained in bauxites or other aluminous ores. U. B. VOISIN (B.P. 306,094, 14.2.29. Fr., 15.2.28).—Ferruginous bauxite or clay is heated at 150–500° in a current of hydrogen chloride with or without the addition of a small quantity of chlorine but without a reducing agent. The gases evolved are passed through a tower filled with pumice through which sulphuric acid is passed, whereby the hydrogen chloride is regenerated. If the ore contains ferrous compounds it may be roasted in the air prior to chlorination.

A. R. POWELL.

Deforming a crystalline aluminium body. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 324,900, 1.8.28).—Aluminium rods having a macrocrystalline structure are rolled to one quarter of their original thickness and annealed at 600° for 20 min., whereby a single-crystal structure is obtained.

A. R. POWELL.

Ternary and polynary aluminium alloys. METALLBANK U. METALLURG. GES. A.-G. (G.P. 456,343, 18.12.21).—The alloy comprises aluminium (>50%) with two or more of the metals silver, nickel, cobalt, chromium, and manganese and more than the usual amount of silicon which is present in ordinary aluminium alloys. The total content of heavy metal in any case exceeds 2%.

A. R. POWELL.

Improving the resistance to corrosion of magnesium alloys. I. G. FARBENIND. A.-G. (B.P. 316,208, 14.3.29. Ger., 25.7.28. Addn. to B.P. 287,450; B., 1929, 60. Cf. also B.P. 305,197; B., 1929, 857).—Corrosion of the alloys by the action of water is inhibited by adding about 1% of dichromate to the water.

H. ROYAL-DAWSON.

Pre-treating etching plates of magnesium alloys for photomechanical printing purposes. I. G. FARBENIND. A.-G. (B.P. 316,207, 7.3.29. Ger., 25.7.28).—Prior to the application of the copying layer the polished surface of photomechanical plates is treated with abrasive powders which are made up with a solution of an alkaline dichromate; such a solution is used also for washing the plates.

C. A. KING.

Treatment of shavings and scrap of light metal [magnesium] and alloys thereof. J. A. GANN, Assr. to DOW CHEM. CO. (U.S.P. 1,739,717, 17.12.29. Appl., 8.11.26).—The material is agitated with a flux, consisting

of magnesium and alkali chlorides, at a temperature just below the m.p. of the metal or alloy; the crucible is then further heated until the whole melts and the purified metal is cast from below the slag.

A. R. POWELL.

Solder for aluminium-magnesium alloys with a high magnesium content. J. F. LOTZKAT (G.P. 457,592, 22.3.25).—The solder consists of tin, zinc, lead, and cadmium in the proportion 50:30:10:20. The joints have the same tensile strength as the remainder of the alloy.

A. R. POWELL.

Flux for soldering. G. BRUNHÜBNER (G.P. 457,024, 16.10.25).—A mixture of a borate with oxalic acid or of an oxalate with boric acid in equimolecular proportions is claimed. If desired, the solder in the form of a powder can be mixed with the flux.

A. R. POWELL.

Flux for tinning etc. W. H. H. PLATT (B.P. 324,611, 4.7.29).—The flux consists of 1 pt. by wt. of sodium fluoride, 2 pts. of anhydrous zinc chloride, and 6 pts. of ammonium chloride.

H. ROYAL-DAWSON.

Treating lead alloys. G. W. THOMPSON, Assr. to NAT. LEAD CO. (U.S.P. 1,740,752, 24.12.29. Appl., 12.8.24).—Lead alloys may be freed from arsenic, tin, and antimony in succession by agitating the molten metal with fused sodium or potassium hydroxide, to which a sufficient quantity of sodium nitrate is added to convert the impurities into sodium salts.

A. R. POWELL.

Lead bearing metal. O. GRÜNBAUM (Austr.P. 109,161, 30.6.26).—The alloy consists of lead with up to 5% Na and a quantity of zinc equal to 0.47–1.5 times the weight of sodium present. The bearings are claimed to have good mechanical properties and to be unaltered by the atmosphere.

A. R. POWELL.

Treatment of material containing tin. S. TAMARU and Y. KOIZUMI (B.P. 324,685, 3.9.28).—Tin may be extracted from low-grade tin ore or tailings by heating the material together with lime or magnesia and a small quantity of a reducing agent, *e.g.*, carbon or zinc, at 850°, preferably in a neutral or reducing atmosphere, and subsequently leaching the product with a dilute acid or alkali. Addition of sodium chloride to the charge improves the extraction.

A. R. POWELL.

Manufacture of anti-friction alloys. A. E. RICARD and L. DANIEL (B.P. 324,206, 3.1.29).—Prior to incorporation of the metals of higher m.p. (tin and antimony), the molten lead, heated to above its m.p., is treated with about 5% of a mixture of an alkali (sodium) chloride, ammonium (or potassium) oxalate, and potassium hydroxide, and energetically stirred.

C. A. KING.

Improvement of coinage alloys. METALBANK U. METALLURG. GES. A.-G. (G.P. 455,252, 2.5.23).—The coins, after stamping, are treated in known manner to produce a structure consisting of a single crystal or of a few very coarse crystals. Forgery is thus rendered difficult.

A. R. POWELL.

Metal cleaning. G. JONES (B.P. 324,026, 7.9.28).—A mixture for removing tarnish from silver or other metals consists of an electropositive metal, *e.g.*, pul-

verised magnesium, zinc, an acid or acid salt, *e.g.*, citric or tartaric acid or their acid salts, and an abrasive. Copper oxide or an essential oil may be incorporated to combine with or mask hydrogen sulphide generated from the film of sulphide.

C. A. KING.

Hard metal compositions [cobalt-tungsten carbides]. BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of F. C. KELLEY (B.P. 303,751, 7.1.29. U.S., 7.1.28).—Tungsten, cobalt, and carbon powders are intimately ground together with 2% of a resin and sufficient acetone to give a plastic mass, which is pressed into rods in a hydraulic press; the rods are placed in a graphite tube in a hydrogen furnace, heated first at 100–150° to harden the resin, then at 350° to carbonise it, and finally at 1375° for about 1½ hrs., and pressed between tungsten blocks at this temperature until forged down to the desired size. The resulting bars are suitable for the manufacture of dies or cutting tools.

A. R. POWELL.

Production of an alloy consisting of carbide of tungsten or molybdenum and of a lower-melting metal or metalloid. F. KRUPP A.-G. (B.P. 310,885, 1.5.29. Ger., 2.5.28).—An alloy of tungsten or molybdenum and a metal of lower m.p., after being melted down with carbon, is pulverised and sintered.

C. A. KING.

Cleaning of liquid mercury. BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of B. L. NEWKIRK (B.P. 302,319, 14.12.28. U.S., 14.12.27).—Mercury is freed from minute particles, not wetted by mercury but forming a skin on it, by passing the liquid in a tortuous path through a container packed with ⅜-in. steel balls, which thereby become coated with the film of impurities. The container is divided into several compartments, the overflow from one to the next being below the surface of the mercury so that portions of the film which rise to the surface in one chamber are not carried to the next.

A. R. POWELL.

Printing plates and processes for use in the production thereof. H. RENCK (B.P. 324,576, 26.3.29).—Copper printing plates are electroplated with nickel or other metal, which repels mercury, the required parts of the coating are then protected with a suitable resisting material, the nickel is removed from the other parts by electrolysis, and the exposed copper is coated with mercury or an amalgam.

A. R. POWELL.

Recovery of metals in electric furnaces. E. A. A. GRÖNWALL (Swed.P. 60,264, 4.9.24).—The combustible gas produced by the reduction of the ore in the electric furnace is returned to the furnace and there burnt with the necessary air. The heat generated results in considerable economy of electric current.

A. R. POWELL.

Electrothermal production of zinc. NORSK HANDELS- OG INDUSTRIELABORATORIUM A./S., Assecs. of F. THARALDSEN (Swed.P. 60,684, 19.9.23. Norw., 23.9.22).—The resistor of the furnace consists of a slag containing 15–25% Fe and 45–60% SiO₂.

A. R. POWELL.

Attachment for electric zinc furnaces. B. RAEDER (N.P. 43,741, 3.6.22).—A mixing device in which the ore and reducing agent are mixed and preheated is provided above the melting chamber.

A. R. POWELL.

Electrodeposition of metals. IMPERIAL CHEM. INDUSTRIES, LTD., and J. HOLLINS (B.P. 324,589, 24.4.29).—A diaphragm for use in the electrodeposition of metals comprises a series of layers of purified fabric wound round a perforated mandrel; between the various layers is placed a thin film of anode slime produced from the same type of anode as that used in the process. The diaphragm is used to surround the cathode so as to prevent access of particles of anode slime thereto.

A. R. POWELL.

Electrolytic precipitation of metals. U. C. TAINTON (U.S.P. 1,739,772, 17.12.29. Appl., 26.1.26).—For the separation of small quantities of a metal from a solution by electrolysis, a salt of a metal higher in the electrochemical series is added, and the conditions of electrolysis are so adjusted that this metal is deposited in a spongy form on the cathode. By vigorous agitation of the solution the sponge is detached from the cathode and precipitates the desired metal from the electrolyte by simple displacement; in this way the latter metal is more rapidly precipitated than by straightforward electrolysis. The process is particularly adapted for removing impurities from electrolytic zinc solutions and for removing silver and gold from cyanide solutions.

A. R. POWELL.

Electrolytic deposition of chromium. W. G. POETZSCH (B.P. 299,395, 24.10.28. Ger., 25.10.27).—A lithium salt is added to the usual chromium bath. A suitable solution contains 450 g. of chromic acid, 5 g. of chromium sulphate, and 3 g. of lithium carbonate, hydroxide, or sulphate per litre; bright deposits are obtained with 4–20 amp./dm.² at 30–40°, and the bath has a good throwing power.

A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 309,072, 5.3.29. U.S., 4.4.28).—In cadmium plating from an acid bath the anode comprises a cadmium plate and a lead plate the ratio of the areas of which is 70:30, so that the cadmium content of the bath is kept constant; the anode current density is 4–30 amp./ft.²

A. R. POWELL.

Production of cast iron of any desired structure. F. GREINER (U.S.P. 1,746,467, 11.2.30. Appl., 6.11.26. Ger., 6.11.25).—See B.P. 260,990; B., 1927, 560.

Magnetic alloy. W. S. SMITH and H. J. GARNETT (U.S.P. 1,746,500, 11.2.30. Appl., 7.5.27. U.K., 27.7.26).—See B.P. 279,549; B., 1928, 20.

Apparatus for hardening products of iron, steel, and other ferromagnetic materials. W. HEIDENHAIN (U.S.P. 1,747,934, 18.2.30. Appl., 21.12.27. Ger., 28.12.26).—See B.P. 282,750; B., 1929, 214.

Improving the properties of iron-beryllium alloys. G. MASING and O. DAHL, Assrs. to SIEMENS & HALSKE A.-G. (U.S.P. 1,746,356, 11.2.30. Appl., 5.4.28. Ger., 12.4.27).—See B.P. 288,579; B., 1929, 133.

Treatment of copper-rich materials. S. I. LEVY (U.S.P. 1,746,313, 11.2.30. Appl., 1.3.28. U.K., 10.2.28).—See B.P. 309,269; B., 1929, 525.

Incorrodible aluminium alloy. H. SCHORN (U.S.P. 1,747,796, 18.2.30. Appl., 15.9.27. Ger., 18.9.26).—See B.P., 277,701; B., 1928, 271.

Production of aluminium-silicon alloys free from carbide. C. VON GIRSEWALD (U.S.P. 1,747,197, 18.2.30. Appl., 8.5.26. Ger., 12.5.25).—See B.P. 252,160; B., 1927, 144.

Conveyors for [sheet-metal annealing] furnaces. INTERNAT. NICKEL CO., Assees. of A. S. SHOFFSTALL and H. M. BROWN (B.P. 315,344, 7.11.28. U.S., 12.7.28).

Vaporisers (B.P. 315,723).—See I. Sulphuric acid from treated titanium ores (B.P. 309,597).—See VII.

XI.—ELECTROTECHNICS.

Energy without cost. WOLLENBERG.—See I. Energy consumption of steel furnaces. KRIZ and KRAL. Metal corrosion and its measurement. TÖDT. Refining of copper. KAMEYAMA and NODA.—See X.

PATENTS.

Electric furnace. J. R. EVES, Assr. to FALLS ELECTRIC FURNACE CORP. (U.S.P. 1,741,411, 31.12.29. Appl., 20.1.28).—An electric furnace comprises a heating chamber open at its bottom and having a door at its front end; a wheelless carrier for the material to be heat-treated, movable into and out of the furnace and forming a closure for the bottom; upwardly-opening channels containing a sealing material extending round the sides and rear of the furnace and disposed below the top of the carrier; aprons depending from the sides and rear end of the carrier and engageable with the material in the channel in order to seal the joint between the carrier and the furnace; and supporting rails adjustably mounted at the sides of the furnace for sustaining the carrier in a position to break the seal between it and the channels.

J. S. G. THOMAS.

Electrically-heated furnace. SIEMENS-SCHUCKERT-WERKE A.-G. (B.P. 302,223, 29.10.28. Ger., 12.12.27).—Subdivided and separately interchangeable resistances, carried by a refractory framework adapted to rest on the side walls of the furnace, are arranged in a horizontal plane beneath, and independent of, the cover.

J. S. G. THOMAS.

[Glass-melting] electric furnace. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (U.S.P. 1,741,469, 31.12.29. Appl., 9.8.28. Fr., 19.8.27).—A metal, e.g., tin, electrode not attacked by molten glass and fusible at the melting temperature of the glass projects into the furnace through a refractory casing and is provided with a lead-in terminal at its outer end. J. S. G. THOMAS.

Electric furnace [for production of glass, cement, etc.]. C. E. CORNELIUS (U.S.P. 1,741,977, 31.12.29. Appl., 10.11.28. Swed., 9.1.28).—Metal blocks, e.g., of iron, freestanding on the bottom of the furnace serve as electrodes and are surrounded on all sides, except the bottom, by the material to be produced and which forms the resistance. To prevent corrosion of the furnace bottom the lining is cooled with air, water, etc. at a point beneath the electrode blocks. J. S. G. THOMAS.

Electrical induction furnace. N. R. DAVIS, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 323,561, 9.10.28).—An earthed metallic screen formed of strips of non-magnetic conducting material, arranged so as to

minimise the production of eddy currents in the screen, is placed between the inductor coil and the charge, and/or between the furnace casing and the inductor coil, so that electrically conducting masses associated with the furnace are not subjected to a high potential derived directly from the inductor coil of the furnace.

J. S. G. THOMAS.

[Drying out the lining of] electric induction furnaces. N. R. DAVIS, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 324,789, 8.1.29).—A conducting cage or basket having constricted portions is enclosed within the lining so that eddy currents developed in the cage ultimately melt the cage and make it sufficiently discontinuous to prevent circulation of eddy currents during the ordinary operation of the furnace.

J. S. G. THOMAS.

Induction furnaces heated by high-frequency coils. HERAEUS VACUUMSCHMELZE A.-G., and W. ROHN (B.P. 324,869, 1.5.29).—Coils are strengthened by refractory insulating bars fixed to the turns of the coils by bolts passing through holes in the bars.

J. S. G. THOMAS.

Thermostatic controlling means for electrically heated ovens etc. N. DENNES, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (METROPOLITAN-VICKERS ELECTRICAL CO., LTD.) (B.P. 323,574, 13.10.28).—A thermostatic element, *e.g.*, a bimetallic strip, mounted within an electrically heated oven is operatively associated with a mercury tilting switch outside the oven, and an auxiliary heating element placed near the thermostatic element is connected in series with the oven-heating elements so that the temperature of the thermostatic element rises and falls more rapidly than that of the oven.

J. S. G. THOMAS.

Incandescence cathode for discharge tubes. W. GERMERSHAUSEN (B.P. 299,431, 25.10.28. Ger., 26.10.27).—A refractory metal core, coated with metal by electro-deposition, is wound with a helix of active or activated wire.

J. S. G. THOMAS.

Mercury-vapour lamp. J. R. ROBERTSON and J. VIELLE (B.P. 323,564, 9.10.28).—Mercury, heated and containing a radioactive material, if desired, is projected or allowed to fall in a finely-divided state through the lamp envelope.

J. S. G. THOMAS.

Electric rectifying device. M. LAZARUS (U.S.P. 1,741,319, 31.12.29. Appl., 12.8.29).—A copper-silver alloy containing about 25–40 oz. of silver per ton of copper, is coated with a layer of copper and silver oxides having a surface film of copper and silver, or tin, etc. In forming the rectifier the metal employed is heated in an atmosphere containing ozone in excess of that commonly occurring in the atmosphere.

J. S. G. THOMAS.

Photo-electric cell. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 323,639, 3.1.29).—A layer of light-reflecting material, *e.g.*, silver, is deposited on the exterior of the envelope of the cell, or on a surface closely surrounding the envelope.

J. S. G. THOMAS.

Electrolytic cells. P. PESTALOZZA (B.P. 324,689, 1.10.28).—The cathode shell is reinforced by an inner and outer network which does not function as a cathodic

element; movement of the electrolyte is reduced by perforated cover plates of inert material and a throttle arranged between the main chamber and a secondary outlet chamber having a surface area substantially smaller than that of the main chamber.

J. S. G. THOMAS.

Electrolytic cells. R. L. MACDONALD (U.S.P. 1,740,659, 24.12.29. Appl., 30.3.28).—A fabric member for feeding electrolyte to the cell is positioned between spaced, perforated electrodes mounted in a casing. One of the electrodes has an outwardly bulged portion which presses the fabric tightly against the other electrode, so that the fabric is anchored in position. Means are provided for removing gases from the cell compartments.

J. S. G. THOMAS.

Apparatus for electrolysis chiefly alkaline chlorides. A. P. H. DUPIRE (U.S.P. 1,741,290, 31.12.29. Appl., 7.12.26. Fr., 16.3.26).—An electrolyte apparatus of the horizontal-diaphragm type comprises a stationary anodic bell fixed on posts, a cathodic vat, having undulating transverse edges, removably secured inside and at the lower part of the bell under the anodes, a cathode fitting over the vat, a diaphragm fitted over the cathode and in the undulations in which the anodes are arranged, and means for supply of current to the anodes and cathode.

J. S. G. THOMAS.

Electrodes [for electrolysis of halogen salts] and their manufacture. C. G. FINK and R. E. LOWE, ASSRS. to BARIO METAL CORP. (U.S.P. 1,740,291, 17.12.29. Appl., 25.8.23).—An alloy of lead and silver, containing a greater percentage of silver than of lead, is coated with a depolariser composed of lead peroxide. If desired, the alloy may contain up to 10% Mn and/or up to 20% Tl.

J. S. G. THOMAS.

Removal of chlorine ions from electrolytic solutions. V. ENGELHARDT, ASSR. to SIEMENS & HALSKE A.-G. (U.S.P. 1,740,165, 17.12.29. Appl., 15.12.26. Ger., 31.12.25).—Metal-bearing, chlorinated electrolytes, *e.g.*, lyes containing zinc sulphate, are electrolysed, employing a silver anode at a voltage sufficient to cause dissolution of silver, for a length of time sufficient to effect combination between silver ions and the chlorine ions in the electrolyte, and at such a temperature that the silver chloride formed is precipitated in a flakey condition.

J. S. G. THOMAS.

[Negative iron electrode for] alkaline storage battery. E. B. MILLER, ASSR. to SILICA GEL CORP. (U.S.P. 1,740,518, 24.12.29. Appl., 4.8.28).—Iron oxide gel is used as the essential active material.

J. S. G. THOMAS.

Improving the efficiency of galvanic battery depolarising compositions comprising manganese dioxide and graphite. I. G. FARBERIND. A.-G. (B.P. 311,272, 22.4.29. Ger., 8.5.28).—A powdered mixture of manganese dioxide, graphite, and, if desired, soot, is ground in a mill until the particles of the respective substances are, as near as possible, of the same size.

J. S. G. THOMAS.

Drying accumulator plates. ACCUMULATOREN-FABR. A.-G. (Austr.P. 109,412, 14.9.26).—The plates are arranged in a cold, airtight vessel and, after the

oxygen of the air contained in the vessel has combined with a little of the finely-divided lead forming the active mass of the plates, these are dried in the residual inert atmosphere at any suitable temperature and of any desired humidity, without oxidation of the residual lead.

J. S. G. THOMAS.

Telegraph cable having very high inductivity. H. SALINGER (G.P. 459,736, 26.10.24).—The inductivity of the cable is made to the greatest extent possible, independent of the current strength in the cable, by surrounding either the whole length of cable, or the turns of individual coils, by a magnetic iron alloy containing 40–50% Ni.

J. S. G. THOMAS.

Apparatus for producing ozone. SOC. ANON. L'AGENCE MÉTALLURGIQUE (B.P. 315,349, 11.3.29. Fr., 12.7.28).—Wire-work armatures made of aluminium or aluminium alloy are separated by a thin sheet of dielectric against which they are pressed by insulating plates having projections in the shape of truncated cones on their inner surfaces, so that channels for the free circulation of air, for cooling purposes, are formed.

J. S. G. THOMAS.

Precipitation electrode for electrical gas purification. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of C. HAHN (G.P. 459,808, 20.4.23).—The electrode surfaces are formed of a number of separate or braided wires so arranged that suspended particles in the gas are precipitated in part upon the face of the electrodes and in part upon pieces of wire arranged in interstices in the electrodes. These pieces of wire are arranged so that their projections upon a plane perpendicular to the direction of flow of current are in juxtaposition. The wires may be helically wound, individual turns of adjacent helices interlocking after the manner of a wire mat.

J. S. G. THOMAS.

[Mounting of electrodes in] apparatus for electrical precipitation [from gases]. F. H. VIETS and C. H. WEISKOPF, Assrs. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,741,352, 31.12.29. Appl., 13.9.27).—Collecting or discharge electrodes, or both, are loosely mounted so that they may be lifted from their supports by pneumatically-operated means which impart a rapid jarring or vibratory motion to the electrodes while free from the supports.

J. S. G. THOMAS.

Purification of monatomic gases. "CLARUM" GLASWARENERZEUGUNGS- & HANDELSGES.M.B.H. (Aust.P. 109,393, 20.11.26).—Monatomic gases containing polyatomic gases and even metallic vapours as impurities are subjected to electric discharges in glass vessels the internal surfaces of which are lined with thin coatings of ultramicroscopic porous materials, e.g., powdered glass or meerschäum, or are etched with hydrofluoric acid. Polyatomic gases are absorbed and tenaciously retained by these linings.

J. S. G. THOMAS.

Electrodes for use in arc-welding. IMPERIAL CHEM. INDUSTRIES, LTD., and J. H. PATERSON (B.P. 323,675, 18.2.29).

[Gas- or vapour-filled] luminous electric-discharge tubes. GEN. ELECTRIC Co., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN (B.P. 315,391, 21.3.29. Ger., 14.7.28).

Photo-electric cell. H. GEFFCKEN and H. RICHTER (B.P. 323,605, 19.11.28).

Photo-electric cells. E. G. O., A. E. M., and K. E. H. PRESSLER (O. PRESSLER THÜRINGER VAKUUMRÖHRENFABR. U. FABR. WISSENSCHAFTLICHER APPARATE) (B.P. 300,917, 19.11.28. Ger., 17.11.27).

Electrical [dry] battery. ANGLO-AMER. CHEM. Co., LTD., and C. M. GOURVISH (B.P. 324,666, 31.10.28).

Primary electric batteries. A. N. HAZLEHURST (B.P. 324,981, 8.11.28).

Discharging liquids (B.P. 305,163). **Revivifying adsorptive materials** (G.P. 459,346).—See I. **Active carbon** (G.P. 459,347). **Determination of moisture** (G.P. 458,927). **Insulating oil** (U.S.P. 1,739,092).—See II. **Saturation of fibrous articles** (U.S.P. 1,738,794).—See V. **Examination of dyed material** (B.P. 325,112).—See VI. **Chromic acid** (U.S.P. 1,739,107). **Lead sulphate** (F.P. 635,134).—See VII. **Recovery of metals** (Swed.P. 60,264). **Zinc** (Swed.P. 60,684). **Zinc furnaces** (N.P. 43,741). **Electrodeposition of metals** (B.P. 324,589). **Precipitation of metals** (U.S.P. 1,739,772). **Electrodeposition of chromium** (B.P. 299,395). **Cadmium plating** (B.P. 309,072).—See X.

XII.—FATS; OILS; WAXES.

Development of rancidity in fats. Z. VON SÁNDOR (Z. Unters. Lebensm., 1929, 58, 375–377).—Development of rancidity in butter, lard, and pumpkin-seed oil kept under various conditions as regards access of air and light for 3 years was studied. In general, all the fats showed the same changes, viz., fall in sp. gr., increase in Reichert–Meissl, Polenske, and acid values, and decrease in iodine value. Access of air greatly augmented the change in the acid and iodine values, and also in the Reichert–Meissl values of lard and pumpkin-seed oil, but it had little effect on the Polenske value and sp. gr. of these, or on the increase in Reichert–Meissl value of butter. The effect of adding sodium chloride to the fats before storage was studied; the results are somewhat inconclusive.

W. J. BOYD.

Neat's-foot oils and greasiness. CUYPERS.—See II.

PATENT.

Manufacture of stable highly chlorinated train oils. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 323,801, 10.10.28).—An exhaustively chlorinated train oil is treated with air or an inert gas until only a faint hydrogen chloride reaction persists, the temperature having been raised to 70° if desired. Air or an inert gas containing ammonia or a like-acting volatile amine is then passed in and a small quantity of a difficultly volatile amine is added. The stable chlorinated train-oil obtained is suitable for use as paint, binding agent, etc.

S. S. WOOLF.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Relation of oil absorption to consistency of pigment-oil pastes. R. V. WILLIAMSON (Ind. Eng. Chem., 1929, 21, 1196–1198).—From determinations of the apparent viscosity of various lithopone and zinc

oxide pastes in linseed oil, it is deduced that no simple relationship exists between oil absorption and consistency for the pigments studied. This is explained by the fact that degree of wetting and soap formation comprise two variables acting in the opposite sense, the relative significance of each altering with such factors as acidity of vehicle. With ground pastes, soap formation is the preponderating influence, since the energy supplied in the grinding process is sufficient to cause the oil to displace adsorbed films of moisture and air, and wet the pigment. Hence the consistencies of ground pastes increase continuously with the acid value of the oil.

S. S. WOOLF.

Significance of formation of lead-oil aggregates ("lead soaps"). W. LAUFENBERG (*Farben-Ztg.*, 1929, 35, 546—549).—The superior durability of lead paints is attributed to the formation of lead soaps therein, and this is shown to be dependent on the basicity of the particular lead pigment present. By storing white lead, red lead, litharge, etc. in linseed oil a series of "lead" oils was obtained containing from 0.12 to 12.4% Pb, respectively. The resistance of films of these oils to water was determined by Hermann's electrical method, and it was shown that increase in lead content corresponds to increase in water-resistance, films containing 8—12% Pb having resistance equivalent to that of tung oil or stand oil. By observations of the weight of films on ageing, increase of lead soap content was shown to cause decrease in the oxidative decomposition of the film and a general slowing down of the ageing process. A comparison of the properties of 16 days- and 1 year-old films of raw linseed oil and linseed oil containing 0.12% and 7.85% Pb, respectively, was made, oxyn-acids, non-oxidised film acids, glycerin and low-molecular decomposition products, and iodine value of oxyn-acids being determined. The results, which are tabulated and discussed, afford further proof that lead-oil aggregates are advantageous to the durability of linseed oil films. The use of basic lead pigments, *e.g.*, red lead, to achieve these advantages is indicated. The relations between composition and degree of lead soap formation of "normal" and "high-dispersion" red leads are described. For red leads of the same type, free monoxide content is a measure of soap formation, but on account of surface action, a "disperse" red lead will form soaps to the same extent as a "normal" red lead containing much more free monoxide. Since a too high content of monoxide militates against stability, a compromise must be sought between improving the film by soap formation and impairing the storage capabilities of the paint. For "high-dispersion" red lead the optimum composition corresponds to a content of 32.5—33.5% of peroxide.

S. S. WOOLF.

Influence of [basic] pigments on ready-mixed paint. E. KINDSCHER (*Farben-Ztg.*, 1929, 35, 549—550).—The tendency to formation of metal soaps induced by the use of basic pigments in oil paints is discussed. By extraction of the vehicles of stored paints and determination of the metal content, it was shown that lead monoxide and red lead are very active in this connexion, whilst white lead, basic lead sulphate, and zinc oxide are much less so. The behaviour of "normal" and "high-dispersion" red leads of various peroxide contents

indicates that particle size as well as chemical composition must be taken into account.

S. S. WOOLF.

Determination of the extent of "rubbing-off" of paints. R. KEMPF (*Chem.-Ztg.*, 1930, 54, 142—143).—A piece of moistened gelatin photographic printing-paper which has been previously fixed (or, if white pigments are under examination, exposed, developed, and fixed so as to give a black ground) is pressed against the painted surface by means of a special metal cylindrical stamp, which permits of a definite known pressure being applied, a pressure of 20 kg. being usually employed. The resulting impressions obtained through the adherence of the loosely-coherent pigment particles to the moist gelatin may be permanently preserved and enable comparative tests to be made of various paints.

H. F. HARWOOD.

Condensation products of phenols and aldehydes. XV. Relation between percentage of *m*-cresol and the quality of the condensation product. T. SHONO (*J. Soc. Chem. Ind., Japan*, 1930, 33, 24 B).—*m*-Cresol should be present in the cresylic acid used for this purpose to the extent of 60—70% for the best results.

C. IRWIN.

Retene and its derivatives. ANON.—See III.

PATENTS.

Extraction of turpentine, pine oil, and rosin from resinous wood. R. C. PALMER, Assr. to NEWPORT Co. (U.S.P. 1,740,115, 17.12.29. Appl., 4.12.26).—Unsteamed resinous wood is subjected to the solvent action of polymerised olefines of b.p. below that of turpentine, *e.g.*, diisobutylene.

S. S. WOOLF.

Graphite anti-oxide paint. J. N. BACON (B.P. 322,779, 13.7.28).—Low-grade amorphous graphite mineral is washed and levigated in suitable apparatus without crushing the foreign matter with which the graphite is associated, and the separated material is dried, pulverised, and calcined for 20 min. at not above 400°. The product is mixed with linseed oil, turpentine, and driers in stated proportions.

S. S. WOOLF.

Colours or stains for graining etc. J. I. BOURKE (B.P. 323,868, 20.11.28).—Such products comprise two or more colouring agents, a paint drier, a linseed oil vehicle, a turpentine solvent (in amount slightly exceeding that of the oil), and a small proportion of terebene. A range of suitable pigments and compositions imitating various woods are cited.

S. S. WOOLF.

Processes of coating and articles produced thereby. BRIT. CELANESE, LTD. (B.P. 298,616, 12.10.28. U.S., 12.10.27).—An undercoat containing a synthetic resin derived from a phenolic substance, *e.g.*, phenol-aldehyde, ketone-phenol-aldehyde, is applied to smooth surfaces, *e.g.*, metal, glass, hardwood, and followed by one or more coatings of a composition containing cellulose acetate or other organic cellulose derivatives. The latter may also be contained in the undercoating.

S. S. WOOLF.

Impregnation of pervious bodies and impregnating solutions therefor. H. WADE. FROM BAKELITE CORP. (B.P. 322,789, 13.9.28).—Pervious bodies are impregnated with a solution consisting of a potentially-reactive phenolic resin capable of liberating ammonia

during its transformation into the infusible state, and furfuraldehyde in quantity at least sufficient to combine with the liberated ammonia. The presence of furfuraldehyde eliminates "bubbling" and improves impregnating qualities in general. S. S. WOOLF.

Production of colloidal white lead [basic lead carbonate]. T. GOLDSCHMIDT A.-G. (B.P. 297,116, 7.9.28. Ger., 15.9.27).—Lead oxide, in the form of an aqueous slime, is treated with carbon dioxide in the presence of alkaline accelerators, *e.g.*, ammonia, hydroxides or carbonates of the alkali metals. [Stat. ref.] S. S. WOOLF.

Manufacture of chrome-green pigment. F. S. LOW, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,738,780, 10.12.29. Appl., 18.2.27).—A mixture of micaceous laminated chromic chloride and finely-divided carbon is treated with superheated steam at 300–450° and then with oxygen at a temperature sufficient for the oxidation of the carbon. S. S. WOOLF.

Manufacture of pencil lead. B. B. GOLDSMITH and H. GROSSMAN, Assrs. to AMER. LEAD PENCIL CO. (U.S.P. 1,738,888, 10.12.29. Appl., 5.8.22).—Suitable components, *e.g.*, china clay and graphite, are deflocculated and mixed, suitable deflocculating agents being 0.5% of caustic soda for the clay and 5% of tannin or 1% of magnesium oleate for the graphite. The mixture is flocculated, *e.g.*, by an acid agent, and marking elements are formed therefrom. S. S. WOOLF.

Improvement of printers' ink. P. BROWNSEY, G. MADDERS, J. A. GRIMSHAW, and H. REDFEARN (B.P. 322,873, 20.11.28).—Printers' ink is maintained in good condition and its printing qualities are improved by the addition of a mixture of stearine, white wax, lard, potassium bromide, and boiled oil, the requisite proportion of the last-named varying with the roughness and porosity of the paper to be printed. S. S. WOOLF.

Treatment [cleaning] of copal or similar gums. J. C. VAN DER TAELEN, and SOC. COLONIALE ANVERSOISE SOC. ANON. (B.P. 319,263, 14.1.29. Ger., 19.9.28. Addn. to B.P. 309,254).—Metallic filings, powder, etc. are used as the scouring agent instead of sand, as in the prior process. The metal is subsequently separated from the copal magnetically. P. E. L. FARINA.

Manufacture of lacquers and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,780; 8.8.28. Addn. to B.P. 301,133; B., 1929, 104).—In the process previously described, the treated oils may be replaced wholly or in part by gum varnishes, a non-volatile or difficultly volatile plasticiser of b.p. above 150° being added; the urea-formaldehyde condensation products may be replaced by similar products obtainable from thiourea (or a mixture of urea and thiourea) and formaldehyde or from dimethylolurea; and nitrocellulose may be replaced by another cellulose ester or ether. S. S. WOOLF.

Manufacture of oil lacquers and varnishes. I. G. FARBENIND. A.-G. (B.P. 297,034, 10.9.28. Ger., 12.9.27).—The incorporation of aromatic hydroxycarboxylic acids, *e.g.*, β -hydroxynaphthoic acid, or derivatives thereof, into oil lacquers and varnishes improves their

flowing qualities and permits the use of considerably larger quantities of pigment than the normal.

S. S. WOOLF.

Varnishes, lacquers, and like coating compositions. BRIT. CELANESE, LTD. (B.P. 296,675, 3.9.28. U.S., 3.9.27).—Compositions comprising cellulose acetate or other organic derivatives of cellulose, a phenol-aldehyde resin of the "resol" type prepared by condensing phenol and formaldehyde in substantially equimolecular proportions in the presence of an acidic catalyst (hydrochloric or phosphoric acid, stannous chloride), a volatile solvent liquid, and, if desired, natural resins, plasticisers, dyes, or pigments, are claimed. S. S. WOOLF.

Resinous compositions and varnishes made therefrom. BAKELITE CORP. (B.P. 324,025, 9.7.28. U.S., 7.7.27).—A phenol is heated with a fatty oil, *e.g.*, tung oil, and a non-phenolic acid resin other than a "glyptal" resin, *e.g.*, rosin, Manila copal. An active methylene-containing substance is then added in quantity sufficient to combine with the phenol and the mixture is heated until the reaction is complete, giving a viscous liquid or hard, tough solid soluble in common oil-varnish solvents. (Cf. B.P. 293,453; B., 1930, 204.) S. S. WOOLF.

Synthetic resins and their manufacture. BRIT. CELANESE, LTD. (B.P. 299,065, 20.10.28. U.S., 21.10.27).—1 G.-mol. of a ketone-phenol condensation product, *e.g.*, that from acetone, is condensed at above 100° with less than 2 g.-mols. of an aldehyde, *e.g.*, formaldehyde, in the presence of an acid catalyst, *e.g.*, phosphoric, boric, hydrochloric, or sulphuric acid, until a lower layer solidifying at 50–60° is formed. The latter is separated and refined, the resin produced being suitable for use in cellulose acetate lacquers. S. S. WOOLF.

Manufacture of synthetic resins and products containing the same. BRIT. CELANESE, LTD. (B.P. 299,066, 20.10.28. U.S., 21.10.27).—Ketone-phenols, *e.g.*, β -(bishydroxyphenyl)propane, are condensed with ketones in presence of strong acid, *e.g.*, hydrochloric acid, giving water-resistant resins suitable for use in lacquers. The isolation of the ketone-phenol is not essential; *e.g.*, suitable quantities of phenol and acetone may be reacted directly. S. S. WOOLF.

Manufacture of artificial resins. SOC. CHEM. IND. IN BASLE (B.P. 302,737, 21.12.28. Switz., 21.12.27).—Hydrophobe resins are produced from urea or its derivatives and formaldehyde or a polymeride thereof by conducting the resinification process for a sufficient time in presence of free hydroxyl ions, a substance capable, under these conditions, of forming with formaldehyde or with urea-formaldehyde condensation products resins insoluble in water being added at the beginning of the condensation or later. S. S. WOOLF.

Urea-formaldehyde condensates. E. G. BUDD MANUF. CO., Assees. of L. SMIDT (B.P. 294,254, 19.7.28. U.S., 21.7.27. Cf. B.P. 294,253; B., 1929, 1047).—An acid substance, *e.g.*, formic acid, is added to the initial condensation product of formaldehyde and urea, the water accompanying the process being removed subsequent to or at an intermediate stage of the addition of

the acid material. Thiourea may be added prior to the removal of the water. Preferred proportions are: 2 mols. of formaldehyde, 1 mol. of urea, and 0.2 mol. of thiourea. [Stat. ref.]

S. S. WOOLF.

Manufacture of aldehyde condensation products. KUNSTHAERZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 301,798, 6.11.28. Austr., 5.12.27).—1 Mol. of urea or a derivative of urea reacts in acid solution with less than 2 mols. of formaldehyde or other aldehyde, so that, relative to the quantity of urea introduced at any instant and not yet combined, an excess of free aldehyde is present at each stage of the process, *i.e.*, the rate of addition of urea is progressively reduced. The acid process may be interrupted or preceded by a neutral or alkaline stage.

S. S. WOOLF.

Synthetic gummy or resinous material and its manufacture. CANADIAN ELECTRO PRODUCTS CO., LTD., Assees. of F. W. SKIRROW (B.P. 295,322, 1.8.28. Can., 9.8.27. Addn. to B.P. 280,246; B., 1929, 144).—Softer products are obtained by the reactions described in the prior patent when the proportion of aldehyde is in excess of 15% of the weight of ester employed. The presence of sulphur inhibits the reaction, whilst iron and copper are detrimental and must be avoided in the apparatus used.

E. B. HUGHES.

Manufacture of aldehyde-amino-resinous compounds. SCOVILL MANUF. CO., Assees. of R. W. BELFIT (B.P. 292,912, 15.6.28. U.S., 25.6.27).—Lacquers, plastics, moulding compounds, etc. are prepared by the addition of a hydroxy-aromatic acid, *e.g.*, salicylic acid, or a derivative containing a hydroxy-aromatic group, to a condensation product of the urea-aldehyde type, together with suitable solvents, plasticisers, natural resins, and nitrocellulose lacquers as desired.

S. S. WOOLF.

Manufacture of linoleum [with pattern or background in relief]. LINOLEUM MANUFACTURING CO., LTD., and A. A. GODFREY (B.P. 325,297, 20.11.28).

Azo-dye pigments (B.P. 302,599).—See IV. Train oil (B.P. 323,801).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Conditions affecting the vulcanisation of rubber.

III. Some properties of rubber against solvents.

Y. TOYABE and K. FUKUNAGA (J. Soc. Chem. Ind., Japan, 1930, 33, 7—9 B).—The addition of a small proportion of ethyl alcohol, acetone, or nitrobenzene increases the solubility of unvulcanised rubber, containing 20 vol.-% of zinc oxide, in benzene or gasoline. Inorganic bases, *e.g.*, potassium hydroxide or ammonia, or certain organic accelerators, also cause an increase of the solubility. Whereas the final gel skeleton of rubber disperses only with difficulty in benzene, it disperses easily in a mixture of benzene and alcohol. Using a range of mixtures of benzene and alcohol in different proportions it is found that maximum peptisation of rubber occurs at the same composition as the maximum molecular polarisation.

D. F. TWISS.

Caustic sludge. LORENZ.—See V.

PATENTS.

Treatment of rubber and like material. GOODYEAR TIRE & RUBBER CO., Assees. of A. M. CLIFFORD (B.P. 305,195, 8.11.28. U.S., 2.2.28).—The age-resisting properties of rubber are enhanced by the addition of the reaction product of a quinol and an amine. By effecting the reaction in the presence of a dehydrating agent, but without a solvent, *pp'*-dihydroxydiphenylamine and *pp'*-diphenylphenylenediamine are obtained from quinol and aniline; these products do not interfere with the vulcanisation process. Reaction products of diphenylamine and *p*-aminophenol are also mentioned.

D. F. TWISS.

Production of micro-porous rubber. K.D.P., LTD., Assees. of H. BECKMANN (B.P. 309,575, 12.3.29. Ger., 14.4.28).—In the production of micro-porous rubber from rubber latex (cf. B.P. 240,430; B., 1925, 1000), the formation of the coherent continuous coagulum, necessary as an intermediate stage, can be ensured even with concentrated latices by the addition of albuminous or albumin decomposition products, especially casein, before or with the coagulant.

D. F. TWISS.

Impregnation and/or coating of fabric with rubber. DUNLOP RUBBER CO., LTD., and G. W. TROBRIDGE (B.P. 324,664, 25.10.28).—A mould or former is covered with fabric, and the latter is wetted with water, or with a stabilising solution such as ammonia, or with a volatile coagulating and/or dehydrating agent such as acetone or alcohol, before being dipped in a concentrated and/or compounded aqueous dispersion of rubber. Alternatively, fabric, treated as described, may be caused to pass over a length of stationary or travelling band, *e.g.*, of metal, immersed in the dispersion. Setting of the deposit after removal from the bath is effected in known manner.

D. F. TWISS.

XV.—LEATHER; GLUE.

Hydration of animal skin by the volume-change method. II. Effect of cure on hydration. III. Effect of temperature and time period upon hydration of skin during soaking and liming. E. R. THEIS and H. A. NEVILLE (Ind. Eng. Chem., 1930, 22, 64—66, 66—69; cf. B., 1929, 405).—II. Samples of well-cured and poorly-cured skins were placed in the improved dilatometer (cf. following abstract) and the hydration was determined. Maximum hydration was attained by the poorly-cured skin in 4 hrs., and by the well-cured skin in 24 hrs., after which the hydration diminished. By soaking the hides in water and lime-water, respectively, it is shown that the diminution in hydration was due to bacterial and enzyme activity and was completely checked by immersing the hide in lime-water containing excess of lime. The potential hydration capacity of the skin was diminished by post-mortem changes. The greatest ultimate hydration was produced by saturated solution of potassium chloride and much less hydration by sodium sulphate.

III. The hydration of skin increased with lowering temperature, which is in direct contrast to its effect on swelling. It was diminished to a greater extent

by bacterial action at higher temperatures. Hydration of the skin by lime liquors was less at higher temperatures. Less hydration was observed in skins which had been soaked in old soak liquors, and their ultimate hydration in the lime liquors was less than for skins soaked in fresh water. The hydration of hide in the lime liquors steadily increases, but the swelling may decrease. The swelling of the hide at different p_H values must be due partly to hydration and partly to osmotic effects. The ultimate hydration of hide in liming was diminished by the presence of magnesia in the lime liquor. The hydration of hide was diminished by certain concentrations of acid; this is attributed to the lower hydration of the protein ions as compared with the protein molecules. D. WOODROFFE.

Some properties of gelatin. I. Hydration of gelatin and its relation to swelling. H. A. NEVILLE, E. R. THEIS, and R. B. K'BURG. **II. Method for determining transition temperatures of gels and sols.** H. A. NEVILLE, E. R. THEIS, and C. T. OSTWALD (Ind. Eng. Chem., 1930, 22, 57—60, 60—62).—I. Experiments with gelatin flakes were made in a simple dilatometer, consisting of a bottle with a ground-glass stopper, through which was sealed a calibrated capillary tube. The final contraction of the system, and hence the degree of hydration of the gelatin, increased as the temperature was lowered. Equilibrium was attained more quickly at higher temperatures. Measurements of the hydration of such a gelatin-water system at different p_H values show that hydration and swelling are not parallel effects. Hydration was a maximum at approximately the p_H of minimum swelling. The swelling of gelatin at the isoelectric point cannot be due to osmotic force, but may be attributed to hydration, i.e., to the compression of a shell of liquid about the particles. Completely hydrated gelatin, when placed in solutions of different p_H values and the expansion of the system determined dilatometrically, showed greatest expansion, i.e., minimum hydration, at p_H 2. The acid solutions dehydrated the gelatin, and since swelling is found to be accompanied by an increase or a decrease in the total volume of the gelatin solution, it is concluded that the two effects must be largely independent. The hydration of limed hide was reduced by immersing it in weak solutions of alum or zinc chloride.

II. For measurement of the gelation and melting temperatures of gelatin solutions a modified dilatometer was used consisting of a pyrex tube drawn into a spiral of 2 mm. diam., and attached at its lower end to a U-tube of 15 c.c. capacity; the other end was joined to a separating funnel and stopcock. The apparatus and gelatin solution after being brought to a constant temperature, e.g., 45°, in a water-bath are filled with the solution to slightly above the top of the spiral, the stopcock is closed, mineral oil is poured into the tube above the spiral, and a rubber stopper fitted with a capillary tube is inserted. The oil rises in the capillary tube, and its position is followed by a scale behind it. The water-bath is provided with a stirrer and means for rapid heating or cooling. When the gelatin in the spiral forms a gel, its adhesion to the glass prevents the registration of further contraction of the larger volume of the gelatin in the oil manometer. This point is called

the gelation temperature, and the m.p. is obtained similarly by heating the water-bath. A sudden change in slope of all cooling curves was noted at about 36°, termed the transition temperature. It is attributed to the change of gelatin sol form A into gel form B. Typical results for transition, gelation, and melting temperatures are given for gelatins of different Bloom strengths. D. WOODROFFE.

PATENTS.

Production of water-soluble condensation products [tanning agents]. I. G. FARBENIND. A.-G., Assees. of A. VOSS (G.P. 456,931, 19.7.23. Addn. to G.P. 408,871; B., 1925, 414).—Zinc chloride and *p*-sulphobenzyl chloride are added gradually with stirring to "Novolak" (prepared by acid condensation of phenol with formaldehyde) maintained at 110—120°; when evolution of hydrogen chloride slackens, the product is run slowly into water and the solution is filtered and concentrated. The syrupy product on dilution and adjustment of its acidity yields a tanning bath giving a soft, full-grained leather. An acidified aqueous solution of the product obtained by heating β -naphthol with "sodium sulphotolyl chloride" under pressure at 170—180° for 6 hrs. serves also for tanning.

L. A. COLES.

Water-soluble condensation products (B.P. 320,056).—See III. **Dyes for leather** (B.P. 306,477).—See IV. **Artificial leather** (B.P. 299,023 and 301,759).—See V.

XVI.—AGRICULTURE.

Classification of soils of Rotorua country. L. I. GRANGE (New Zealand J. Sci. Tech., 1929, 11, 219—228).—A detailed classification is followed by a brief discussion of the incidence of bush sickness in cattle pastured on land poor in iron and possibly in calcium.

E. HOLMES.

Pakihi lands of the Nelson Province. T. H. EASTERFIELD, T. RIGG, and J. A. BRUCE (New Zealand J. Sci. Tech., 1929, 11, 231—241).—Following a description and classification of the "pakihi" or marshy lands of Nelson Province, notes are given on the nature of the soil pan, the feeding value of pakihi rush (*Cladium teretifolium*), and methods of pasture formation and flax (*Phormium tenax*) growing on them. E. HOLMES.

Some peculiar low-lying soils of Central Travancore. T. R. N. PILLAI and V. SUBRAHMANYAN (J. Indian Inst. Sci., 1929, 13, 1—10).—Poor fertility in the soils examined is traced to the accumulation of straw and plant residues under conditions of very imperfect aeration. There results a "locking up" of available nitrogen by the slowly decomposing organic matter, and the development of considerable organic acidity during rapid fungal growth. A. G. POLLARD.

Influence of decalcification and acidity of littoral sands on vegetation. M. HOCQUETTE (Compt. rend., 1930, 190, 514—516).—Examination of a series of successive sandy deposits shows that decalcification increases with age, but that the rapidity of the process varies with the environment of the deposit. There is no marked parallelism between calcium-content variation and acidity, which ranges from p_H 6.5 to p_H 7.9. De-

creased calcium content and decreased acidity leads to the appearance of vegetation not indigenous to the dunes themselves. An account of the calcifuginous species observed and the conditions of their growth is given.

C. W. SHOPPEE.

Reduction of nitrates in arable soils. T. M. ZACHAROVA (Landw. Jahrb., 1929, 70, 311—340; Bied. Zentr., 1930, 59, 51—53).—Seasonal changes in the numbers of denitrifying organisms (*B. stutzeri*) occurring in cropped soils are examined. Maximum numbers occur at two or more periods between June and September, according to the nature of the crop. In general, the numbers of these organisms vary inversely with the nitrate content of the soil, except where there is an absolute nitrate deficiency for the crop. Acid soil conditions check denitrification. Soluble non-oxidisable organic matter in cropped soils does not influence denitrification, except possibly under clover where nitrate assimilation is low. In fallowed land high proportions of easily oxidisable organic matter corresponded with relatively large numbers of denitrifying bacteria. No relationship was observed between the intensity of denitrification and the content of water or soluble phosphate in soils. Dressings of lime, alone or with stall manure, increased denitrification. The general nature of the variations in the numbers of *B. stutzeri* were similar in manured and unmanured soils.

A. G. POLLARD.

Neutralising action of silicates of hydraulic lime on the soil. C. BRIOUX and E. JOUIS (Compt. rend., 1930, 190, 444—446).—The authors' method (B., 1930, 254) has been applied to a product containing 52.6% CaO (total), 23.1% SiO₂ (gelatinous), 4.5% of siliceous sand, 3.8% Al₂O₃, 1.7% MgO, and 2.4% Fe₂O₃. The *p*_H of a non-humic alluvial soil was raised from 5.76 to a constant value of 6.67 in 20 hrs. by addition of 0.5 g. of sample per kg. The *p*_H of a humic grubbed soil was changed from 5.57 to 6.37 after 3 hrs. in contact with 1.5 g./kg. The active lime content varied from 40 to 44%, and corresponded closely with the solubility of the hydraulic lime in contact with water saturated with carbon dioxide for 4—6 hrs. (*loc. cit.*)

J. GRANT.

Microbiological analysis of soils. F. BAUMGÄRTEL and K. SIMON (Landw. Jahrb., 1929, 10, 345—357; Bied. Zentr., 1930, 59, 50—51).—Waters containing much calcium are unfavourable to the growth of *Azotobacter chroococcum* in Beijerinck's cultures. In soils this effect is not due to the flocculation of soil colloids, but is the result of a specific physiological action of calcium bicarbonate.

A. G. POLLARD.

Methods for studying replaceable bases in calcareous soils. P. S. BURGESS (J. Amer. Soc. Agron., 1929, 21, 1040—1044).—Percolation with 0.1N-barium chloride in 68% ethyl alcohol is preferred. The residue on evaporation is taken up in water, the barium being removed as chromate for calcium or magnesium determinations or as sulphate for sodium or potassium determinations, the bases being subsequently separated. Alkali salts, if present, are removed by leaching with water previous to percolation.

CHEMICAL ABSTRACTS.

Use of artificial zeolites in studying base-exchange phenomena. O. C. MAGISTAD (J. Amer. Soc. Agron., 1929, 21, 1045—1056).—Synthetic zeolites are preferred to soils; their behaviour is similar. The base-exchange capacity of a soil varies with reaction, moisture, and other factors.

CHEMICAL ABSTRACTS.

Determination of the base-exchange capacity of soils. W. P. KELLEY (J. Amer. Soc. Agron., 1929, 21, 1021—1029).—A discussion. The author's method is described, and Hissink's method is criticised.

CHEMICAL ABSTRACTS.

Determination of exchangeable hydrogen in soils. F. W. PARKER (J. Amer. Soc. Agron., 1929, 21, 1030—1039).—Determinations (a) by titration to *p*_H 7.0 with barium hydroxide, (b) by leaching with neutral barium acetate solution, and (c) as the difference between the exchange capacity of the soil and its content of exchangeable bases, gave similar results, whilst conductometric titration was unsatisfactory. Acid soils do not contain appreciable amounts of exchangeable iron and aluminium. A method for the determination of exchangeable hydrogen involving the use of barium acetate and ammonium chloride is described.

CHEMICAL ABSTRACTS.

Determination of the "effect values" of soil nutrients in soil strata of different depths. J. SOUČEK (Z. Zuckerind. Czechoslov., 1930, 54, 284—288).—The nutrient values of lower soil layers were examined by means of double culture jars in which the treatment of the upper and lower layers of soil could be varied. The assimilability of the nutrients of the lower soil layers was consistently smaller than that of the surface soils, average proportions being 90% in the case of nitrogen, 70% for phosphate, and 60% for potash.

A. G. POLLARD.

Present-day laboratory methods for determination of the fertiliser requirement of soils. O. LEMMERMANN (Z. Pflanz. Düng., 1930, 9B, 1—17).—A general discussion of a number of accepted methods based on reports of the 2nd Comm. Internat. Soc. Soil Sci., Budapest, 1929; cf. B., 1930, 257).

A. G. POLLARD.

Mitscherlich's method [for determining soil nutrient values] with various crops. J. PÁZLER (Z. Zuckerind. Czechoslov., 1930, 54, 273—283).—Culture experiments showing the fertiliser requirement for a number of crops by Mitscherlich's method are described and the results examined mathematically. Per unit of added nitrogen, increases in yield relative to the maximum varied with the nature of the crop. On this basis plants (with the exception of legumes) may be classified into groups of similar nitrogen requirements. Similar results are obtained in the case of potash, but differences are less marked with phosphates. The significance of these values in the applications of the Mitscherlich process are discussed.

A. G. POLLARD.

Root-solubility of the phosphoric acid of super-neutral, Reform, and Algerian phosphates. C. DREYSPRING, C. KRÜGEL, and R. PANTKE (Superphosphate, 1929, 2, Nos. 4 and 5; Bied. Zentr., 1930, 59, 55—59).—Comparative tests are described and the

relationships between water- and citric-solubility and the assimilability of these fertilisers are discussed.

A. G. POLLARD.

Action of the complete fertiliser "Nitrophoska" in comparison with other fertiliser combinations.

O. ENGELS (Forts. Landw., 1929, 4, 418; Bied. Zentr., 1930, 59, 61—62).—"Nitrophoska" compares favourably with mixtures of simpler fertilisers both in price and efficiency; transport and distribution costs are lower.

A. G. POLLARD.

Fertilisation with carbon dioxide and its importance for the coal industry. SCHULTE-OVERBECK (Brennstoff-Chem., 1930, 11, 28—30).—The increased rate of growth of plants brought about by increasing the concentration of carbon dioxide in the surrounding atmosphere has led to the development of methods for supplying carbon dioxide to growing crops. In Riedel's system the combustion gases produced by burning coke are cooled and washed in water towers, passed over chalk to remove impurities, and then distributed over the surface of the fields through a suitable pipe-system. Development of these processes will not only create a new market for coke, but will also stimulate the demand for artificial manures.

A. B. MANNING.

Combined sprays for destroying the overwintering eggs of the European red mite and apple aphids at the delayed dormant period of the apple tree. T. J. HEADLEE and J. M. GINSBURG (New Jersey Agric. Exp. Sta. Bull., 1929, No. 469, 1—15).—The eggs are destroyed without injury to the trees by oil emulsion with addition of either free nicotine or 5% of crude cresylic acid. The volatility of nicotine is greater from free nicotine solutions than from nicotine sulphate solutions when mixed with either lime-sulphur (1:9) or oil emulsion (3% of oil), and the toxicity to aphid eggs is parallel to the volatility of the nicotine.

CHEMICAL ABSTRACTS.

Lead arsenate studies on cranberry hogs in New Jersey. B. F. DRIGGERS (New Jersey Agric. Exp. Sta. Bull., 1929, No. 480, 1—36).—The acid water did not materially increase the solubility of lead arsenate unless much organic matter was present. Depression of the solubility appears to be due to iron. Injury of the plants is discussed.

CHEMICAL ABSTRACTS.

Field tests with treated seed corn [maize]. T. A. KIESSELBACH (J. Agric. Res., 1930, 40, 169—189).—Treatment of maize seed with the commercial products of mercury, Uspulun, Bayer dust, Semesan, and Merkö, gave partial control of the *Diplodia* seedling disease.

E. HOLMES.

Biological values and supplementary relations of the proteins in alfalfa [lucerne] hay and in corn [maize] and sunflower silage. J. SOTOLA (J. Agric. Res., 1930, 40, 79—96).—From feeding experiments on lambs fed for 10-day preliminary and 10-day experimental periods, it is concluded that the biological value, or percentage retention, of proteins in lucerne hay, maize silage, and sunflower silage were 56, 94, and 67, respectively. The proteins of a mixture of lucerne hay (1 pt.) and maize silage (3 pts.), such as is commonly used, had a value of 81, whilst the value calculated on

the basis of the nitrogen contributed by each was 64. The difference of 17 was due to the supplementary effect of the different proteins. Similarly, the proteins of a 1:3 mixture of lucerne hay and sunflower silage had a value of 62 (calc. 58).

E. HOLMES.

Hydrogen and hydroxyl ions in the ionic layer of suspended particles and dispersed ultramicros. G. WIEGNER and H. PALLMANN (Z. Pflanz. Düng., 1930, A16, 1—57).—See B., 1930, 252.

Peat-forming plants. STADNIKOV and BARYSCHEVA.—See II. **Caustic sludge.** LORENZ.—See V.

PATENTS.

Fertiliser. STICKSTOFF-WERKE A.-G. RUŠE, and V. EHRLICH (Austr.P. 109,402, 6.10.26).—Dry calcium superphosphate is mixed with oiled calcium cyanamide. The oil prevents the formation of dicyanodiamide.

A. R. POWELL.

Fertiliser from calcium cyanamide. E. JOHNSON (N.P. 43,729, 3.7.23).—Carbon dioxide is passed through a suspension of calcium cyanamide, a substance free from nitrogen, and a catalyst, and the mass is dried and ground.

A. R. POWELL.

Non-caking mixed fertilisers containing sodium or potassium nitrate. NORSK HYDRO-ELEKTRISK KVAELSTOFKARTIESELSKAB (N.P. 43,535, 3.9.25).—The fertiliser is obtained by evaporating to dryness a solution containing sodium (or potassium), calcium, and ammonium nitrates.

A. R. POWELL.

Protective material for dusting on to plants. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of K. GÖRNITZ and H. GOEBEL (G.P. 452,459, 12.7.24).—The material consists of the fine dust particles containing unburnt carbon which are carried along with the flue gases of a furnace, mixed with, e.g., arsenates or copper salts, or impregnated with, e.g., nicotine, pyridine, nitrobenzene; non-reacting powdered material may also be added.

L. A. COLES.

Insecticidal composition. W. R. VEAZEY, Assr. to Dow CHEM. Co. (U.S.P. 1,737,132, 26.11.29. Appl. 21.2.23).—The composition comprises an insoluble (lead) arsenate and $\frac{1}{2}$ —2% of an alkali or ammonium ferrocyanide, or a mixture of these with zinc ferrocyanide, as deflocculator.

L. A. COLES.

Utilisation of cellulose waste liquors in agriculture. V. CASABURI (F.P. 611,545, 22.2.26, and Addn. 32,516, 19.8.26).—(A) Insecticides, fertilisers, and the like are incorporated with the waste liquors obtained in the manufacture of alkali-cellulose, the adhesive character of which causes the material to remain on the subjects sprayed. (B) Hemicellulose obtained from carob beans is used for emulsifying insecticides or fertilisers used in spraying plants.

A. R. POWELL.

Retention of nitrogen in liquid manure. J. BRAUNSCHILD (G.P. 458,192, 18.6.25).—The liquid is treated with a small quantity of turpentine or pine oil preferably mixed with a non-drying oil. The oil mixture may be saturated with oxygen or ozone prior to use. Liquid manures so treated show no signs of fermentation or loss of nitrogen after storage for 7 months.

A. R. POWELL.

Calcium cyanamide (G.P. 458,028).—See VII. Sugar-refining waste liquors [as fertiliser] (U.S.P. 1,740,276).—See XVII. Insecticides etc. (B.P. 322,193).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Dextrorotatory substances other than sucrose in [French] beets of 1929. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 40—41).—Beets analysed between Oct. 20 and Nov. 9, 1929, by hot aqueous digestion, had an average polarisation of 15.86, which was 1.09 higher than the percentage of sucrose by the Herzfeld-Creydt method and 0.78 higher than that by Clerget's method. The average polarisation of the molasses obtained during this period was 2% higher than the Herzfeld-Creydt sucrose content. J. H. LANE.

Agostini's [colour] reaction [for detection of dextrose]. J. PIERAERTS and L. L'HEUREUX (Bull. Assoc. Chim. Sucr., 1930, 47, 42—43).—Agostini's reaction (B., 1887, 534) is shown by all the common sugars as well as by soluble starch and certain glucosides. It is most intense if 5 drops of a 1% solution of the sugar are mixed with 5 drops of a 0.1% solution of auric chloride and 2 drops of 5% potassium hydroxide solution. The coloration, purple changing rapidly to deep blue, is deeper and more lasting in the cold than when the mixture is heated to boiling as recommended by Agostini. J. H. LANE.

Extraction of crystallised l  vulose. P. V. GOLOVIN, N. A. BRYUKHANOVA, and A. I. FRIDMAN (J. Appl. Chem., Russia, 1929, 3, 140—142).—Chicory or artichoke was washed and pressed with cold, and then hot, water, the juice being inverted with sulphuric acid, cooled, neutralised with lime to p_H 7.5—8.0, and filtered. The juice containing not more than 10% of sugar was treated with cold milk of lime and the precipitate, after being collected and washed with ice water, was suspended in water and treated with carbon dioxide. After concentration in a vacuum, with intermediate removal of calcium carbonate, crystalline l  vulose was obtained.

CHEMICAL ABSTRACTS.

Determination of the composition of potato starch from the size of the starch granules. G. BREDEMANN and O. NERLING (Z. Spiritusind., 1930, 53, 42—44, and Chem.-Ztg., 1930, 54, 87—88).—The determination is made on the starch extracted from 1 kg. of potato strips which are obtained by dividing individual potatoes into quarters and retaining one portion. After maceration and various sievings the starch is finally passed through a sieve of 0.1—0.2 mm. mesh and dried at 100—110°. By microscopical examination of the starch mounted in olive oil, a field containing over 1000 grains is divided into 20 groups according to the average diameters of the grains. The diameter of grains of the first group are 5 μ or less, of the second 6—10 μ , and of the last 96—100 μ . The number of grains in each group is counted and their combined weight calculated from the weight of a million of such grains, which had been experimentally determined previously by measurement of the variations of the deposition rate of starch grains in liquid according to the magnitude of the grains. From the total weight

of the starch in the field the percentage weight of each group of starch grains is calculated. For the final result the grouping limits according to the size of the diameters are widened, and the percentage weights are recorded for the granules of 1—20 μ , 21—40 μ , and over 40 μ , with an addendum showing the values between 1 and 10 μ , and over 60 μ . C. RANKEN.

Metal corrosion and its measurement. T  DT.—See X.

PATENTS.

Manufacture of sugar. M. S. HERSHEY (U.S.P. 1,740,693, 24.12.29. Appl., 7.6.28).—Clarified raw cane or beet juice is boiled under vacuum to a very thick syrup, containing about 9—10% of moisture, and then forced out of the vacuum pan and simultaneously cooled, agitated, and dried by a blast of air, so as to yield a powdery sugar product containing all the solid matters of the original juice. J. H. LANE.

Manufacture of a sugar product. W. H. DICKERSON, Assr. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,739,064, 10.12.29. Appl., 29.12.22).—Dry, finely-divided sugar, containing substantially all the accompanying constituents of the original juice, is prepared from raw cane, sorghum, or beet juice, after removal of mechanical impurities and colouring matters if necessary, by atomising the juice in a current of hot gas, preferably above 100°, and, when the particles have lost most of their moisture, cooling the gas, preferably to below 65°, so that the particles can be collected as a dry powder. J. H. LANE.

Treatment of waste material [from sugar refineries]. A. G. STILLWELL (U.S.P. 1,740,276, 17.12.29. Appl., 21.2.29).—Waste liquors from sugar refineries or distilleries are converted into a dry, non-hygroscopic product, suitable for use as a fertiliser, by evaporating at a temperature sufficiently high to eliminate hygroscopic constituents, but not high enough to decompose proteins or cause charring. A temperature of 170° is considered suitable. It is claimed that at this temperature glycerin is volatilised in presence of evaporating water.

J. H. LANE.

Synthetic gummy material (B.P. 295,322).—See XIII.

XVIII.—FERMENTATION INDUSTRIES.

Citric acid content of grapes at various degrees of ripeness and the determination of citric acid. A. HEIDUSCHKA and C. PYRIKI (Z. Unters. Lebensm., 1929, 58, 378—381).—Unripe grapes contain 0.040—0.199 g. of citric acid per kg. During ripening the citric acid content remains almost constant, although total titratable acid decreases greatly. As already reported (B., 1928, 312), von der Heide's modification of the Stahre-Kunz method gives low results. A correction of 0.030 g. per litre of wine or must should be added to the citric acid content found thereby. W. J. BOYD.

Lead content of must and wine from grapes treated with insecticide containing lead. E. KIELH  FFER (Z. Unters. Lebensm., 1929, 58, 382—386).—The lead content of must and wines from grapes of the Moselle territory, the vines having received a moderate

application of lead insecticide, amounted to 1.4 mg. per litre of must, 1.1 mg. per litre of wine after the first drawing, and 0.5 mg. after the second drawing. Considerable quantities of lead were found in the yeast. Clarification by means of ferrocyanide removed only an inconsiderable proportion of the lead. In view of these results the use of lead insecticides for vines is inadvisable.

W. J. BOYD.

Sulphiting of musts at vintage. L. F. TELLE (Bull. Assoc. Chim. Sucr., 1929, 46, 529—534).—In the southern vine-growing parts of France and in Algeria it is the general practice to sulphite musts at the time of vintage, but in the more northerly districts, including Champagne, this treatment is usually adopted only in rainy years or when the must is specially liable to infection. There is a fear that it may give rise to a flavour of hydrogen sulphide in the wine, or delay the primary or secondary fermentation, or suppress the malo-lactic fermentation which moderates the acidity of young wines. The author considers these fears groundless if the treatment is properly applied. Only in the free state is sulphur dioxide liable to reduction to sulphide or does it retard fermentation. A must containing 20 mg. of free sulphur dioxide will not ferment, but in presence of 10 mg. fermentation will proceed without any formation of hydrogen sulphide, although the total sulphur dioxide content may amount to 150, 200, or even 250 mg. per litre. The author advocates sulphiting champagne musts in all cases, as a safeguard against maladies and discoloration; the amount recommended in normal years is 70—80 mg. SO_2 per litre, but this may be reduced to 50—60 mg. in very good years and increased to 100—120 mg. in bad years. The sulphur dioxide is most conveniently added in the form of standardised solutions of alkali bisulphites or mixed phosphates and sulphites, these solutions being very stable.

J. H. LANE.

Blue [haze] in Champagne wines. P. MARSAIS (Bull. Assoc. Chim. Sucr., 1929, 46, 534—539).—During the cellar treatment of bottled champagne a haze is sometimes observed which appears bluish when viewed by transmitted light from a lamp, especially if the light is partly obscured by interposition of the spread fingers between the lamp and the bottle. This haze deposits with extreme slowness, and it sometimes does not appear until after the usual sediment has been removed from the bottles. Mazé and Pacottet claim to have isolated a coccus which produces the haze, but other authorities maintain that non-bacterial causes are often responsible, and it is regarded by some as a form of *casse ferrigue*. Strict oenological hygiene is the best safeguard against this and other defects.

J. H. LANE.

Composition of musts and wines of the year 1927 from different varieties of apple and pear of the Moselle region. E. KIELHÖFER (Z. Unters. Lebensm., 1929, 58, 386—389).—The results of complete analysis are tabulated.

W. J. BOYD.

Production of baker's yeast from waste sulphite liquors. E. HÄGGLUND (Papier-Fabr., 1930, 28, 65—68).—A factory has been erected in Finland to manufacture yeast according to the method of Heikenskjöld and Nielsson. The hot, waste sulphite liquor

is run into wooden vessels and mixed with a sludge of finely-ground chalk so that the acidity is reduced to 2.5—3°. The neutralisation is carried further by lime and soda which are added until the alkalinity to litmus is 0.3°. Vigorous aeration of the liquid during the neutralisation process is essential. After settling, the clear liquid is decanted, filtered, and centrifuged. Prior to being seeded into the sulphite-liquor at 30°, the yeast, which amounts to about 20% of the expected reproduction, is prepared by successive inoculation into malt and molasses. Ammonium sulphate, ammonium phosphate, and ammonia are added as nutrients and as regulators of the acidity of the liquid, which is aerated during the whole course of fermentation so that the yield of alcohol is small. The yeast is centrifuged, washed, and pressed, about 22.5 kg. of yeast being obtained from every m^3 of sulphite liquor. The washing of the yeast is important and is effected by water purified by treatment with aluminium sulphate and chlorine after filtration through sand. Yeast suitable for fodder cannot be produced from pure cultures, but if wild yeast is used for seeding, the product may be used for that purpose.

C. RANKEN.

Artificial distillery slop. E. LÜHDER (Z. Spiritus-ind., 1930, 53, 25—26).—A modified "potato soup" is prepared from potatoes which are steamed at a moderate pressure and subsequently mashed at 70—80° with 0.3—0.5% of malt. This amount, which is sufficient to ensure liquefaction but not saccharification of the starch, must not be exceeded. In order that the composition of the artificial product shall approximate to that of normal distillery slop, the liquefied mass is mixed with water and a calculated amount of fodder containing a high percentage of nitrogen. The whole is sterilised by boiling and fed hot to the cattle. It is difficult to manufacture an artificial fodder with exactly the same chemical composition as that obtained from the usual fermentation residues owing to the absence of substances derived from the yeast. Unless the greatest care is taken in its preparation and all vessels are kept sterile, rapid deterioration of the slop takes place and causes sickness among the cattle. Part of the toxic effects is attributed to the presence in the sprouting potatoes of solanin, which is not a constituent of the normal slop.

C. RANKEN.

Physiological selection of ferment by alcohol. L. SEMICHON (Rev. Viticulture, 1929, 71, 85—90, 101—111, 117—124; Chem. Zentr., 1929, ii, 2118).—To produce a pure alcoholic fermentation and to gain other practical advantages in the preparation of wine, the fermentation is commenced in presence of 4% of alcohol.

A. A. ELDRIDGE.

Purification of absolute alcohol. FRITZWEILER and K. R. DIETRICH (Z. Spiritusind., 1930, 53, 27).—Absolute alcohol which has been freed from aldehyde, methyl alcohol, fusel oil, etc. by the azeotropic method still contains traces amounting to 0.1% of the benzene and benzol which are used as dehydrating agents in that process. These traces are completely removed by adding at least 40 g. of activated pine-wood charcoal to every 300 c.c. of alcohol and filtering the mixture 18—20 hrs. later. Unless the charcoal has been acti-

vated by preheating to red heat, the impurities are not adsorbed. Shaking and raising the temperature of the alcohol-wood charcoal mixture give less effective results, but exclusion of the air by preventing the absorption of moisture by the alcohol favours the removal of the impurities. After filtration, the alcohol should be redistilled to remove small traces of resinous substances which may have been extracted from the charcoal by the alcohol. In addition to the benzene and benzol, traces of other impurities, which affect the flavour and odour of the alcohol, are also removed by the charcoal treatment.

C. RANKEN.

Influence of salts on the distillation of alcoholic mixtures. The case of chlorides. C. MARILLER (Bull. Assoc. Chim. Sucr., 1929, 46, 489—496).—Published data on the solubility of calcium chloride in boiling alcohol are too high. The author found that for alcohol of 99.6% by vol. the saturated solution at the temperature of ebullition under 760 mm. pressure contains 16.4% CaCl_2 and the elevation of the b.p. is 7.4° . For alcohol of 95% and 91% by vol., respectively, the corresponding calcium chloride contents are 28.7 and 31.8%; and the corresponding elevations of the b.p. are 10° and 11.8° . The rectification of alcohol is facilitated by the presence of calcium chloride in the liquid undergoing distillation (cf. B., 1925, 733). A form of laboratory rectifying apparatus is described, with a Robert column, by which alcohol of 99.7—100% can be obtained; part of the final condensate is returned to the head of the column after passing through a vessel containing anhydrous calcium chloride. Under similar conditions sodium acetate and sodium hydroxide are much less effective than is calcium chloride; zinc chloride is unsuitable as it produces some ether. Anhydrous calcium chloride can be recovered from solutions by heating to 150° under a vacuum of 700 mm.

J. H. LANE.

Alcoholometry. C. MARILLER (Bull. Assoc. Chim. Sucr., 1929, 46, 497—504).—The history of alcoholometric tables is briefly sketched. The most accurate table is probably that compiled by Stastny and Renz ("Alkoholové Tabulky," Prague, 1927) from the data of Osborne, Kelvy, and Bearce (B., 1913, 303). The author reproduces this table, showing d_{4}^{20} and d_{4}^{15} for percentages of alcohol by wt. and by vol., and tabulates in parallel the discrepancies between it and the official American, Russian, German, Austrian, and French tables. These discrepancies are least in the case of the American table (max. 0.000038), which is also based on the data of Osborne, Kelvy, and Bearce, and greatest in the case of the French table (max. 0.000358).

J. H. LANE.

PATENTS.

Manufacture of aromatic sparkling wines. C. MANCEAU (F.P. 578,297, 9.5.23).—An aromatic extract is added to the wine either before or after fermentation in the bottle.

C. RANKEN.

Production of absolute alcohol. P. LORIETTE (F.P. 578,480, 24.5.23).—Alcohol freed from water and fore-runnings is rectified under pressure, and the heat from the vapour of the alcohol which is distilled

under pressure is used for the dehydration and rectification of the raw spirit.

C. RANKEN.

Destructive fermentation of organic refuse. L. BOGGIANO-PICO (F.P. 570,376, 30.8.23. Ital., 30.8.22).—Compressed air is blown through finely-ground organic refuse sprayed with water and the ammonia in the resulting gases converted into ammonium sulphate by passing the gases over calcium sulphate.

C. RANKEN.

XIX.—FOODS.

Examination of some varieties of Hungarian wheat. A. GÖMÖRY and L. PAP (Z. Unters. Lebensm., 1929, 58, 363—374).—Results of the examination of 23 kinds of Hungarian wheats chemically and by baking tests are given.

W. J. BOYD.

Some properties of wheat flours. E. CAZAUBON (Bull. Assoc. Chim. Sucr., 1930, 47, 44—45).—Using Chopin's method and apparatus for measuring the baking value of flour in terms of the work required to expand a "bubble" of dough to the point of rupture (cf. B., 1921, 316 A), the author has studied the influence of various constituents of flour on the value so determined. Fatty materials tend to lower the tenacity of dough and its baking value, but practically the fat content of flour is too small to render this effect important. A cold-water extract of flour, used in place of water for preparing dough, increases the baking value of the latter. Regarding gluten, Fleurent's theory that there is an optimum ratio between the gliadin and glutenin contents of flour is confirmed, but flours identical in these respects may have different baking values, and the author suggests that the colloidal state of the gliadin and glutenin is a factor of importance.

J. H. LANE.

Laboratory determination of the baking value of wheat flour. C. SCHWEIZER (Mitt. Lebensm. Hyg. Schweiz. Gesundh., 1927, 18, 78—107; Bied. Zentr., 1930, 59, 67—68).—The water-holding capacity of the flour is determined by kneading a prepared dough in the hands until it just fails to adhere, and then ascertaining its water content. Other routine baking tests are described.

A. G. POLLARD.

Change in acidity of bread on storage. L. KARÁCSONYI (Magyar Chem. Fol., 1929, 35, 22—25; Chem. Zentr., 1929, ii, 7865).—The hydrogen-ion concentrations of carbon dioxide-free aqueous extracts of bread (fresh or 48 hrs. old) showed that storage is attended by a diminution of acidity; the change is least in white wheaten, and greatest in rye, bread. The increased acidity sometimes observed is attributed to bacterial action.

A. A. ELDRIDGE.

Physical properties of unpolished and polished rice grains. I. Hygroscopicity of unpolished and polished rice grains and of the husk. II. Drying of unpolished and polished rice grains and of the husk. III. Hardness of unpolished and polished rice grains during drying period. M. KONDO and T. OKAMURA (Ber. Ohara Inst. landw. Forsch., 1929, 4, 127—146, 147—162, 163—171).—I. Unpolished and polished rice grains are equally hygroscopic, although the husk itself is less hygroscopic than either.

II. The presence of the husk does not hinder the rate of drying of the polished grain, which, indeed, dries more quickly than the unpolished grain.

III. The polished grain is more easily crushed but less easily broken than the unpolished grain during the period of drying.

O. J. WALKER.

Polished rice as source of vitamin-B. H. E. MUNSELL (Volksernähr., 1929, 4, 167—168; Chem. Zentr., 1929, ii, 1813).—Both the anti-polyneuritis and -beri-beri (vitamin-*F*) and the anti-pellagra (vitamin-*G*) factors of vitamin-*B* promote growth. For addition of sufficient vitamin-*F* to a diet more yeast is necessary than for that of vitamin-*G*. Polished rice contains little vitamin-*G*, but much vitamin-*F*.

A. A. ELDRIDGE.

Vitamin-B content of cows' milk. P. REYHER (Arch. Kinderheilk., 1928, 84, 55—87; Chem. Zentr., 1929, ii, 1813).—Winter milk contains less vitamin-*B* than summer milk. Human milk in two cases where the children showed signs of spasmophilia caused polyneuritis in pigeons.

A. A. ELDRIDGE.

[Milk] solids-not-fat. G. UXA (Milchwirt. Forsch., 1929, 8, 367—374; Chem. Zentr., 1929, ii, 2122).—The content of solids-not-fat in cows' milk slowly falls from January to April, then rises when green-feeding begins, falls in the summer to a minimum value in August, and subsequently rises again.

A. A. ELDRIDGE.

Detection of adulteration in milk. SZELINSKI (Chem.-Ztg., 1930, 54, 173—174).—The determination of the f.p. is the most reliable physical test for ascertaining if milk has been watered. The f.p. of milk taken from diseased cows is identical with that of normal pure milk, hence the view that this method does not afford a distinction between adulterated milk and that from diseased cows is incorrect.

H. F. HARWOOD.

Butter fat losses in buttermilk. W. H. UDY (New Zealand J. Sci. Tech., 1929, 11, 249—254).—Wide variations occur in losses of butter fat from buttermilk at different factories. Methods of determination of these losses are indicated.

E. HOLMES.

Seasonal variation in the vitamin content of New Zealand butter. J. MALCOLM and E. A. POPE (New Zealand J. Sci. Tech., 1929, 11, 228—230).—New Zealand butter from mixed and Jersey herds was relatively high in vitamin-*D*, but there was a sharp falling-off at the latter end of the lactation period, May, June, and July. In the case of vitamin-*A*, the later butters were frequently richer than the earlier ones.

E. HOLMES.

Enzyme activity of ice-cream improvers. P. H. TRACY and H. A. RUEHE (Univ. Ill. Agric. Exp. Sta. Bull., 1929, No. 333, 483—508).—Of 12 improvers, 11 contained pepsin or rennin, or a mixture of the two. The ripening activity is due primarily to the enzyme. The action of rennin in milk is inhibited by addition of sodium hydrogen carbonate and accelerated by that of calcium salts. When pasteurised at 62° for 30 min., but not when heated under 15 lb. steam pressure, unless calcium chloride is added, milk is coagulated by rennin or pepsin. The thermal stability of the enzymes,

and conditions leading to accelerated action, are discussed.

CHEMICAL ABSTRACTS.

Influence of rennin on the ripening of cheese. Russian Limburg cheese. J. HAWESSON (Lait, 1929, 9, 2—11, 148—161, 358—379, 500—517; Chem. Zentr., 1929, ii, 2122).—The presence of an additional quantity of rennin in the production of the cheese causes a considerable increase in the formation of soluble nitrogenous substances as products of primary degradation of casein. In Russian Limburg cheese these may account for 46% of the total nitrogen. Further decomposition by proteolytic enzymes takes place much more rapidly in cheeses with such additions.

A. A. ELDRIDGE.

Suitability of active charcoal for caffeine adsorption, especially from coffee infusions. F. SARTORIUS and W. OTTEMAYER (Z. Unters. Lebensm., 1929, 58, 353—363).—The adsorptive capacities of various brands of powdered and granular charcoal have been compared. All those examined gave much better adsorption than fluorspar or silica gel. Most of the caffeine was adsorbed from solution almost immediately; on longer contact the absorption curve reached a maximum which was followed by a slight decline. From dilute caffeine solutions almost 100% adsorption was obtained under determined conditions with the best charcoals. For a definite quantity of solution there is a minimum quantity of charcoal giving optimum adsorption. With uninterrupted flow through charcoal filters, temperature and quantity of solution strongly influence the efficiency of adsorption. Increased temperature raises the efficiency at first, but hastens exhaustion of the filter. Adsorption was less from coffee infusions than from pure caffeine solutions. The adsorption did not affect colour or aroma under the given conditions. The use of granulated or powdered charcoal for determination of caffeine is unsatisfactory as the caffeine cannot be completely recovered from the charcoal. Accurate determinations by Gomberg's iodine method (B., 1896, 384) are possible only when excess of iodine is present equal to 25 c.c. of 0.02*N*-iodine solution.

W. J. BOYD.

Feeding value of reed silage. A. ZAITSCHEK (Forts. Landw., 1929, 4, 392; Bied. Zentr., 1930, 59, 69—72).—Results of chemical analyses and feeding trials with fresh, hayed, and ensilaged reeds are recorded, the last-named proving much the most palatable. Only a portion of the total roughage required in a cattle ration could be supplied by reed silage. During ensilage heavy losses in dry weight occurred and the production of volatile fatty acids was considerable.

A. G. POLLARD.

Comparative analyses of cured pork products. C. R. BARNICOAT (New Zealand J. Sci. Tech., 1929, 11, 245—248).—The New Zealand pork tested showed unevenness of curing, too much salt being present in the outer layers, and too little in the inner. A content of 5% of salt is considered satisfactory to ensure proper keeping qualities.

E. HOLMES.

Are lipoids to be reckoned among the fats [in the analysis of organic materials]? B. REWALD (Chem.-Ztg., 1930, 54, 134; cf. B., 1929, 147).—Polemical

against Fincke (Kasett, 1929, Nos. 19 and 21). The view is emphasised that in analyses the lipoids should be included in the fats. H. F. HARWOOD.

Bakers' yeast. HÄGGLUND. **Artificial distillery slop.** LÜHDER.—See XVIII.

PATENTS.

Preservation of eggs. A. MILROY, and EGG PATENTS, LTD. (B.P. 325,045, 11.12.28).—Egg yolk is mixed with egg white which has been stabilised by neutralisation with an acid, *e.g.*, phosphoric acid, or by the removal of certain proteins, *e.g.*, ovaglobulin. Glycerin (2.5–10%) is then added, and the whole dried either partially or completely. E. B. HUGHES.

Edible compound including egg products. J. C. IRWIN, JUN., and S. TRANIN (B.P. 324,637, 26.10.28).—A food product to be used as a substitute for whole eggs consists of an emulsion of dry skim milk, peanut oil, and water, which is then mixed with egg white. E. B. HUGHES.

Preservative for stored potatoes and other roots. C. G. PETTERSSON (B.P. 313,946, 29.10.28. Swed., 20.6.28).—The potatoes etc. are stored in layers 4–8 in. deep and a preservative consisting of a mixture of 75–90% of slaked lime and 25–10% of charcoal is sprinkled over them. E. B. HUGHES.

Purifying food products. E. H. REYNOLDS, ASSR. to REYNOLDS BLEACHER CO. (U.S.P. 1,738,318, 3.12.29. Appl., 24.12.27. Renewed 17.4.25).—A method and apparatus for the neutralisation of surface acidity on cereals, nuts, and similar materials is described. The cereal etc. is covered with a coating of finely-divided calcium hydroxide, stored for 24–48 hrs. to complete the reaction, scoured free from excess alkali, and kept for a further 24 hrs. to enable the gaseous products of neutralisation to escape. Acidity due to residual sulphur dioxide remaining after bleaching processes is said to be removable by this process. E. B. HUGHES.

Recovery of volatile aromatic substances produced in the roasting of coffee, cocoa, etc. H. V. DE CHRISTIANI (F.P. 581,162, 4.8.23, and Addn. 32,553, 20.6.24).—(A) The material is roasted in a vacuum and the vapours evolved are passed through a coil immersed in a freezing mixture. (B) The vapours are fractionally condensed in suitable apparatus. The process effects almost complete recovery of the caffeine evolved during roasting. A. R. POWELL.

Treatment of coffee. D. J. KENNEDY (B.P. 325,039, 30.11.28).—Admixture of 20–25% of cocoa or chocolate to partly or completely roasted coffee is claimed. The coffee berry prior to roasting may be soaked in dilute citric acid for 72 hrs. E. B. HUGHES.

Treatment of waste food. Grading and separating apparatus [for waste food etc.]. F. W. GEE (B.P. 324,116–7, 19.10.28).—Material (hog wash etc.) to be graded is passed through three concentric, conical screens arranged with alternating taper, and at the same time it is washed with hot water to remove the fat, after which the graded residue is pulped, dried, sterilised, and baked. E. B. HUGHES.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation of liquid extract of ergot. F. WOKES and G. K. ELPHICK (Quart. J. Pharm., 1930, 2, 539–555).—Samples of powdered defatted ergot were percolated with 50% alcohol containing hydrochloric acid (2% of concentrated acid) or tartaric acid (1%) and the ergotoxine contents of the extracts were assayed. If 20 g. of the powder is employed, the first 20 c.c. of percolate contain less than 50% of the available ergotoxine. The subsequent fractions progressively decrease in their ergotoxine content, and the first 120 c.c. contain about 97–98% of the total available ergotoxine. The quantity of ergotoxine in the extracts bears no relation to their content of total solids. W. O. KERMACK.

Pectin substances of tobacco. V. BALABUCHA-PORZOVA (U.S.S.R. State Inst. Tobacco Invest., Bull. 59, 1929, 25 pp.).—After removal of carbohydrates from tobacco by washing with water below 50°, boiling water removes hydropectin which can be divided into two principal fractions. The first is soluble in alcohol, constitutes 62% of the hydropectin from fermented, 46% from non-fermented, and 60% from fresh tobacco, and appears to be a product of largely transformed pectic substances. It contains the carbohydrates, *lævulose*, galactose, and pentoses, with impurities. The second fraction, precipitated from the aqueous solution of hydropectin by alcohol, constitutes the true pectic complex or pectic acid. The chief constituent of this is galacturonic acid combined as tetragalacturonic acid to the extent of 55% in the present preparations. The carboxyl groups of galacturonic acid are combined partly with methyl alcohol, and partly as salts with calcium and magnesium. Pectic acid also contains 8.5–11.5% of pentose, probably arabinose. Acetic acid was detected qualitatively. There was little difference in the pectic acid obtained from fresh, non-fermented or fermented tobacco. E. HOLMES.

Composition of cigarettes and cigarette smoke. E. BOGEN (J. Amer. Med. Assoc., 1929, 93, 1110–1114).—The moisture content ranged from 8 to over 50%; an average value for cigarettes as taken from the package was 12%. The inorganic ash is generally inversely related to the nicotine content; the following values for ash and nicotine, respectively, were obtained: American 11.9, 2.50; blends 13.9, 1.90; oriental 15.6, 1.44; denicotinised 15.8, 1.10; West Indian 18.3, 0.86; tobacco-free 8.6, 0%. The composition of the smoke varies with the amount of suction. "Side-stream" smoke collected from the burning end (12 min.) without suction contained: CO₂ 320 c.c., CO 8 c.c., nicotine 13 mg., ammonia 2.2 mg., aldehyde 1.4 mg., furfuraldehyde 0.4 mg., alkali 4.2 c.c. (0.1N). "Main-stream" smoke drawn through the cigarette with intermittent suction (10 min.) contained: CO₂ 21 c.c., CO 3 c.c., nicotine 0.2 mg., ammonia 0, aldehyde 0.4 mg., furfuraldehyde 0, acid 0.5 c.c. (0.1N). Stumps (10 min.) afforded 6.8 mg. of nicotine.

CHEMICAL ABSTRACTS.

Manufacture of cocaine. DUVILUS (Chem.-Ztg.,

1930, 54, 31).—The process used for the large-scale extraction and refining of cocaine is briefly described.

S. K. TWEEDY.

Copper content of drugs arising from sprays.

M. JANICSEK (Magyar gyógyszer. Társ. Ertés., 1929, 5, 317—322; Chem. Zentr., 1929, ii, 1842).—The copper retained by *Melissa officinalis* and *Mentha crissa*, after being sprayed with Bordeaux or Burgundy mixture, was determined.

A. A. ELDRIDGE.

Chemical characteristics of drugs. VII. Microsublimation in D.A.B. VI. L. ROSENTHALER (Apoth.-Ztg., 1929, 44, 744—746; Chem. Zentr., 1929, ii, 1833—1834).—Application of micro-sublimation methods in the following cases is criticised: Cortex Frangulæ, Folia Juglandis, Lichen islandicus, Radix Colombo, Radix Gentianæ, Radix Ononidis, Radix Pimpinellæ, Rhiz. Rhei.

A. A. ELDRIDGE.

Determination of the arsenic content of organic arsenicals. F. E. CISLAK and C. S. HAMILTON (J. Amer. Chem. Soc., 1930, 52, 638—640).—After digestion of the arsenical with sulphuric acid containing potassium hydrogen sulphate, the resulting solution is diluted with dilute sulphuric acid and titrated electrometrically with potassium bromate (cf. Zintl and Wattenberg, A., 1923, ii, 253).

H. BURTON.

Limits for impurities in pharmacopœial chemicals. T. T. COCKING (Quart. J. Pharm., 1930, 2, 570—591).—Tables are given showing the results obtained in the routine analyses of medicinal chemicals for impurities during the years 1914—1929. Suggestions are made for certain changes in the standards of purity demanded by the British Pharmacopœia.

W. O. KERMACK.

Identification of pine-needle extract. H. UHLMANN (Süddeut. Apoth.-Ztg., 1929, 69, 483; Chem. Zentr., 1929, ii, 1833).—A few drops of the extract are diluted with water until colourless, and a few drops of ammonia or potassium hydroxide solution are added, when the pure extract gives a greenish-brown surface zone. Further, when 5 c.c. of a 10% solution of the extract are shaken with 5 c.c. of ether, alcohol (4—5 c.c.) is added until the two layers separate, and a few drops of ammonia solution are added, a light or dark green coloration is observed.

A. A. ELDRIDGE.

Oxidation of pinene in presence of catalysts.

G. DUPONT and J. CROUZET (Bull. Inst. Pin., 1929, Special No., 107—108; Chem. Zentr., 1929, ii, 1792).—In addition to positive and negative catalysts, reversible catalysts exist. The oxidation product of pinene (Engler's hypothetical peroxide) itself accelerates the oxidation. The oxidation of nopinene proceeds more slowly. On addition of a positive catalyst, e.g., cobalt resinate, the velocity of oxidation at first increases and then falls rapidly as the salt is decomposed and cobalt is precipitated. In presence of cobalt resinate light has little, and heat a marked, accelerating effect. Iodine, potassium iodide, and iron, lead, chromium, or nickel resinate are less active positive catalysts. The following are negative catalysts: quinol, pyrogallol, gallic acid, active carbon, ceria, thoria, mercuric acetate,

stannous chloride, titanio acid, platinum-black, reduced copper, freshly-prepared copper resinate, and sulphur. Manganese resinate is the most active reversible catalyst, being at first negative and then positive, the positive catalytic activity falling after a maximal effect has been developed. Addition of oxidised pinene or of fatty oil diminishes or suppresses the anti-oxygenic action.

A. A. ELDRIDGE.

Preparation and analysis of iodinated sesame oil. S. A. SCHOU and A. JACOBSEN (Dansk Tidsskr. Farm., 1930, 4, 25—30).—Lindblom's method for the preparation of iodinated sesame oil (treatment of a chloroform solution of the oil with iodine chloride dissolved in glacial acetic acid) has been modified by passing a current of air through the product at 60—70° for 24 hrs. instead of for 20 min. only. During this operation most of the chlorine is removed as hydrogen chloride, but the final product still contains 0.3 of an equivalent of chlorine for each equivalent of iodine present. The iodinated oil thus obtained is a faintly coloured, neutral liquid which is quite stable even on exposure to air; 90% of the iodine originally employed is recovered in the product. The methods for the determination of the iodine content are briefly discussed.

H. F. HARWOOD.

PATENTS.

Suture. [Surgical thread.] C. H. WATSON, Ass. to DAVIS & GECK, INC. (U.S.P. 1,741,893, 31.12.29. Appl., 13.8.27).—Thread constructed of spun fibrous material, e.g., silk or linen, is steeped in an aluminium salt (acetate) solution, sterilised, and dried.

L. A. COLES.

Anæsthetics. I. G. FARBERIND. A.-G. (B.P. 317,327, 12.8.29. Ger., 13.8.28. Addn. to B.P. 302,337; B., 1930, 121).—A stable, liquid form of tribromoethyl alcohol is obtained by incorporation of a secondary or tertiary alcohol (e.g., 20—30% of trimethylcarbinol or isopropyl alcohol) other than amylene hydrate.

C. HOLLINS.

Manufacture of insecticides, fungicides, bactericides, and veterinary medical preparations. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 322,193, 29.8.28).—The distillation residues from alcohols made according to B.P. 238,319 and 254,819 (B., 1925, 900; 1926, 721) are dispersed in water with the aid of sulphonated oils, butylnaphthalenesulphonic acid, etc. and used for combating insect pests and against mange etc. in animals.

C. HOLLINS.

Production of durable solutions of suprarenal preparations mixed with anæsthetics. M. WOELM (B.P. 322,170, 27.7.28).—For injection purposes the acidity of mixtures of suprarenal preparations with β -diethylaminoethyl *p*-aminobenzoate hydrochloride is reduced by addition of disodium hydrogen phosphate immediately before use. The dry mixture and the phosphate solution, or the three solids and the water, may conveniently be kept in a double phial and mixed when required.

C. HOLLINS.

Manufacture of halogen-substituted aminobenzoic alkamine esters [local anæsthetics]. W. P. WILLIAMS. From SCHERING-KAHLBAUM A.-G. (B.P. 321,968, 23.7.28).—Alkamine esters of aminobenzoic

acids are brominated or iodinated, or a halogenated nitro- or amino-benzoic acid is esterified with an alkamine (or a chloroalcohol, with subsequent amination) and, if necessary, reduced. The following products are described: β -diethylaminoethyl 3-iodo-4-aminobenzoate (hydrochloride, m.p. 176°), 3:5-dibromo-p-aminobenzoate (hydrochloride, m.p. 226°), 3-bromo-5-iodo-p-aminobenzoate, 4-iodo-2-aminobenzoate (hydrochloride, m.p. 205°; tartrate, m.p. 63°), 4-iodo-2-nitrobenzoate (hydrochloride, m.p. 168°), 4-iodo-3-aminobenzoate (sulphate, m.p. 163°), 4-iodo-3-nitrobenzoate, m.p. 198°, and 2-bromo-4-aminobenzoate (hydrochloride, m.p. 146°); β -1-piperidinoethyl 3:5-dibromo-4-aminobenzoate (hydrochloride, m.p. 248°), and 4-iodo-2-aminobenzoate; γ -diethylamino- β -ethoxypropyl 3:5-dichloro-4-aminobenzoate.

C. HOLLINS.

Manufacture of compounds of diphenolisatins [3:3'-di-(p-hydroxyphenyl)oxindoles]. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 321,959, 22.8.28).—A diphenolisatin (1 mol.), dissolved in alkali, is added to a solution of a base of the alkoxy-quinoline or -isoquinoline series (6:8-diethoxyquinoline or papaverine) in dilute acid. Double compounds, without taste or odour, valuable both for atonic and spasmolytic constipation, are obtained.

C. HOLLINS.

Manufacture of quinoline compounds containing an aliphatic amino-substituted side-chain. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 321,974, 21.8.28).—The Friedländer quinoline synthesis from o-aminobenzaldehydes or o-aminophenyl ketones and ketones is applied to aliphatic ketones containing an amino- or alkylated amino-group. 2-Methyl-3- β -diethylaminoethylquinoline, b.p. 159°/2 mm., and its 4-methyl homologue, b.p. 145–150°/1.5–2 mm., are obtained from o-aminobenzaldehyde and o-aminoacetophenone, respectively, with methyl γ -diethylaminopropyl ketone.

C. HOLLINS.

Manufacture of pharmaceutical products. W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER, Assrs. to WINTHROP CHEM. Co. (U.S.P. 1,747,531; 18.2.30. Appl., 24.8.25. Ger., 11.9.24).—See B.P. 267,169; B., 1927, 379.

[Manufacture of] 8-amino-6-alkoxyquinolines. W. SCHULEMANN, F. SCHÖNHÖFER, and F. MIETZSCH, Assrs. to WINTHROP CHEM. Co. (U.S.P. 1,747,532, 18.2.30. Appl., 16.5.28. Ger., 29.4.25).—See B.P. 267,457; B., 1927, 379.

Preparation of benziminazolone-[5]-arsinic acids. W. KOLLE, K. STREITWOLF, and A. FEHRLE, Assrs. to WINTHROP CHEM. Co. (Re-issue 17,599, 18.2.30, of U.S.P. 1,674,368, 19.6.28).—See B., 1928, 625.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Some properties of sodium selenosulphate and its use for the combined fixing and toning of chlorobromide papers. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1929, 16, 320–322).—No complex of definite composition can be isolated either by evaporation or by precipitation with excess alcohol of the saturated solution of selenium in sodium sulphite.

Sulphite is deposited first, followed by selenium, and the remaining solution contains less than the theoretical quantity of selenium for a true selenosulphate, indicating possibly a complex of sodium sulphite and sodium selenosulphate. The solution dissolves appreciable quantities of the silver halides, even of the iodide, but the silver complexes are very unstable, rapidly depositing silver selenide. An attempt to use the selenosulphate in combination with an excess of sodium thiosulphate as a combined fixing-toning bath was not successful owing to the instability of the silver complexes, although the process could be worked on a limited amount of material with very dilute selenosulphate solution and large excess of thiosulphate.

J. W. GLASSETT.

Fate of the iodide in the development of bromo-iodide emulsions. M. L. DUNDON and A. E. BALLARD (Brit. J. Phot., 1930, 77, 90–91).—After development of a partially exposed bromo-iodide emulsion a greater percentage of iodide is found in the remaining halides than is present in the original emulsion, a result which suggests that during the reduction of the halides the iodide enters the solution as iodide ions, which then deposit on unchanged silver bromide to re-form silver iodide. The presence of traces of iodide in the developer is an important factor in the prevention of fog.

J. W. GLASSETT.

Inversion of amateur films. A. SEYEWETZ (Bull. Soc. Franç. Phot., 1929, 16, 317–320).—The following developer is recommended for the first development: potassium metabisulphite 72 g., caustic potash 60 g., p-aminophenol hydrochloride 14 g., water 1000 c.c. Instructions for its use in cases of under- and over-exposure are given.

J. W. GLASSETT.

[Writing on] documents and envelopes. O. MEZGER (Chem.-Ztg., 1929, 53, 965–968, 985–987, 1006–1009).—Methods developed during the past 25 years are fully described, with illustrations. Chemical tests should not be applied until all physical tests have been exhausted. Direct photography in ultra-violet light frequently reveals erased writing, by fluorescence of the sizing of the paper base or of the natural proteins in parchment palimpsests. Mechanical impressions made in the paper itself or in the underlying sheet may be transferred by use of carbon paper and photographed direct. Chemical tests include silver nitrate "print-out" tests for anions (e.g., Cl and SO₄) and tests for cations (principally Fe³⁺); the technique is due largely to Hanikirsch (Z. Unters. Nahr. Genussm., 1917, 74), the most potent test being the production and subsequent photographing (on an ordinary plate) of ferric thiocyanate, by oxidation of the residual iron salts with chlorine water and treatment with the vapour of thiocyanic acid. All these tests may be successfully applied not only to the paper originally written upon, but also to sheets which have lain in contact with it for a few hours, provided that the writing was not more than 2–3 weeks old at the time of first contact. This leads to one method of detecting additions to documents. Other problems studied include the deciphering of postmarks, the detection of superposed pen (or rubber-stamp) and pencil script (including the order in which

the two media were used), and the forged pagination of leaves exchanged for the originals in account books etc.

L. V. CHILTON.

Rubbing-off of paints. KEMPF.—See XIII.

PATENTS.

F Production of screen-positives for use in making photo-mechanical printing surfaces. MEISENBACH, RIFFARTH, & Co. A.-G. (B.P. 302,887, 10.10.28. Ger., 23.12.27).—To facilitate the correction of tone values, the wet-plate screen positive is treated with an aqueous glycerin solution which prevents complete drying of the film, leaving it in a condition suitable for partial etching; the parts to be protected are covered with a varnish, which is finally removed. J. W. GLASSETT.

Manufacture of light-sensitive layers [for diazo-types]. KALLE & Co. A.-G. (B.P. 297,363, 19.9.28. Ger., 19.9.27).—A diazo-compound capable of self-coupling in alkaline media is applied in neutral or acid solution to the support; after exposure under a positive the print is developed with gaseous ammonia. Suitable diazo compounds are those from H-acid, 3-amino-6-N-piperidinobenzoyl-H-acid, and 2:7-aminonaphthol. C. HOLLINS.

Production of photographic images. W. T. S. BECKER, and COLOUR PHOTOGRAPHS (BRITISH & FOREIGN), LTD. (B.P. 323,800, 10.10.28).—The yellow image, formed by exposure of sensitive layers containing silver lactate admixed with metallic oxalates and ferric salts as in B.P. 280,252 (B., 1928, 108), is stabilised by treatment with an aqueous solution of an alkali thiocyanate (preferably of sodium or potassium) which forms a soluble compound, removable by washing, with the insoluble silver and/or iron salts present in the layer, without materially affecting the colour of the image. J. W. GLASSETT.

Photographic reversal process. I. G. FARBERIND. A.-G. (F.P. 632,680, 12.4.27. Ger., 24.12.26).—Positives of sharper contrast are obtained if potassium thiocyanate, which has a lower solubility for silver halide, is added to the primary developer instead of sodium thiosulphate. The thiocyanate may also be used in a clarifying bath prior to reversal. S. K. TWEEDY.

[Submerger means for development in] photography. G. C. BEIDLER (B.P. 325,187, 11.9.29).

Waterproof cellulosic material (B.P. 324,911).—See VI. **Photomechanical printing plates** (B.P. 316,207).—See X.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Nitroguanidine (B.P. 322,427).—See III. **Concentration of nitric acid** (U.S.P. 1,740,549).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Improvements in the chemical purification of potable water and the modern use of activated carbon and earths. O. KOENIG (Gas- u. Wasserfach, 1929, 72, 1065—1072, 1091—1099).—Numerous experiments in the purification of water from the Elbe at the

Magdeburg waterworks are described in which various chlorinating agents, bleaching agents, and flocculating agents were used in laboratory and large-scale tests. Direct chlorination of the water reduced the permanganate and chlorine values, the iron and manganese content, and the bacteria count, but served only to modify the unpleasant odour and taste, and the use of excessive chlorine caused excessive turbidity and interference with the bacterial action during filtration. The use of organic chlorinators of the Duamine type, especially the copper compound, reduced the content of iron and manganese salts combined with humic acid, but worked satisfactorily only at the beginning, activity falling off rapidly with further use. Treatment with activated carbon, alum, and slaked lime in succession resulted in the precipitation of the humic acid and a certain improvement in the physical properties, but the carbon particles became coated with aluminium hydroxide; when, however, the succession was carbon, lime, aluminium sulphate, the humic acid and bacteria contents were not sufficiently reduced. Alum alone or in combination with sulphuric acid and carbon removed manganese completely and iron almost completely, but failed to improve the odour or taste; a preliminary chlorination did not effect any improvement in this treatment. A satisfactory tasteless and odourless water, free from iron and manganese and with a low content of organic substances, was finally obtained by pumping the water directly through granular activated charcoal in closed containers to remove the substances having an unpleasant odour and taste and treating the effluent with fuller's earth and aluminium sulphate with efficient agitation before passing the water to the filters. Purification of the carbon with steam was found to be necessary every 24 hrs. A table is given showing the quantities of reagents used in the various purification processes described above and the cost of treatment, and other tables show the composition and properties of the water before and after the various treatments. Illustrations and diagrams of the plant used at Magdeburg are also included. A. R. POWELL.

Corrosion of water-pipes. ROMBACHER.—See X.

PATENTS.

Filtering and sterilising of liquids. G. A. KRAUSE (B.P. 325,004, 9.11.28).—Efficient sterilisation by the use of an oligodynamic metal is only possible when the metallic surface is kept clean. The sterilising apparatus may be either preceded or followed by mechanical filtration for the removal of suspended matter. In the latter case the material on which the active metal is deposited should be of such size that the bulk of the suspended solids are not retained thereby, and the apparatus containing it of such form that the material may be readily washed (cf. B.P. 279,085 and 293,385; B., 1929, 540; 1930, 84). C. JEPSON.

[Apparatus for supplying chlorine for] purification of water. F. P. CANDY (B.P. 325,161, 16.4.29).

Cyanogen chloride-hydrocyanic acid (U.S.P. 1,738,280).—See VII. **Fermentation of refuse** (F.P. 570,376).—See XVIII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 25 and MAY 2, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat transmission in surface feed-heaters.

J. T. M'INTYRE (J. Roy. Tech. Coll., Glasgow, 1930, No. 2, 241—250).—Results of experiments on the rate of heat transmission through a feed-water heater tube as influenced by variation of the pressure and temperature showed that the change in pressure on either the water or the steam side of the tube had no measurable effect. At velocities of 3.91 and 2.88 ft./sec. the change was just noticeable, probably due to a slight increase in temperature affecting the film on the water side. The range of temperature through which the feed water has been heated had a very definite effect on the heat-transmission coefficients; *e.g.*, when heating through the range 55—130° F., and from 130° to 200°, 210°, 220°, 240°, and 250° F. a progressive value was obtained. If, however, the heating was started at 180° F. through any higher range, the values when plotted all formed part of the same curve. The difference is attributed to the expulsion of dissolved air from water heated from lower temperatures with increase in the thermal resistance of the laminar film of water. When the temperature is sufficiently high no more air is evolved, and the bubbles already formed are carried away in a more rapidly moving current and so no longer affect the conductivity.

C. A. KING.

Steam storage in relation to the peak-load problem in industrial steam plants.

E. G. RITCHIE (J. Inst. Brew., 1930, 36, 121—130).—With ordinary boiler equipment the steam pressure varies with any attempt to carry fluctuating demands for steam. This has the effect of making the adjustment of the fire bed and draught difficult, and from 10 to 20% of fuel is wasted. The shortage of steam results in increased radiation loss per unit of output, and with open vessels the thermal loss may amount to 15% of the total heat consumption. The fluctuating pressure exerts its greatest effect on the rate of output of the manufacturing units, every drop in steam pressure corresponding to a definite loss of output. The difficulty is overcome by the Ruths steam accumulator, which stores steam during periods of low-load demand and gives it out again during periods of peak-load demand when momentarily more steam is required than the boilers are capable of giving. For steam storage the accumulator is only economical if it is used continuously, but it has the advantage of storing steam over the week-end.

C. RANKEN.

Refrigeration by evaporation and its employment in the saltpetre industry.

M. WARNECKE (Caliche, 1929, 11, 298—330).—The theory of refrigeration by evaporation induced by passing a current of air

through a solution is described and data are given relative to the composition of the salts which separate from various types of saltpetre liquors when treated by this process.

H. F. GILLBE.

Formation and growth of crystals. W. E. GIBBS (Inst. Chem. Eng., 1930. Advance proof. 16 pp.).—A lecture. The formation of nuclei and the growth of crystals in undercooled melts and supercooled solutions are discussed and the practical applications indicated.

C. W. GIBBY.

Influence of the proximity of a solid wall on the consistency of viscous and plastic materials.

R. K. SCHOFIELD and G. W. S. BLAIR (J. Physical Chem., 1930, 34, 248—262).—The flow of a plastic material is discussed in the light of the data obtained from clay pastes and glycerol-water mixtures by using the modified Bingham plastometer previously described (B., 1929, 153). When it is assumed that the velocity gradient at any point in a narrow tube through which a plastic material is flowing depends only on the stress at that point, it follows that the mean velocity for a given stress at the wall of the tube is directly proportional to the radius of the tube. Thick soil pastes agree with this, but thinner pastes show marked discrepancies which can be accounted for by assuming that the plastic properties are modified in the immediate proximity of the wall; this gives increased velocity to the bulk of the material. When this increase is first deducted a viscosity constant independent of tube dimensions is obtained.

L. S. THEOBALD.

Simplified Goodrich plastometer. E. KARRER, J. M. DAVIES, and E. O. DIETERICH (Ind. Eng. Chem. [Anal.], 1930, 2, 96—99).—Softness and retentivity (and hence plasticity) of rubber stocks are determined in a simplified form of the power plastometer already described (B., 1929, 761). Full details of the apparatus and method of procedure are given and typical results obtained with it, *e.g.*, plasticity of various rubber stocks, variation of plasticity with temperature, are tabulated and graphed. The effects of varying the size and shape of the test-piece, the load during compression and recovery, the time of compression and recovery, and lubrication of the test-piece are discussed.

S. S. WOOLF.

[Hellig comparator for] p_H control in various industries. H. MAGNUS (Chem.-Ztg., 1930, 54, 108—110).—Reference is made to the importance of control of p_H values in a number of industries. The "Hellig comparator" is an apparatus of simple design in which the colour developed with an appropriate indicator is matched with a standard by observation through a prism.

C. IRWIN.

* The remainder of this set of Abstracts will appear in next week's issue.

Temperature control of exothermic gas reactions.

O. ROELEN (Brennstoff-Chem., 1930, 11, 68—70).—The maintenance of the desired reaction temperature in exothermic gas reactions involves the rapid removal of the excess heat developed and the avoidance of temperature fluctuations within the reaction chamber. The excess heat may be carried away by the gases themselves, the rate of removal being controlled by suitable dilution of the reactants, or by the use of a cooling fluid, *e.g.*, air, metals of low m.p., water under pressure. Temperature fluctuations may be avoided by subdivision of the contact space and uniform distribution of the gases therein. The concentration of the active catalyst may also be varied in the direction of the gas flow, or a certain quantity of the reaction product may be added to the gases in order to control the rate of development of heat. Accurate temperature control is particularly necessary in the Fischer-Tropsch benzene synthesis, in which the heat of reaction is high.

A. B. MANNING.

Reagents for gaseous impurities in technical gases. E. A. J. H. NICOLAS (Chem. Weekblad, 1930, 27, 103—104).—Silica gel impregnated with a solution of an appropriate reagent affords a much more sensitive medium for detecting impurities in a gas than the usual absorbent paper, owing to the increased concentration of the gas and its attendant impurities on the surface of the gel.

H. F. GILLBE.

Graphical solution of problems involving solvent recovery by scrubbing. I. L. MURRAY (Ind. Eng. Chem., 1930, 22, 165—167).—In the n th plate of a scrubber, if X and Z = mol. fractions of the more volatile components in the liquid and gas, respectively, O and W = the mols. of liquid entering and leaving the scrubber, and P = mols. of gas leaving the scrubber, then $Z_n = X_{n+1} W / \{(W - O) + P\} + Z_p P / \{(W - O) + P\}$. The less volatile liquid approximately obeys Raoult's law $Z = P_A X / D$ (D = total pressure, P_A = vapour pressure), whilst the more volatile liquid obeys Henry's law $Z = S P_A X / D$, where S is an experimentally determined variable. A method is given for the graphical determination of S for each value of X from the liquid-vapour curve at the b.p., and an example of the solution of a problem involving the recovery of acetone from air by water-scrubbing is added.

C. IRWIN.

Burner design in furnaces. SEIL and others.—See II. **Creep of steel under stress.** BAILEY.—See X. **Determinations of particle size.** DUNN.—See XIII.

PATENTS.

Annular kiln. H. KOPPERS (G.P. 457,413, 21.11.24).—Fixed walls, provided with regulable apertures, are suspended at intervals along the hearth of the kiln, whilst the firing points on the hearth are bounded on one side by preferably stepped structures and on the other by movable perforated plates which lean against the material being heated. The latter thus never comes into contact with the fuel. S. K. TWEEDY.

[Boiler] furnaces. J. N. D. HEENAN (B.P. 325,390, 18.2.29).—A water-cooled wall or floor for the combustion chamber of a boiler is constructed of steel

tubes upon which are shrunk cast-iron rings formed with longitudinal and circumferential fins. After the erection of the wall it is coated with refractory material by means of a cement-gun, in such a quantity that at least the inner surface is flat. The fins on the metal afford a good key and distribute the heat so that it is transmitted through the whole circumference of the steel tubes.

B. M. VENABLES.

Means for indicating density and change of density [electrical conductivity] of fluid in evaporators, boilers, and other vessels. W. C. CROCKATT, and W. CROCKATT & SONS, LTD. (B.P. 325,277, 16.11.28).—A pair of insulated electrodes are inserted through the wall of a pipe or vessel and connected to a supply of power and to an ammeter, which is graduated in density instead of ampères. The parts of the electrodes in contact with the liquid are coated with non-corrosive material, *e.g.*, platinum.

B. M. VENABLES.

Heat-exchangers. SCHMIDT'SCHE HEISSDAMPF-GESELLSCHAFT (B.P. 307,068, 21.2.29. Ger., 3.3.28).—A heat-exchanger having several groups of tubes arranged in series has headers which are relatively movable to allow for expansion and only one (either the inlet or outlet), is fixed in relation to the casing. The last but one header is connected directly to the outlet header by a small pipe to act as a by-pass for condensate. Other preceding headers may be similarly connected.

B. M. VENABLES.

Heat-exchanger. W. DYRSSEN, ASSR. to BLAW-KNOX CO. (U.S.P. 1,741,225, 31.12.29. Appl., 31.12.27).—Two groups of discs of heat-absorbing material are assembled on and rotated by two parallel shafts which are at such distance apart that the discs will intercalate, the discs of one pile forming the spacers for the other pile. The discs extend through the walls of a single inner conduit to a pair of outer conduits carrying the other fluid, and both fluids flow at right angles to the shafts through the spaces between the discs, the intercalation and extra baffles being placed at the centre of the inner conduit to prevent flow round the edges of the discs.

B. M. VENABLES.

Manufacture of heat-exchange apparatus. E. F. A. D. BECK (B.P. 304,272, 18.1.29. Belg., 18.1.28).—After an apparatus comprising a number of ribs or plates threaded over tubes has been subjected to an ordinary internal pressure, with the object of fixing the ribs in place, a super-pressure is applied which is great enough to swell the tubes in the zones where they are not supported by the ribs. The result is that the tubes are shortened and the upturned edges or other devices which are provided to act as spacers for the ribs are drawn tightly together and the unit becomes a stiff girder instead of having merely the strength of the tubes.

B. M. VENABLES.

Crushing and disposal of furnace residues and other solid materials. ASH CO. (LONDON), LTD. From F. B. ALLEN (B.P. 325,422, 18.5.29).—The material is crushed and delivered to a hopper by means of a pair of rolls, then sluiced periodically through the outlet of the hopper by jets of water, and is water-borne to the place of disposal. The rolls are cooled by water applied

to the parts of their circumference away from the nip, but this water is prevented as far as possible from mingling with the dry material in the hopper and is led direct to the outlet.

B. M. VENABLES.

Continuous mixing machine. H. K. WILDER, Assr. to KELLOGG Co. (U.S.P. 1,741,176, 31.12.29. Appl., 30.10.26).—An annular stream of comminuted solid is allowed to fall into a centrifugally produced mist of a liquid. The apparatus, which is suitable for pre-mixing the feed to a colloid mill, comprises a high-speed, inner, hollow shaft carrying a spraying device for the liquid at its lower end, and a surrounding lower-speed shaft carrying a distributor for the solid matter which works in conjunction with a stationary hopper and sleeve and drops the material just above the liquid spray. The rate of supply of both materials is regulable.

B. M. VENABLES.

Treatment of continuously-moving material. C. R. WEIHE, Assr. to C. WEIHE (U.S.P. 1,742,110, 31.12.29. Appl., 9.7.27).—In processes such as the neutralisation of waste liquors a soluble solid is added to the liquid and the mixture is caused to cascade while the undissolved solid is continuously dragged counter-current by suitable means, thus preventing its escape in the outflowing liquid. An apparatus shown comprises an inclined rotating cylinder with internal helical rib.

B. M. VENABLES.

Continuous settling apparatus. S. I. BOUSMAN, Assr. to DORR Co. (U.S.P. 1,741,498, 31.12.29. Appl., 29.3.28).—The rakes of a traction thickener are caused to sweep the corners of a non-circular tank by permitting the inner end of the rotating truss to reciprocate horizontally upon the central support, the traction motor on the outer end of the truss following a track which agrees with the contour of the tank, *e.g.*, square, with round corners. The central support preferably takes the form of a pillar upstanding from the bottom of the tank, thus obviating any fixed girder and reducing the loss of head and lift of sludge pumps from tank to tank, when there are several in series, because both feed-inlet and clear overflow may be below the traction rail on opposite sides of the tank. The pillar is surmounted by a cap or ring which simply rotates and carries, on trunnions, a frame which has four or more flanged rollers upon which the inner end of the upper boom of the truss or traction arm is supported. The main part of the floor may be level and the rakes for this portion are suspended from the traction arm in the usual manner; the central portion of the floor is sloping (conical), and is dealt with by a number of raking arms suspended from the rotating ring, but the primary object of the reciprocating rakes is to cover the corners, and the sloping floor may be extended even to the wall of the tank, being dealt with still by the rotating rakes. There are no bearings below water-level, and the thick sludge is pumped away from the centre in the usual way.

B. M. VENABLES.

Synthesis of chemical substances. M. POLANYI and S. VON BOGDANDY (B.P. 293,302, 21.6.28. Ger., 2.7.27).—One (liquid) reagent is distilled on to a rapidly-moving surface, upon which the other reagent (or reagents), previously activated by radiation or otherwise,

is made to impinge. Disturbances due to the action of the activating agent on the liquid reagent are thus avoided.

C. HOLLINS.

Filter. K. F. PIETZSCH, Assr. to SELDEN RES. & ENG. CORP. (U.S.P. 1,741,334, 31.12.29. Appl., 7.6.28).—A closed chamber, preferably cylindrical in plan, is provided with a filter bottom and over it are rotated a number of ploughs, or combined ploughs and rollers, so shaped that when running in one direction they compact the cake, and on reversal dig it up and discharge it through doors in the wall of the chamber.

B. M. VENABLES.

Air filter. R. IRVIN (U.S.P. 1,741,367, 31.12.29. Appl., 5.11.26).—A number of louvre-like plates are attached to a pair of endless chains which run vertically and dip into a bath of liquid when turning round the lower sprockets. The air passes horizontally through both runs of plates, which are V- or W-shaped in order to render the course of the air devious.

B. M. VENABLES.

Treatment of liquids with gases. H. and J. SCHEIDEMANDEL (G.P. 460,612, 20.5.24).—The gas is passed into the liquid through narrow, vertical tubes from which only a rapid train of fine bubbles emerge which do not coalesce. Good circulation of the liquid is thus induced.

A. R. POWELL.

Rotary furnace. C. B. WISNER, Assr. to COAL PROCESS CORP. (U.S.P. 1,748,815, 25.2.30. Appl., 13.1.25).—See B.P. 246,118; B., 1927, 431.

Rotary retort. W. R. HUME (U.S.P. 1,748,178, 25.2.30. Appl., 25.9.24. Austral., 12.10.23).—See B.P. 250,302; B., 1926, 520.

Disintegrating machine. E. ROTH (U.S.P. 1,748,679, 25.2.30. Appl., 4.1.26. Ger., 16.2.25).—See B.P. 247,526; B., 1926, 471.

Absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 304,762, 28.12.28. Ger., 26.1.28).

Continuous absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 306,506, 8.2.29. Ger., 22.2.28).

Rotary refrigerators. A.-G. BROWN, BOVERI & COE. (B.P. 301,871, 29.11.28. Ger., 7.12.27).

Mechanical work from coal (Swiss P. 123,928 and 124,135).—See II. Laminated products (B.P. 301,428).—See V.

II.—FUEL; GAS; TAR; MINERAL OILS.

Smokeless fuels. P. WEISS (Chim. et Ind., 1930, 23, 3—14).—After references to anthracite and high-temperature coke, three low-temperature carbonisation processes are described. The Ab-der-Halden process works with a fuel bed 10—15 mm. thick and carbonisation only requires a few minutes. Two rotating hearths are used: in the upper one the coal is dried by contact with combustion gases; the lower one is indirectly heated. The semi-coke is obtained as a powder. Both gas and solid fuel are used for heating. Little or no dust is carried forward with the gas, and the tar, with a free carbon content of 3·5%, does not emulsify with

water. On a large scale it is intended to separate the operations of drying and carbonisation. The K.S.G. process is distinguished by its method of temperature regulation employing a circulation of cooled gases of combustion around the drum. Coal dust is used and the products are 82% of coke, 5.3% of tar, 0.4% of "benzine" scrubbed from the gas, and 85 cub. m. of rich gas per ton. The coke is fairly hard and contains only 0.3% of volatile matter. A process in operation at Nœux carbonises ovoid briquettes with steam superheated to 650°; the exhaust steam is used in turbines. After passing the primary and secondary condensers, the gas is used in the superheaters. A battery of 20 retorts is used which are heated systematically. The internal heating is stated to give accurate control and better yields, and partial hydrogenation of the heavy oil occurs. Tar acids are recovered from the aqueous condensate. Low-temperature tar treated by the usual methods is less valuable than high-temperature tar, and its successful utilisation may depend on hydrogenation under pressure.

C. IRWIN.

Study of certain American coals at temperatures near their softening points. A. M. BALL and H. A. CURTIS (Ind. Eng. Chem., 1930, 22, 137—140).—The softening points of coal samples were determined by measuring the resistance offered by the heated sample to a current of nitrogen, a thermocouple being embedded in the coal. Results varied from 340° to 440°, and are only approximate. It was found that addition of sodium nitrate solution to the coal greatly modified the pressure-temperature curves, and a similar effect was given by prolonged preheating of the sample. The softening temperature was not varied by working under reduced pressure. The temperature at which the surface of a single coal particle under a microscope became distorted agreed closely with the softening temperature determined as above. Experiments are also described on the plasticity of coal at temperatures slightly below the softening point.

C. IRWIN.

Chemical studies of peat. I. Water in peat. G. STADNIKOV (Kolloidchem. Beih., 1930, 30, 197—229).—Determination of the content of moisture, the calorific value, and chemical composition of peat shows that in these respects peat occupies a position between wood and coal. The examination of peat has led to its definition as a conglomerate of bitumen, humic acid and its salts, various other decomposition products of organic matter through the occlusion of air, and undecomposed structural elements of plants, thus differentiating peat sharply from brown coal. Natural peat contains 85—91% of water, part of which can be expressed by mechanical means, the remainder being regarded as colloiddally-bound water. The bound water is lost on drying in the same way as in typical hydrogels. The water remaining in the air-dry material is regarded as water of adsorption and varies between 34 and 43%. A study of the effect of various coagulating agents on aqueous suspensions of peat has shown that colloidal ferric hydroxide is the most effective agent. Relatively concentrated solutions of aluminium sulphate are also effective, and gypsum is effective at lower concentrations.

E. S. HEDGES.

National [Italian] lignites: Fusibility of the ash. C. MAZZETTI (Annali Chim. Appl., 1930, 20, 3—18).—Compositions of a number of Italian lignites and their ashes, and the m.p. of the latter, are given. The value of the ratio $(\text{Al}_2\text{O}_3 + \text{SiO}_2) : (\text{Fe}_2\text{O}_3 + \text{CaO} + \text{MgO})$ gives no indication of the fusibility of the ash, which must be determined directly. The fusibility of the ash and the composition are given for mixtures of the lignites with imported coals.

T. H. POPE.

Obtaining and preparing average samples of solid mineral fuels. G. N. BEZRADECKI (Izvestia Teplotekh. Inst. Moscow, 1929, No. 9, 35—40).—On the basis of experimental data, recommendations are made concerning the sampling of coal and coke.

T. H. POPE.

Heat evolved on the treatment of different varieties of coal with concentrated sulphuric or nitric acid. D. J. W. KREULEN (Brennstoff-Chem., 1930, 11, 41—43).—The temperature rise on adding 0.5 g. of coal to 10 c.c. of 95% sulphuric acid or 50% nitric acid was measured in a calorimeter of the type used by Burstin and Winkler (B., 1929, 667). The fairly regular increase in the heat evolved, from about 30 to 120 g.-cal./g., with increasing volatile content of the coals used, on mixing them with nitric acid, indicated that it was mainly the volatile constituents which were undergoing oxidation. The results with sulphuric acid were much less regular, oxidation with this acid probably involving the other coal constituents as well. Of the banded constituents clarain and vitrain showed the greatest heats of oxidation, and fusain the least: the values for the last-named were similar to those obtained with wood charcoal. The heat of oxidation by acid was increased by a limited preheating of the coal in air, but was again decreased by a more vigorous preheating.

A. B. MANNING.

Distribution of carbon, hydrogen, nitrogen, sulphur, and oxygen in the hydrogenation products of an eocene brown coal. I. VON MAKRAY (Brennstoff-Chem., 1930, 11, 61—64).—By hydrogenation of the coal (2200 g.) in the presence of ferric oxide (330 g.) in a 20-litre autoclave at 470° (initial hydrogen pressure 110 atm.), 45.2% was converted into oil. Of the carbon introduced into the system 53.4% appeared in the oil, 21.7% in the gas, and 24.6% in the residue: 29.8% of the total hydrogen appeared in the oil, 21.8% remained unchanged in the gaseous form, and the remainder was distributed among the water formed, the gaseous hydrocarbons, and the residue. The yield of water of decomposition was smaller than that obtained by direct carbonisation at 500°. 96.7% of the total sulphur content of the coal (4.23%) was taken up by the ferric oxide. 41.0% of the nitrogen of the coal appeared as ammonia and 31.4% as bases in the oil. Of the total oxygen 46.0% appeared as carbon dioxide, 35.6% as water, and 15.5% as phenols in the oil.

A. B. MANNING.

Plassmann process of low-temperature carbonisation. D. BROWNIE (Brennstoff-Chem., 1930, 11, 44—46; cf. Plassmann, B., 1926, 228; 1927, 644; 1928, 395, 631).—Some details are given of the plant at present under construction at Barking. It comprises three units and has a daily throughput of about

66 tons. A washed coking coal of less than 5–6% ash content is to be carbonised; to this may be added 25% of washed anthracite duff of less than 2% ash content. The coke will be sold as a domestic smokeless fuel.

A. B. MANNING.

Nature of the coking process. F. FISCHER (Brennstoff-Chem., 1930, 11, 64–66).—The separation of the oily and solid bitumen from coal and the part played by these constituents in the coking process are briefly described. A distinction is drawn between the primary coking due to the melting and decomposition of the bitumen content of the coal, and the secondary processes due to the condensation of tar on the cooler parts of the charge and the subsequent coking of the mixture of coal and tar so produced (cf. B., 1930, 128). The influence of pressure on the coking process is also briefly discussed (cf. B., 1930, 172).

A. B. MANNING.

Heat economy of coke ovens. K. BAUM (Brennstoff-Chem., 1930, 11, 47–51).—The accurate determination of the heat efficiency of a coke-oven battery is discussed and a method based on the observation of the heat losses from a single oven is described (cf. Baum, Glückauf, 1929, No. 23–25). The efficiency can be increased by a wider use of the regenerative principle, by decreasing the amount of excess air used, as far as that is possible, by improved heat insulation, and by increased uniformity in the carbonisation of the charge. From the efficiency of a single oven (η_1) that of the battery (η) can be calculated from $\eta = \eta_1 V/V_1$, where V_1 and V are the heat consumptions per kg. of coal carbonised for the single oven and the battery, respectively. V alone is no measure of the value of the oven design.

A. B. MANNING.

Determination of the calorific value of solid substances. W. A. ROTH (Brennstoff-Chem., 1930, 11, 46–47).—Paraffin oil is recommended as a secondary standard for the determination of the water equivalent of the calorimeter etc.; moreover, the addition of a few drops to substances which are difficult to ignite or cannot easily be compressed into pellets greatly facilitates their combustion in the bomb. Such a secondary standard ("Tested paraffin oil for calorimetric purposes") of cal. value $10,990 \pm 2$ g.-cal./g. can now be obtained from Schering-Kahlbaum A.-G., Berlin.

A. B. MANNING.

Determination of calorific value of bituminous coals with high volatile matter content. N. TSCHIZHEVSKI and M. VERKHOVITZEV (J. Russ. Met. Soc., 1927, No. 1, 93–101).—The calorific value of coal containing more than 40% of volatile matter can be calculated (± 5 –6%) from the equation $Q = 82C + aV$, where C is the percentage of non-volatile, ash-free, coal-substance, V is the percentage of volatile substances, and a is the calorific value of the volatile substances. Analyses to determine the value of a were made. The equation is inapplicable to brown coal.

CHEMICAL ABSTRACTS.

Determination of nitrogen in organic substances by hydrogenation. V. F. OPOTZKI (J. Chem. Ind., Russia, 1929, 6, 532–533).—For the determination of nitrogen in coal and coke, ter Meulen's method has been modified. An iron combustion tube is employed, and the sample is mixed with equal parts of sodium hydroxide

and carbonate. A burette delivers small quantities of water, as required, to the incoming hydrogen. The results for bituminous coal are considerably higher than those obtained by the Kjeldahl method.

CHEMICAL ABSTRACTS.

Quick and accurate method of determining moisture in coal and coke. A. THAU (Gas World, 1930, 92, Coking Sect., 24–25).—15–20 G. of the sample, of grain size not exceeding 3 mm., are boiled with 100 g. of absolute alcohol, for 2 min., the mixture is filtered, and 25 c.c. of the filtrate are transferred to a test-tube containing 25 c.c. of petroleum. The temperature at which turbidity occurs on cooling is noted, and the moisture present in the original sample is then calculated, using data obtained from standard tests.

C. B. MARSON.

Moisture content of carbon blacks. W. B. PLUMMER (Ind. Eng. Chem. [Anal.], 1930, 2, 57–58).—The results of a series of experiments indicate that the "additional moisture" shown by the xylene-mineral oil method is not in fact present as such in the carbon black, but is formed by the reaction of the mineral oil with the oxygen adsorbed on the black.

S. I. LEVY.

Dry purification of [coal and producer] gas.

(i) E. KAUDELA (Gas- u. Wasserfach, 1930, 73, 110–112).

(ii) G. OFFE (*Ibid.*, 164–165).—(i) The operation of the purification plant at the Leopoldau gasworks near Vienna is described. The plant is arranged in series of four purification chambers, the coal gas and the producer gas being treated in separate series of chambers. The pipe lines are so arranged that it is possible to pass the gas into the individual chambers either in the middle or at the top and bottom, and these directions are changed every few days so that the mass is more efficiently utilised than if the gas is always passed in the same direction. The chambers, which are 12 m. long, 9 m. broad, and 2.2 m. deep, contain six layers of purifying material, each being 16–18 cm. thick. The new material contains about 25% Fe (47.5% of non-volatile matter), 45% H₂O, and 7.5% of other volatile matter, and the completely used material 34% of non-volatile residue, 12% H₂O, 45% S, and 9% of other volatile matter. The spent mass from the producer-gas purifiers contains only 20–25% S, and is used further in the purification of coal gas. The final spent mass from the coal-gas chambers by operating in the above-described manner contains 6–9% of Prussian-blue and the gas supplied to the town contains 6–10 g./100 m.³ of hydrogen cyanide and 1.5 g./100 m.³ of hydrogen sulphide. The average life of the purification mass is 14–16 months.

(ii) A discussion of Kaudela's paper (preceding). If, when a purification chamber is refilled with new iron oxide, the crude gas is passed through this chamber for some days as the first of the series and the chamber is then made the last, much more efficient removal of hydrogen cyanide is obtained, the effluent gas from the chambers then containing only 1 g./100 m.³ of cyanide, when they have all been treated in this manner. Addition of ferrous sulphate to the contact mass adversely affects the absorption of sulphur.

A. R. POWELL.

Chemical reactions in the Petit process [of gas purification]. W. TER-NEDDEN (Brennstoff-Chem.,

1930, 11, 67—68; cf. B., 1930, 227).—The crude gas is washed with a solution of potassium carbonate which absorbs all the hydrogen sulphide and part of the carbon dioxide. The higher rate of absorption of the less strongly acidic hydrogen sulphide is attributed to the relatively slow rate of conversion of the dissolved carbon dioxide into carbonic acid, which then reacts with the potassium carbonate, whereas the hydrogen sulphide reacts directly with the salt. The first stage of regeneration in the saturator, represented by $\text{KHS} + \text{CO}_2 + \text{H}_2\text{O} = \text{KHCO}_3 + \text{H}_2\text{S}$, is also a time reaction. From the equilibrium constant $[\text{KHCO}_3][\text{H}_2\text{S}]/[\text{KHS}][\text{CO}_2]$, which equals 2.06—2.3 at 20°, and the sulphide content of the wash-liquor, the composition of the gases leaving the saturator can be calculated. The fact, however, that the decomposition of the potassium hydrogen sulphide begins only after the conversion of the excess carbonate into bicarbonate must be taken into consideration. The hydrogen sulphide evolved may be absorbed by ferric oxide and subsequently recovered as sulphur of a high degree of purity, or may be oxidised to sulphuric acid. The second stage of regeneration consists in the conversion of the bicarbonate into carbonate by heating.

A. B. MANNING.

Naphthalene and water in [coal] gas. K. ZIMPELL (Gas- u. Wasserfach, 1929, 73, 135—136).—Experiments in the Augsburg gasworks on the cooling of coal gas showed that by the use of an ammonia refrigerator combined with a heat interchanger it was possible, by cooling the gas to 0°, to remove the naphthalene as an oil and the water and ammonia as a strong ammonia liquor, leaving 2.7—3.7 g. of naphthalene, 410—490 g. of water, and 0.2—0.3 g. of ammonia per 100 cub. m. in the gas, the cost of the process being only 1.48 RM. for 2000 m.³/hr., which is less than the cost of pumping the condensate from the various sumps in the town.

A. R. POWELL.

Determination of the calorific value of a small quantity of gas by the Union calorimeter. A. BLACKIE (J. Sci. Instr., 1930, 7, 84—89).—The chief source of error is the formation of ozone in the generation of the electrolytic gas used as a standard.

C. W. GIBBY.

Calculation of gas calorific values by nomogram. F. JONES (Gas World, 1930, 92, Coking Sect., 31).—The construction and use of a nomogram for calculating the calorific value of a combustible gas from its composition is described.

C. B. MARSON.

Chemistry and physics of the combustion of gaseous fuels. Burner design and combustion of fuels in industrial furnaces. G. E. SEIL, H. A. HEILIGMAN, and C. N. WITHEROW (Ind. Eng. Chem., 1930, 22, 179—185).—The importance of the oxidising or reducing nature of the furnace atmosphere in the burning of bricks and pottery and in metallurgical operations and also the degree of control which can be obtained when using different types of fuel are discussed. Various methods suitable for burning gas on an industrial scale are described and the use of an annular orifice-type burner is discussed. This burner can be supplied with air which is too hot to be compressed. Curves and experimental data obtained

in the operation of this burner with producer gas, town's gas, and with oil fuel are given.

H. INGLESON.

Influence of pressure on the ignition velocity of explosive mixtures of methane and air. E. TERRES and J. WIELAND (Gas- u. Wasserfach, 1930, 73, 97—103, 125—133).—The rate of propagation of the flame in exploding methane-air mixtures under varying conditions has been determined by means of a tuning fork and two magnetic circuits at either end of a measured length of a bomb, the circuits being broken when the flame melted tin-foil strips in its path and the time recorded on a moving band which also recorded the movements of the tuning fork from which the interval between the two breaks could be measured. When the gas mixture contains sufficient oxygen for complete combustion of the methane the rate of propagation of the flame is at first reduced by increasing the pressure, then is hardly affected by further increase of pressure above a definite value, and finally at very high pressures becomes more rapid again. If insufficient oxygen is present, however, an increase of pressure increases the rate of propagation of the flame and also increases the explosive range, i.e., it causes mixtures to explode which would be safe at lower pressures. The maximum rate of explosion occurs with a mixture containing 10% of methane, and with increasing pressure the rate falls from 6.1 m./sec. at 0 atm. to 4.5 m./sec. at 60 atm.; at the same time the explosive range broadens from 7—13% of methane to 6.5—28% of methane.

A. R. POWELL.

Extinction of ethylene oxide flames with carbon dioxide. G. W. JONES and R. E. KENNEDY (Ind. Eng. Chem., 1930, 22, 146—147).—The comparative toxicity of a mixture of ethylene oxide (1.8%) and air, used as a fumigant, introduces hazards due to the possibilities of explosions. The upper and lower limits of inflammability of ethylene with air roughly dried with calcium chloride were 3 and 80% by vol. Carbon dioxide had a marked extinctive effect on the upper-limit mixtures with the proportions of 0—2% vols. of carbon dioxide to 1 vol. of ethylene oxide. When the proportions are above 2:1 the effect is in more general agreement with that of other gases, e.g., methane and ethylene. At least 7.15 vols. of carbon dioxide per vol. of ethylene oxide are required to ensure non-inflammability under all conditions at normal temperatures and pressure.

C. A. KING.

Primary industrial tar. J. M. PERTIERRA (Anal. Fis. Quím., 1930, 28, 137—145).—A sample of residual tar obtained from low-temperature carbonisation retorts employing bituminous coal had d_4^{20} 1.1086 and calorific value 9.472 g.-cal./g., and contained matter insoluble in ether 2.9%, phenols 26.8%, basic substances 3.2%, and neutral oil 65.9%; of the last-named, 21.2% consisted of polycyclic compounds.

H. F. GILLBE.

Direct recovery of standard road tars and other tar constituents of coal-distillation gases by fractional condensation. F. COOKE (Gas World, 1930, 92, Coking Sect., 27—30).—A fractional condensation process for the distillation of tar (cf. B.P. 301,645; B., 1929, 120) and its advantages are described.

C. B. MARSON.

Chemical nature of the light hydrocarbons recovered by compressing cracking gases and their industrial utilisation. D. ROMOLI-VENTURI (*Annali Chim. Appl.*, 1930, 20, 18–26).—The hydrocarbons obtained by compressing the gases formed during the cracking of petroleum residues (mazout, fuel oil, etc.) or of the primary tars resulting from the low-temperature carbonisation of lignite pitch, shale, and asphaltic rock, amount to 5–10% of the original material. These hydrocarbons, formed by the pyrogenic reactions accompanying the dissociation of the hydrocarbons of the raw material, consist of diolefines and diacetylenes, and are quite free from the corresponding olefinic and aliphatic terms. It is not possible to obtain, from their halogenated products, alcohols, ketones, and aldehydes of use as lacquer solvents, nor can such products be used as substitutes for tetrachloroethane, dichloroethylene, etc. as they decompose when distilled.

T. H. POPE.

Calculating heat for flashing petroleum hydrocarbons. S. D. TURNER and J. W. HARRELL (*Chem. Met. Eng.*, 1930, 37, 98–99).—If S = sp. heat of the liquid, s = sp. heat of the vapour, l = latent heat of vaporisation, x = weight fraction vaporised, and Q = heat added during vaporisation, then $Q = (l + aS) \times x - \frac{1}{2}a(S - s)x^2$, making certain approximations to render integration possible. A comparison is made between results obtained, using this equation and those given if (1) the changes in gravity of vapour and liquid which approximately offset each other are neglected, (2) the liquid is assumed to be heated to the highest temperature and then vaporised, (3) the liquid is assumed to be first vaporised and the vapour heated. Appreciable errors are introduced by the last two assumptions.

C. IRWIN.

Problems in the determination of unsaturated hydrocarbons in gases. II. Limitations in separations by sulphuric acid. H. S. DAVIS and D. QUIGGLE (*Ind. Eng. Chem. [Anal.]*, 1930, 2, 39–41; cf. Davis, B., 1929, 583).—Errors in certain methods which have been proposed and in some case are still used for determining the separate olefines in gases by absorption in sulphuric acid are discussed. The rates of absorption of propene and *n*-butenes are too close to permit their separation by selective absorption, and gas analyses based on such procedure are misleading. The saturation of 87% sulphuric acid with silver or nickel sulphate decreases its effectiveness for separating ethylene from propene in an Orsat pipette more than one hundredfold.

H. S. GARLICK.

Anti-knock motor fuels from Pennsylvania oil. G. EGLOFF and E. F. NELSON (*Brennstoff-Chem.*, 1930, 11, 91–94).—Motor fuel of high anti-knock value (50% benzene equivalent) can be produced by cracking Pennsylvania oil at 500° under a pressure of 200 lb./in.² The yield is over 65% and the production of gas is small. The cracked distillate is readily refined, the consumption of sulphuric acid being only 1½ lb. per barrel. The plant used in the process is briefly described.

A. B. MANNING.

Flame characteristics of "pinking" and "non-

pinking" fuels. G. B. MAXWELL and R. V. WHEELER (*Fuel*, 1930, 9, 121–129).—See B., 1929, 878.

Analysis of American crude oils. S. A. VUISHE-TRAVSKI (*Azerbeid. Neft. Choz.*, 1929, No. 6–7, 100–104).

Determination of olefine and aromatic hydrocarbons. FARAGHER and others. **Separation of phenols from cresols etc.** MILLER and URBAIN.—See III. **Creosote oil as preservative for timber.** RHODES and GARDNER.—See IX. **Gasoline storage tanks.** PRESSEL.—See X. **Lacquer diluents.** BRUNKOW.—See XIII. **Gas-liquor as fertiliser.** RAUPP.—See XVI.

See also A., Mar., 286, **Nature of "active carbon"** (LOWRY). 322, **Formation of petroleum of the naphthene type** (PETROV). 331, **Cracking of cyclic hydrocarbons with hydrogen at high pressures** (IPATIEV and others). **Thermal decomposition of perhydro-fluorene and -acenaphthene in presence of hydrogen** (ORLOV and BELOPOLSKI). 332, **Thermal decomposition of coal-tar constituents** (KOSAKA). **Separation of xylenes** (NAKATSUCHI).

PATENTS.

Retorts, gas producers, and like apparatus. BABCOCK & WILCOX, LTD., E. G. WEEKS, and W. A. RILEY (B.P. 324,853, 23.3.29).—Coal is fed from a pre-heater to one end of the retort or producer etc., in the form of a ribbon the full breadth of the retort, which is charged by means of a continuously running scraper which scrapes the coal from the charging end and distributes it evenly over the length of the retort. Any fall in the level of the fuel bed due to extraction at a greater rate than the coal is fed to the retort is utilised to regulate the speed of the extraction rollers, the hydraulic driving gear of the latter being controlled by the motion of a float on the surface of the fuel bed. Any excess coal charged to the retort falls through an overflow channel and is conveyed back to the bunker by means of an elevator.

A. B. MANNING.

Quenching coke. BABCOCK & WILCOX, LTD., E. G. WEEKS, and W. A. RILEY (B.P. 324,852, 23.3.29).—The hot coke while still giving off gas is delivered from the retort directly into a closed container wherein it is conveyed through a pool of water. The steam and gas are passed from the container to a combustion chamber from which combustion products are supplied to the retort (cf. B.P. 249,236; B., 1926, 429).

A. B. MANNING.

Purification of gas for distance transmission. F. LENZE (G.P. 457,264, 26.6.23).—The gas is cooled to a temperature, e.g., between 0° and –10°, sufficiently low to ensure the complete precipitation of the naphthalene while still permitting the use of ammoniacal washing water for the removal of ammonia.

A. B. MANNING.

Complete removal of ammonia from distillation gases. E. CHUR (G.P. 457,230, 15.6.27).—The gases are treated repeatedly with saturated and superheated steam, so that the gas is repeatedly raised to a higher temperature and the added steam is each time again condensed with the condensate.

A. B. MANNING.

Manufacture of ethyl alcohol and other products from coal-distillation gases. COMP. DE BÉTHUNE (B.P. 303,176, 29.12.28. Fr., 29.12.27).—Ethane and ethylene are removed from coal gas at -140° to -200° ; the ethylene is then absorbed in sulphuric acid, and the ethane is dehydrogenated at $600-800^{\circ}$ to form ethylene, which is condensed in the cooling apparatus. The utilisation of the ethane increases the yield of alcohol 30%.

C. HOLLINS.

Dehydration of gas. W. J. KLAIBER, ASSR. to KOPPERS Co. (U.S.P. 1,740,248, 17.12.29. Appl., 28.8.26).—The gas is brought into contact with a concentrated hygroscopic solution, e.g., a saturated solution of calcium chloride, at about the same temperature as the gas, e.g., 27° , in the lower part of a scrubbing tower, and with a similar solution at a lower temperature, e.g., 13° , in the upper part of the tower. The concentration of the diluted solutions is restored in evaporators and the solutions are then cooled to the required temperatures for recirculation in the respective stages of the process.

A. B. MANNING.

Purification and bleaching of mineral oils and other fatty materials. G. MICHOT-DUPONT (F.P. 553,338, 26.6.22, and Addn. F.P. 32,762, 21.12.26).—(A) The oil etc. is treated with 10% of its weight in sulphuric acid and, after settling, the partially purified oil is decanted off and treated again with 10% of its weight in sulphuric acid and with 5% of sodium sulphite. The mixture is agitated, heated at $110-120^{\circ}$ with superheated steam, and the oil decanted. A further acid treatment may be given if necessary. (B) When the oil contains glycerides of fatty acids the glycerol is recovered from the acid layer by known methods and the fatty acid is collected as a scum containing 1–7% of sulphuric acid. This scum is purified by treatment with per-salts and the acids are recovered by distillation.

A. R. POWELL.

Distillation of hydrocarbon oils. H. WADE. From STANDARD OIL Co. OF INDIANA (B.P. 324,376, 21.8.28).—Hot residual oil at $360-400^{\circ}$ is mixed with superheated steam at atmospheric pressure and $480-650^{\circ}$. The mixture is expanded through a mixing device into a wide conduit leading to a chamber maintained at a pressure of 25.75 mm. The velocity of the mixed oil and vapour in the conduit is 200–600 ft. per sec. and substantial equilibrium of oil and vapour is obtained. The equilibrium is completed in the large chamber and unvapourised oil runs into a receiver maintained under vacuum. The oil vapours and steam pass to a condenser maintained at such a temperature that the oil is condensed and passed to a receiver. The steam is carried, through a trap to remove entrained oil, to a condenser, the condensate being received in a vessel under vacuum. The consumption of steam is 2–10 lb. per gal. of distillate, and 70–80% of the oil is obtained as distillate.

T. A. SMITH.

Separation of liquid [hydrocarbon] mixtures. W., K., L., W., and F. MERCK (E. MERCK) (B.P. 300,266, 20.10.28. Ger., 11.11.27).—A mixture of paraffins is added to a liquid which has different solvent powers for the constituents of the mixture. On progressive cooling,

successive fractions are deposited from which oils of different densities may be recovered. Alcohols and chlorinated hydrocarbons, particularly ethyl alcohol and carbon tetrachloride, or their mixtures, are suitable liquids for the purpose. From a lubricating oil of d 0.9283, fractions of d 0.9344 and 0.9398 may be obtained in the presence of a 1:1 mixture of ethyl and propyl alcohols.

T. A. SMITH.

Separation of (A) hydrocarbon mixtures, (B) liquid mixtures, by fractional distillation. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 324,350 and 324,357, 20.7.28).—(A) Mixtures of saturated and unsaturated hydrocarbons, such as benzene and cyclohexane, *n*-pentane and isoprene, can be separated by fractional distillation if first mixed with one of the following liquids or mixtures thereof: ethylene chlorohydrin, glycol mono- or di-acetate or monomethyl ether, glycerin di- or tri-acetate, lactonitrile, diethyl tartrate, furfuraldehyde, or aromatic bases such as aniline, toluidine, or phenylhydrazine. The liquids which can be separated by this means are characterised by the fact that they have closely adjacent b.p. but different degrees of saturation. A 4:1 mixture of cyclohexane and benzene mixed with 70 pts. of ethylene chlorohydrin, on distillation in a column several metres in height at 100 mm. and 25° , gives a distillate consisting of practically pure cyclohexane, the benzene remaining behind in the chlorohydrin. Mixtures of mono- and di-olefines may be similarly separated. (B) Low-boiling liquids, such as mixtures containing an ether or organic chloride having b.p. below 20° , may be separated by fractional distillation under elevated pressure. The separation is facilitated by the addition of a proportion of high-boiling liquid. Thus crude methyl ether, containing methyl alcohol and water, on fractionation under 8 atm. gives a product boiling within 1° of the b.p. of the pure ether.

T. A. SMITH.

Conversion of heavy into light hydrocarbons. H. MARCHAND (F.P. 633,133, 5.8.26).—The heavy hydrocarbons are mixed with carbonaceous materials, and the mixture is briquetted and coked. The gases evolved are washed with water to remove steam, and the tar, fatty substances, and hydrocarbons are separated in the liquid form by condensation.

A. B. MANNING.

Obtaining hydrocarbons of low b.p. from hydrocarbons of high b.p. P. DANDSWARDT (U.S.P. 1,742,165, 31.12.29. Appl., 23.4.27).—Liquid hydrocarbons are heated to boiling in a closed still and hydrogen is admitted into the vapour space above the liquid. A portion of the mixture of hydrogen and hydrocarbon vapours and gases is continuously withdrawn and subjected to a pressure of over 2000 lb./in.² in a number of compressors connected in series and located within the charge of oil in the still, whereby hydrocarbons similar to natural gasoline are formed. The compressed gaseous mixture is discharged into the body of liquid hydrocarbons, thus effectively distributing the heat thereof, which, together with the heat formed by the operation of compression, is sufficient to boil the contents of the still. The vapours and gases are led to a condenser and the condensate is separated from the fixed gases, which are passed through a gas generator

containing glowing carbonaceous materials, whereby the hydrogen used in the process is generated.

H. S. GARLICK.

Manufacture of liquid fuels by hydrogenation of vaporous substances in the presence of catalysts.

COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 302,683, 5.12.28. Fr., 20.12.27. Cf. B.P. 295,272; B., 1929, 769).—The gases and vapours derived from the distillation of carbonaceous materials are passed successively through a purifier, a catalyst chamber, into which also fresh hydrogenating gas is continuously introduced, and a second purifier. The first purifier, which may be charged with a metal or metal oxide maintained at about 400°, retains all the inorganic sulphur and the major portion of the organic sulphur; in the second purifier the enriched gases are freed from the hydrogen sulphide which is liberated by the constant regeneration of the catalyst. A part of the hydrogen set free in the second purifier may be returned to the cycle. If desired, the apparatus wherein the hydrocarbon vapours are condensed, or absorbed in wash oil or activated carbon, may be interposed between the catalyst chamber and the second purifier.

A. B. MANNING.

Hydrogenation of hydrocarbons, especially cracked products.

H. ROSTIN (F.P. 633,127, 4.8.26).—The hydrocarbons are treated with hydrogen sulphide in the presence of alloys of copper with aluminium, zinc, bismuth, or iron, which interact with the hydrogen sulphide, producing nascent hydrogen. When the metal has been converted into sulphide, it is regenerated by treatment with hydrogen, or gases containing hydrogen, at a higher temperature.

A. B. MANNING.

Absorption of ethylene or its homologues by means of sulphuric acid. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 323,748, 9.7., 19.7., 3.8., and 29.11.28, and 11.3.29).—Potassium ferro- or ferri-cyanide or other complex cyanide catalyses the absorption of olefines in sulphuric acid (etc.) and minimises polymerisation. Sulphoferriicyanic acid, sodium nitroprusside, sodium prussoarsenite, and sodium ferrocarybonylpentacyanide are amongst the catalysts mentioned.

C. HOLLINS.

Treatment of lubricating oil stock. A. H. ACKERMAN, ASST. to CATALYTIC CHEM. CO. (U.S.P. 1,742,021, 31.12.29. Appl., 16.2.27. Renewed 15.4.29).—Agglomerates of heavy hydrocarbons and insoluble waxy deposits are broken up by the addition of a mixture consisting substantially of 120 pts. of naphthalene, 100 pts. of calcium phosphate (phosphate rock), 35 pts. of sodium hydroxide, 18 pts. of anthracene, 6½ pts. of ammonium carbonate, 6½ pts. of sodium phosphate, 15 pts. of sugar, and 1 pt. each of arsenious acid, arsenic sulphide, and golden antimony sulphide; the mixture is added to the oil in the proportion of ¼—1½ lb. per barrel, prior to agitation with sulphuric acid.

H. S. GARLICK.

Apparatus for treating [emulsified mineral] oil. A. B. STEEN, ASST. to TEXAS CO. (U.S.P. 1,740,275, 17.12.29. Appl., 22.9.23).—Oilfield emulsions are preheated to about 95° and pumped at 50—100 lb. pressure through pipe units containing fibrous filtering material

such as "Excelsior," by passing through the pores of which the protective colloid envelope is destroyed and the water is then able to separate. Provision is made at each unit for the withdrawal of the water. The direction of flow through the filtering material is periodically reversed to prevent packing of the filter.

T. A. SMITH.

Volatile spirit from heavy oils. P. BALSAMO (Swiss P. 124,532, 29.3.27).—The heavy oil, *e.g.*, solar oil, resin oil, mazout, naphtha, is passed in the form of vapour through a tube containing catalysts of various metals at 600—650°, and the products are condensed.

A. B. MANNING.

Motor fuel. T. H. STACKHOUSE (U.S.P. 1,741,206, 31.12.29. Appl., 25.11.25).—An anti-knock motor fuel consists of a solution in gasoline of 0.25—5% of carbon bisulphide and nitrobenzene in quantity about 3% of that of the carbon bisulphide.

H. S. GARLICK.

Heating of coke ovens. C. OTTO (U.S.P. 1,748,143, 25.2.30. Appl., 26.1.26. Ger., 4.2.25).—See B.P. 247,205; B., 1926, 621.

Pulverised or powdered fuel burners. H. E. HAZLEHURST and O. MARGETSON (B.P. 325,517, 20.11.28 and 30.4.29).

Apparatus for combustion of gaseous mixtures. PREMIX GAS PLANTS, LTD., and A. DOCKING (B.P. 325,568, 20.11.28).

Conversion of methane into formaldehyde etc. (F.P. 637,050). **Sulphonic acids etc.** (B.P. 300,264). **Wetting agents etc.** (B.P. 324,031). **Condensation products of polynuclear compounds** (B.P. 323,100).—See III. **Impregnation of fibrous materials** (B.P. 322,768).—See VI. **Conversion of ammonium thiocyanate into sulphate and sulphur** (B.P. 314,972). **Catalysts for oxidation of hydrocarbons** (B.P. 325,234).—See VII. **Porous brick** (U.S.P. 1,740,631).—See IX.

III.—ORGANIC INTERMEDIATES.

Determination of olefine and aromatic hydrocarbons. W. F. FARAGHER, J. C. MORRELL, and I. M. LEVINE (Ind. Eng. Chem. [Anal.], 1930, 2, 18—23).—The procedure involves the following steps: (1) Determination of the total olefine-aromatic hydrocarbon content by agitating with 3 vols. of 91% sulphuric acid for 30 min., followed by distillation to within 5° of the former end-point to remove polymerides, and a final extraction of the distillate with 3 vols. of 98% sulphuric acid. The total reduction in volume represents the olefines and aromatics in the oil; (2) removal of olefines with sulphur monochloride, distillation to remove olefine-free oil, and determination of aromatic hydrocarbons in the olefine free oil by means of nitration. Results on known mixtures show good agreement and the method is applied to the analysis of cracked gasoline from 8 American oils.

H. S. GARLICK.

Methyl alcohol from hydrogen and carbon monoxide. III. **Dimethyl ether.** R. L. BROWN and A. E. GALLOWAY (Ind. Eng. Chem., 1930, 22, 175—176; cf. B., 1929, 426).—The experiments were made at a constant total pressure of 200 atm. with a

gas in which the proportions of hydrogen to carbon monoxide was 2:1 and in which about 11% of inert gases were present. A catalyst composed of the reduced oxides of copper and chromium ($\text{Cu}:\text{Cr} = 3:1$) was used in all the experiments. It was prepared by precipitation by sodium hydroxide from a boiling solution of the mixed nitrates. The gas was circulated over the catalyst heated at temperatures between 265° and 355° at a space velocity of 3000. Measurements of the conversion of carbon monoxide into methyl alcohol and into dimethyl ether expressed in mol.-% per pass at 180 atm. nett pressure were, respectively: at 265° , 7 and 4; at 285° , 14 and 6; at 305° , 24 and 22; at 340° , 17 and 20, and at 355° , 11.5 and 13.5%. The region of maximum conversion was between 315° and 320° . It seems probable that the dimethyl ether is formed by the catalytic dehydration of methyl alcohol and not by decomposition of methyl formate formed as an intermediate compound ($2\text{H}\cdot\text{CO}_2\text{Me} = \text{CO}_2 + \text{Me}_2\text{O} + \text{H}\cdot\text{CHO}$), since the percentage of carbon dioxide in the residual gas is insufficient to agree with this equation. H. INGLESON.

Catalytic chlorination of acetic acid. G. D. LYUBARSKI (J. Appl. Chem., Moscow, 1929, 2, 621—627).—Dry, preferably electrolytic, chlorine is passed into a heated (water-bath) mixture of glacial acetic acid with iodine (1%) and red phosphorus (2%); phosphorus pentachloride (4%) is added, and a reflux condenser immediately attached to the vessel. The rate of addition of chlorine is gradually increased from 5 to 10—12 c.c. per sec.; higher rates are possible industrially. The separated monochloroacetic acid is washed with water and then with glacial acetic acid; the liquid is returned to the reaction vessel after addition of iodine (0.5%), phosphorus (1%), and phosphorus pentachloride (2%).

CHEMICAL ABSTRACTS.

Chlorination of acetic acid. E. A. SHILOV (J. Chem. Ind., Russia, 1929, 6, 538—540).—The yield of monochloroacetic acid when a catalyst composed of iodine, phosphorus, and phosphorus pentachloride is used (cf. preceding abstract) is 75—90%. The use of phosphorus pentachloride is unnecessary. The reaction appears to be autocatalytic; on addition of monochloroacetic acid, especially in presence of a small quantity of sulphuric acid, chlorination takes place energetically in the absence of a metalloid catalyst.

CHEMICAL ABSTRACTS.

Quality of commercial [ethyl] ether. H. LEFFMANN and C. C. PINES (Bull. Wagner Inst. Sci. Phila., 1930, 5, 3—6, and Amer. J. Pharm., 1930, 102, 58—62).—Fincke's modification (A., 1914, ii, 303) of Schiff's test for aldehydes and the chromic acid test for peroxides are preferred to the U.S.P. X tests for these impurities in anæsthetic ether. In applying the tests the reagents should be poured into the ether without shaking. Tinned containers favour the formation of aldehydes more than do amber glass bottles. H. E. F. NOTTON.

Quantitative separation of phenol from the cresols and higher phenols. J. N. MILLER and O. M. URBAIN (Ind. Eng. Chem. [Anal.], 1930, 2, 123—124).—A method for determination of phenol in presence of homologues is based on the fact that the former can be completely destroyed by chromic acid under

conditions which do not affect the latter. The phenolic distillates from two equal samples are treated with sulphuric acid, chromic acid being added to one only; after a suitable time for reaction, the phenolic substances are distilled off, and the difference in total phenol content, representing phenol destroyed by the chromic acid, is determined in any suitable way. The colorimetric method, in which diazotised sulphanilic acid is used, is recommended. S. I. LEVY.

Formation of *p*-hydroxybenzoic acid during the manufacture of salicylic acid. G. A. KORZHENNOVSKI (J. Chem. Ind., Russia, 1929, 9, 541—543).—The formation of *p*-hydroxybenzoic acid (3%) is minimal when (1) the pressure is uninterruptedly increased to 6 atm. and the temperature to 140° (attained after maximal pressure is reached) while the sodium phenoxide is exposed to a constant stream of carbon dioxide, and (2) a completely sealed autoclave is used.

CHEMICAL ABSTRACTS.

See also A., Mar., 304, **Catalytic oxidation of toluene by air** (KUSNETSOV and STEPANENKO). 334, **Synthesis of 1:2:3:4-dibenzpyrene and its derivatives** (CLAR). 346, **Action of alkali on 2-aminoanthraquinone at 150°** (MAKI). **Formation-curve of 2-aminoanthraquinone in presence of sodium dichromate** (MAKI). 347, **Decomposition of indanthrene by molten alkali** (MAKI).

PATENTS.

Conversion of methane into formaldehyde, ethylene, and higher hydrocarbons. H. SPINDLER (F.P. 637,050, 29.10.26).—Methane and air in the correct proportion are heated in contact with copper gauze under high pressure to obtain formaldehyde which, together with more methane, forms ethylene with the removal of water when passed over catalysts consisting of iron, cobalt, nickel, platinum, palladium, copper, chromium, vanadium, or cerium compounds at 500° under 1000 atm. pressure. Ethylene at the same temperature and pressure yields, in the presence of zinc chloride, pentane and hexane together with unsaturated aliphatic and cyclic hydrocarbons.

A. R. POWELL.

Production of sulphuric acid and/or halogen derivatives of formaldehyde. K. FUCHS and E. KATSCHER (B.P. 299,064, 19.10.28. Austr., 21.10.27).—Paraformaldehyde or other formaldehyde-generating compound is treated with chlorosulphonic acid or esters, or with hydrogen chloride followed by sulphur trioxide, to give below 70° chloromethyl chlorosulphonate, b.p. $49\text{--}50^\circ/14$ mm., and methylene sulphate, m.p. 153° ; above 70° there are obtained chloromethyl sulphate, b.p. $96\text{--}97^\circ/14$ mm., and dichlorodimethyl ether, b.p. $101.5\text{--}102^\circ$. The chloromethyl sulphate may be converted by chlorosulphonic acid at $70\text{--}80^\circ$ into chloromethyl chlorosulphonate and methylene sulphate. The products are useful as alkylating agents and as insecticides. C. HOLLINS.

Production of liquid hydrocarbons from ethylene and gases containing ethylene. C. EPNER (B.P. 295,705, Appl. [void], 8.6.28. Ger., 18.8.27).—Ethylene or gas containing it is polymerised by subjection to silent electric discharge. C. HOLLINS.

Manufacture of anhydrous formic acid. SCHERING-KAHLBAUM A.-G. (B.P. 308,731, 26.3.29. Ger., 27.3.28).—Aqueous formic acid is dehydrated by means of an acid anhydride which is readily produced from the corresponding acid, *e.g.*, phthalic anhydride, dehydrated boric or phosphoric acids, etc. C. HOLLINS.

Production of concentrated acetic acid. H. SUIDA (B.P. 303,494—5, 10. and 11.10.28. Austr., 5.1.28).—Acetic acid is extracted from (A) liquid, (B) vaporised, dilute acetic acid by means of a mixture of about equal parts of a hydrocarbon (anthracene oil, decahydronaphthalene) and a tertiary base (quinoline, dimethylaniline). C. HOLLINS.

Manufacture of (A) organic acids, (B) organic compounds containing oxygen [from carbon monoxide and alcohols, ethers, esters, or aldehydes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,475 and 323,513, 1.9.28).—In the production of acids and esters by the action of carbon monoxide on alcohols, ethers, aldehydes, or esters in presence of the catalysts described in B.P. 254,819 (B., 1926, 721) and B.P. 320,457 (B., 1930, 232) it is advantageous: (A) to increase the effective area of the catalyst by removal of one or more constituents; *e.g.*, aluminium or antimony may be removed as chloride by heating the catalyst in a stream of phosgene or chlorine, or water, ammonia, etc. may be driven off by heating in a vacuum so as to avoid fusion of the catalyst; or (B) to add as activators metal halides (sodium chloride, potassium fluoride or iodide). In both cases all traces of carbonyl-forming metals must be eliminated. C. HOLLINS.

Catalytic processes of dehydrogenation and dehydration of organic compounds. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 323,713, 30.6.28).—Most effective catalysts catalyse both dehydrogenation and dehydration; one of these activities may be diminished or suppressed by adding to the catalyst a suitable represser. Addition of the basic oxides, hydroxides, etc. of metals of group I or II suppresses the dehydration effect, whilst addition of acid oxides such as those of manganese, sulphur, chromium, phosphorus, arsenic, silicon, titanium, tin, boron, aluminium, or acid salts suppresses the dehydrogenation effect. Thus isopropyl alcohol, passed at 400° over a zinc oxide catalyst containing 4.5% of sodium carbonate, gives 99 mols. of acetone to every mol. of propylene formed; but the substitution of 7% of zinc sulphate for the sodium carbonate results in the production of 60 mols. of propylene to every 40 mols. of acetone. C. HOLLINS.

Catalytic synthesis of aliphatic alcohols. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 323,240, 22.6.28).—For the production of higher alcohols (than methyl alcohol) by a continuous process from carbon monoxide and hydrogen at about 450° and 200–300 atm. pressure in presence of a catalyst for methyl alcohol modified by addition of an alkali-metal compound, the space-velocity is increased to more than five times that which would give the maximum conversion into methyl alcohol, *i.e.*, the methyl alcohol equilibrium space-velocity. *E.g.*, a gas containing

35% of carbon monoxide and 50% of hydrogen is passed at 280 atm. and a space-velocity of 40,000 over a zinc-potassium chromate catalyst at 450°. C. HOLLINS.

Preparation of unsaturated esters. RÖHM & HAAS A.-G. (B.P. 316,547, 10.6.29. Ger., 30.7.28).—Ethyl β -chloropropionate is treated with alcoholic alkali at 15°, or ethyl β -chloroisobutyrate with basic iron chloride. The corresponding unsaturated esters are obtained in good yield, hydrolysis being avoided. C. HOLLINS.

Manufacture of nitriles. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,948, 11.2.29).—Acid amides are converted into nitriles by heating with benzotrichloride at 150–200°. The ammonium salts may be used in place of amides. The reaction is catalysed by zinc chloride, ferric chloride, aluminium chloride, sulphuric acid, or phosphoric acid. The preparation of acetonitrile, benzonitrile, and crotonitrile is described. C. HOLLINS.

Manufacture of condensation products of $\alpha\beta$ -substituted acroleins with aromatic bases [vulcanisation accelerators]. I. G. FARBENIND. A.-G. and W. KROPP (B.P. 323,512, 3.10.28. Addn. to B.P. 264,674; B., 1927, 230).—In the process of the prior patent the components are caused to react in presence of an acid, especially a fatty acid, *e.g.*, acetic, *n*-butyric, or stearic acid. The products are vulcanisation accelerators. C. HOLLINS.

Manufacture of emulsions. IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, A. SHEPHERDSON, and A. STEWART (B.P. 323,720, 2.10.28).—Urea, thiourea, alkali nitrates, or dextrans, with or without wetting agents, are added to emulsions or emulsifying mixtures made with the help of gelatinisable substances such as glue. Concentrated emulsions which are pourable and readily dilutable are obtained, the urea etc. acting as a liquefying agent for the glue. C. HOLLINS.

Manufacture of isatins. I. G. FARBENIND. A.-G. (B.P. 308,980, 2.4.29. Ger., 29.3.28).—Cyanofarmaryl-amides, obtainable by the action of hydrogen cyanide on arylcarbimides, are converted into isatins by heating with aluminium chloride or zinc chloride, preferably in nitrobenzene or trichlorobenzene, at 60–140°. *Cyanoform-6-chloro-o-toluidide*, m.p. 106–108° (decomp.), gives 6-chloro-7-methylisatin, m.p. 245–246°; the 3:5-dichloroanilide, m.p. 133°, gives 4:6-dichloroisatin, m.p. 252°; and the β -naphthylamide, m.p. 143–145°, gives α -naphthisatin, m.p. 248°. C. HOLLINS.

Manufacture and use of sulphonated organic condensation products. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 322,737, 8.9.28).—Furfuraldehyde is condensed, preferably in sulphuric acid, with naphthalene or other carbo- or hetero-cyclic compound, which may, if desired, be previously sulphonated; if necessary, the product may be sulphonated to give dispersing and wetting agents. C. HOLLINS.

Manufacture of *n*-butylated naphthalene- β -sulphonic acids [wetting and emulsifying agents]. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER and G. BALLE (G.P. 459,605, 19.3.26. Cf. B.P. 246,817;

B., 1926, 850).—*n*-Butyl alcohol is condensed with naphthalene-2-sulphonic acid in 100% sulphuric acid at 60–70°.

C. HOLLINS.

Manufacture and use of sulphonated organic compounds. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 323,788, 8.9.28).—An aromatic compound (particularly naphthalene) is condensed with a natural resin and sulphonated (or sulphonated before condensation) to give wetting and dispersing agents for use in the dyeing of acetate silk (etc.) with insoluble dyes.

C. HOLLINS.

Manufacture of sulphonic acids of the halogeno-nitrodiaryl ketones and sulphones. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,090, 15.10.28).—Diaryl ketones or sulphones carrying halogen and nitro-substituents are sulphonated, *e.g.*, with 20% oleum. The reactive halogen in the products may be replaced by amino-, hydroxyl, or thiol groups, or the nitro-groups may be reduced. 4-Chloro-3-nitrobenzophenone gives on sulphonation the 3'-sulphonic acid, which is condensed with aniline to yield 3-nitro-4-anilinobenzo-phenone-3'-sulphonic acid. 4-Chloro-3-nitro-4'-methoxybenzophenone, m.p. 95–96°, 2- and 4-chloro-3 : 5-dinitrobenzophenones, 4-chloro-3-nitrodiphenyl sulphone, m.p. 128, and 2-chloro-5-nitrodiphenyl sulphone, m.p. 171–172°, are similarly sulphonated in the *m*-position in the unsubstituted nucleus. 4-Chloro-3-nitro-3'-sulphodiphenyl sulphone is condensed with thio-*p*-cresol to give 2-nitro-4-*m*-sulphobenzenesulphonylphenyl *p*-tolyl sulphide.

C. HOLLINS.

Manufacture of sulphonated isatoic anhydrides and of the corresponding *o*-aminocarboxylic acids therefrom. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,018, 13.10.28).—An isatoic anhydride, obtained by phosgenation of an *o*-aminoarylcboxylic acid of the benzene, naphthalene, or anthraquinone series, is sulphonated with 20% oleum at 10–15°, and the product is converted into a sulpho-*o*-amino-acid by treatment with hot sodium carbonate solution.

C. HOLLINS.

Purification of sulphonic acids, naphthenic acids, etc. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 300,264, 11.10.28. Holl., 11.11.27).—To the aqueous solution of naphthenic acids or sulphonated mineral oils benzene is added and the emulsion is flocculated by addition of ammonia. The naphthenates or sulphonates are recovered, after separation of the aqueous salt solution, by distilling off water, benzene, and ammonia.

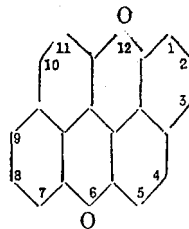
C. HOLLINS.

Manufacture of wetting, emulsifying, cleansing agents, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,031, 8.10.28).—The unsaturated compounds produced by dehydration of oxidised paraffins, waxes, etc., sulphonated or chlorinated if desired, are converted into water-soluble salts by the action of alkalis or organic bases.

C. HOLLINS.

Manufacture of derivatives of dinaphthylene dioxide. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 323,806, 11.10.28).—A di- β -naphthol obtained by oxidation of a β -naphthol (particularly a 2 : 3-hydroxy-naphthoic arylamide), is heated in nitrobenzene or

trichlorobenzene with a metal oxide such as copper oxide or manganese dioxide, whereby products of the type shown in the annexed formula result.



2 : 3-Hydroxynaphthoic *o*-toluidide gives *dinaphthylene dioxide*-5 : 11-dicarboxyldi-*o*-toluidide, m.p. 398–400° (*diamide*, m.p. 370–372°; *di-m-nitroamidide*, m.p. above 500°; *di-p-anisidide*, m.p. 425–426°; *di- α -naphthylamide*, m.p. 415–416°; *di- β -naphthylamide*, m.p. 375–376°; free acid and ethyl ester described). Ethyl 2 : 6-hydroxynaphthoate yields the ethyl 2 : 8-dicarboxylate, 2-hydroxy-4 : 5-naphthalic anhydride the 3 : 4 : 9 : 10-tetracarboxylic acid (*anhydride*, m.p. about 360°), 6-methoxy- β -naphthol the 2 : 8-dimethoxy-compound, m.p. 315–316°, and 7-benzamido- β -naphthol the 1 : 7-dibenzamido-compound, decomp. 370°.

C. HOLLINS.

Manufacture of dinaphthylene dioxide quinone. I. G. FARBENIND. A.-G. (B.P. 298,640, 10.10.28. Ger., 13.10.27).—Dinaphthylene dioxide is oxidised at 40–50° in aqueous suspension by chromic acid to give the 4 : 10-quinone (cf. B.P. 323,806; preceding abstract).

C. HOLLINS.

Manufacture of aralkylamines and their derivatives. I. G. FARBENIND. A.-G. (Swiss P. 122,523 and 122,526–8, 23.3.26. Ger., 30.3.25. Addns. to Swiss P. 120,519. Cf. B.P. 249,883; B., 1927, 572).—Further to the B.P. the following are described : 1 : 4-bis(*phthalimidomethyl*)benzene, m.p. 279–280°, and 1 : 4-xylylene-diamine, b.p. 150–160°/14 mm.; N-(2-hydroxy- α -naphthylmethyl)phthalimide, m.p. 195°, and N-(2-hydroxy- α -naphthylmethyl)amine, m.p. 141° (*hydrochloride*, m.p. 225°); *lactam*, m.p. 259°, of 5-aminomethyl-*o*-cresotic acid (*phthalimide* compound, m.p. 238°); N-(5-nitro- α -naphthylmethyl)phthalimide, m.p. 207°, and 5-aminomethyl- α -naphthylamine, m.p. 62°.

C. HOLLINS.

Manufacture of 4-methyl-6-halogenohydroxythionaphthens [6-halogeno-4-methylthioindoxyls]. I. G. FARBENIND. A.-G. (B.P. 295,593, 13.8.28. Ger., 11.8.27).—5-Chloro-2-cyano-*m*-toluidine, obtained from 5-chloro-3-nitro-*o*-toluidine by the Sandmeyer reaction and reduction, is converted in the usual manner by way of its diazo compound into the cyanothioglycollic acid and hence into the thioindoxyl. The cyano-group is not attacked by the aqueous alcoholic alkali used for hydrolysis of the xanthate.

C. HOLLINS.

Manufacture of alicyclic lactones of hydrogenated aromatic polycyclic series of hydrocarbons [vermicides]. G. SCHROETER and A. GLUSCHKE (B.P. 323,187, 23.8.28).—A sodiomalonic ester is condensed with an α -halogeno-derivative of a polycyclic ketone, and after hydrolysis the product is reduced and the resulting lactone decarboxylated. An alkyl group may be introduced before hydrolysis or before decarboxylation. The polycyclic ketone, without halogenation, may be condensed with ethyl oxalate in presence of sodium, and the product, reduced and hydrolysed, yields a dihydroxy-acid which may be isomerised to a γ -keto-acid (reducible to a lactone as before), or may be converted into a hydroxylactone. All the lactones so obtained are

useful as vermicides. 6-Bromo-5-keto-5:6:7:8-tetrahydronaphthalene with methyl sodiomalonate yields a *keto-ester*, m.p. 96—97.5°, the *acid* of which, m.p. 165°, is decarboxylated to the 5-keto-5:6:7:8-tetrahydro-6-naphthylacetic acid, m.p. 110° (3-nitro-acid, m.p. 192—193°; 3-amino-acid, m.p. 171—172°; 3-hydroxy-acid), or may be reduced to the *lactone*, m.p. 140—141°, of 5-hydroxy-5:6:7:8-tetrahydro-6-naphthylmalonic acid (*methyl lactone-ester*, m.p. 65—66.5°), which on decarboxylation yields a *lactone*, m.p. 106°, also obtainable by reduction of the acetic acid. 6-Bromo-1-methoxy-5-keto-5:6:7:8-tetrahydronaphthalene, m.p. 89—91°, similarly gives an *acetic acid*, m.p. 177—178°, reducible to the *lactone*, m.p. 134.5°, of 5-hydroxy-1-methoxy-5:6:7:8-tetrahydro-6-naphthylacetic acid; the *carboxylactone* is also described. From 5-keto-1:4-dimethyl-5:6:7:8-tetrahydronaphthalene, m.p. 21°, by bromination, condensation, hydrolysis, and reduction there is obtained the *carboxy-ketone*, m.p. 148—150°, which on decarboxylation gives the *lactone*, m.p. 129—131°, of 5-hydroxy-1:4-dimethyl-5:6:7:8-tetrahydro-6-naphthylacetic acid; the *ethyl ester* of the *carboxy-ketone* is converted by methylation and decarboxylation into *r*-hyposantonin, m.p. 113—114°. A *keto-acetic acid*, m.p. 167°, and *lactone*, m.p. 174°, are obtained from 3-bromo-4-keto-1:2:3:4:5:6:7:8-octahydroanthracene. Condensation of ethyl oxalate with 5-keto-5:6:7:8-tetrahydronaphthalene leads to a *diketo-acid*, m.p. 116—117°, which on reduction with sodium amalgam yields 5-hydroxy-5:6:7:8-tetrahydro-6-naphthylglycollic acid; the *syn*-isomeride, m.p. 165°, is converted by treatment with acetic anhydride into the *acetate*, m.p. 114.5°, of the *hydroxylactone*, m.p. 143.5°, whilst the *anti*-isomeride gives a *hydroxylactone*, m.p. 160—161°. Similarly, from 1-methoxy-5-keto-5:6:7:8-tetrahydronaphthalene there are obtained a *diketo-ester*, m.p. 44—45°, and a *hydroxyglycollic acid*, which is reduced to *lactones*, m.p. 182—183° and 160°, respectively, of isomeric 5-hydroxy-1-methoxy-5:6:7:8-tetrahydro-6-naphthylglycollic acids, or may be isomerised by acids to 1-methoxy-5-keto-5:6:7:8-tetrahydro-6-naphthylacetic acid, m.p. 177—178°, reducible to a *lactone*, m.p. 134.5—135.5°. 1-Keto-1:2:3:4:5:6:7:8-octahydroanthracene yields with ethyl oxalate a *diketo-ester*, m.p. 75—76° (*acid*, m.p. 120—122°), and eventually a *hydroxylactone*, m.p. 235—236°.

C. HOLLINS.

Manufacture of *o*-carboxyamido-arylthioglycollic acids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 319,075, 19.6.28).—An *o*-cyanoarylsulphonyl chloride, obtained from an *o*-aminoarylsulphonic acid by Sandmeyer reaction followed by treatment with phosphorus pentachloride or chlorosulphonic acid, is reduced and hydrolysed to give the *o*-thiolbenzamide, which is then condensed with chloroacetic acid. The reduction and hydrolysis may take place in one operation, using zinc dust and hydrochloric acid. 5-Chloro-2-cyanotoluene-3-sulphonyl chloride, m.p. 74—75°, the 6-chloro-isomeride, m.p. 74—75°, 4-cyanophenetole-3-sulphonyl chloride, and 5-chloro-2-cyanobenzenesulphonyl chloride are thus converted by way of the thiolbenzamides into benzamide-2-thioglycollic acids having as substituents 4-chloro-6-methyl (m.p. 172—174°), 5-chloro-6-methyl

(m.p. 173—174°), 4-ethoxy- (m.p. 208—210°), or 4-chloro- (m.p. 206°).

C. HOLLINS.

Manufacture of condensation products of polynuclear compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 323,100, 3.12.28).—Olefines (e.g., cracked gases) are condensed with polynuclear, carbocyclic hydrocarbons (especially naphthalene) or their oxygen-free derivatives (except fully hydrogenated products), in presence of aluminium chloride, under pressure at about 100° and preferably in presence of a solvent (decahydronaphthalene). Mono- and di-ethylnaphthalenes, b.p. 140—200°/20 mm.; tetraisopropylnaphthalene, m.p. 125—126°; tetrabutyl-naphthalene, b.p. 186—190°/5 mm.; and other condensation products are described.

C. HOLLINS.

Manufacture of anthraquinone derivatives. IMPERIAL CHEM. INDUSTRIES, LTD., F. LODGE, and W. W. TATUM (B.P. [A] 322,576, 2.10.28, and [B] 323,026, 18.9.28 and 30.4.29).—(A) β -Sulphonic groups are removed from 1:4-diaminoanthraquinone-2-sulphonic acids by reduction with dextrose and alkali. This reagent attacks only such sulphonic groups as are *ortho* or *para* to an α -amino- or substituted amino-group. (B) The sulphonic group in 1:4-diaminoanthraquinone-2-sulphonic acids is replaced by alkoxyl by treatment with sodium alkoxides or with solutions of sodium hydroxide in the appropriate alcohols. The preparation of 1-amino-2-methoxyanthraquinones containing the following substituents are described: 4-anilino-, 4-amino-, 4-sulphoanilino-, 4-sulpho-*p*-toluidino-, 8-sulpho-4-*p*-toluidino-, 4-methylamino-; also 1-amino-4-*p*-toluidino-2-ethoxy- and -2-*n*-butoxyanthraquinones, and 1:4-diamino-2-*n*-butoxyanthraquinone.

C. HOLLINS.

Manufacture of condensation products of the anthraquinone series [di-*o*-carboxyanilinoanthraquinones]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,750, 6.9.28).—The condensation of 1:5-dichloroanthraquinone with potassium anthranilate in presence of copper compounds proceeds best in alcohol, or especially in *n*-butyl alcohol, at 170—180°.

C. HOLLINS.

Manufacture of 2-[4-]methylbenzanthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 322,745, 7.9.28. Cf. B.P. 322,253; B., 1930, 315).—4-Benzanthronyl thioethers of the type, $\text{Bnz} \cdot \text{S} \cdot \text{CH}_2 \cdot \text{COR}$, are converted by alkaline hydrolysis (aqueous potassium hydroxide at 125—130° or boiling alcoholic potassium hydroxide) into 4-methylbenzanthrone. In the examples R is methyl or phenyl.

C. HOLLINS.

Manufacture of substitution products of the anthanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,750, 25.8.28).—Anthanthrones are mononitrated with 96% nitric acid in hot nitrobenzene, the products reduced, and the resulting aminoanthanthrones are condensed with halogenated compounds. Vat dyes are obtained by condensing aminoanthanthrone with isophthaloyl chloride (red), *m*-methoxybenzoyl chloride (red), 1-chloroanthraquinone-2-carboxylic chloride (brown-red), 2-chloroanthraquinone (grey), dibromoanthanthrone

(brown), *o*-chlorobenzaldehyde (red), 1-amino-2-aldehydroanthraquinone (blue-red), cyanuric chloride (blue-red), and octobromocyclohexane (grey). C. HOLLINS.

Manufacture of aromatic azides. RHEINISCHE KAMPFER-FABR. G.M.B.H., Assees. of S. SKRAUP and K. STEINRUCK (G.P. 456,857, 11.12.25).—Aromatic diazonium salts are treated with ammonia and sodium toluenesulphonchloroamide, and after some hours the aryl azide is distilled in steam. The preparation of phenyl azide, b.p. 80°/30 mm., *o*-tolyl azide, b.p. 70/71°/12 mm., and *p*-tolyl azide, b.p. 72°/12 mm., is described. Chloroamine, from hypochlorite and ammonia, when added to *p*-nitrodiazobenzene in presence of sodium acetate precipitates *p*-nitrophenyl azide immediately. C. HOLLINS.

Manufacture of diazoamino-compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,041, 15. and 24.10.28 and 11.3.29).—A diazo compound is coupled with a primary amine of the benzene series carrying a carboxyl and a sulphonic group or two or more carboxyl groups; or a diazo compound carrying negative substituents (nitro-, halogen, sulphonamido-, etc.) is coupled with a water-soluble, polynuclear, primary amine which does not form azo compounds when coupled; or an energetically coupling diazo compound carrying negative groups is coupled with a di- or poly-sulphonated amine of the benzene series. The resulting diazoamino-compounds are salted out and are stable to heat and alkalis; acids resolve them into diazo salts and arylamine-carboxylic or -sulphonic acids. The compounds are used in dyeing and printing, and as insecticides. Amongst the examples are diazoamino-compounds from: *m*-chloroaniline, sulphanilic acid, 4-chloro-*o*-toluidine, or α -aminoanthraquinone with 4-sulphoanthranilic acid; *o*-chloroaniline with 5-aminoisophthalic acid; 4-chloro-*o*-toluidine with 4-amino-2-sulphophenyloxamic acid; 2:5-dichloroaniline with β -naphthylamine-4:6:8-trisulphonic acid; *m*-chloroaniline or 2:5-dichloroaniline with *p*-toluidine-*o*-sulphonanilide; *p*-nitroaniline with 4-amino-2-benzene-sulphonamidotoluene. C. HOLLINS.

Production of mercaptans of the furfuryl series. H. STAUDINGER and T. REICHSTEIN, Assrs. to INTERNAT. NAHRUNGS- U. GENUSSMITTEL A.-G. (INGA) (U.S.P. 1,748,527, 25.2.30. Appl., 9.6.28. Ger., 16.12.26).—See B.P. 286,152; B., 1928, 327.

Ethyl alcohol etc. from coal gas (B.P. 303,176). **Separation of hydrocarbon mixtures** (B.P. 300,266, 324,350, and 324,357). **Absorption of ethylene** (B.P. 323,748).—See II. **Intermediates of dinaphthylenedioxide series** (B.P. 324,017).—See IV. **Polymerisation of diolefines** (B.P. 324,004).—See XIV.

IV.—DYESTUFFS.

Black chrome dye from aminodisulphobenzoic acid. V. G. GULINOV (J. Chem. Ind. Moscow, 1929, 6, 432—433).—The trisodium salt of 6-hydroxy-3-[4-(2-hydroxynaphthylazo)naphthylazo]-2:4-disulphobenzoic acid has been prepared as follows. Sulphite-reduction of nitrososalicylic acid affords 5-amino-2-hydroxy-4:6-disulphobenzoic acid (+ 2H₂O, eliminated

at 103—105°), 37.1 g. of which, dissolved with 10.6 g. of sodium carbonate in 2 litres of water, are cooled to 7—10°, acidified, and diazotised. The acidified liquid at 3—5° is treated with 14.3 g. of α -naphthylamine in aqueous hydrochloric acid, stirred for 1.5 hrs., and treated with sodium acetate. After 24 hrs. hydrochloric acid is added, the solution is heated at 50—60°, and the dye is collected; the paste is dissolved in dilute sodium carbonate, cooled to 0°, acidified, and diazotised. After 3 hrs. the diazonium compound is added to an alkaline solution of 14.4 g. of β -naphthol at 0°, or is preferably first separated as a paste. The solution is maintained at 3—5° for 1 hr., heated at 30—35°, and kept for 24 hrs., the dye being salted out at 80—83° with 18% of salt. Wool is dyed black with a slightly violet shade, and the colour is as fast as that of diamond-black. By using *p*-cresol instead of β -naphthol, a corresponding compound which dyes wool brownish-black was obtained.

CHEMICAL ABSTRACTS.

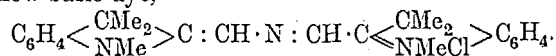
Determination of metals in dyes. THRUN.—See XIII.

See also A., Mar., 346, Anthraquinoneazo- β -naphthols (MAKI). 355, [Diazotised] aryl antimony compounds (RIDDELL and BASTERFIELD).

PATENTS.

Manufacture of water-soluble compounds of indigoid dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 234,119, 19.10.28).—Indigo or an indigoid dye, containing at least one NH group, is condensed with a chlorosulphonylbenzoic acid in presence of pyridine. The products are not sulphonamides but sulphobenzamide derivatives; they are readily soluble in water and regenerate the vat dyes when treated with cold dilute caustic alkali. *N-m*-Sulphobenzoyl derivatives of indigo and of the product from dibromoisatin α -chloride and α -naphthol are described. C. HOLLINS.

Manufacture of dyes from indoline bases. I. G. FARBENIND. A.-G., Assees. of O. WAHL (G.P. 459,616, 4.5.26).—2-Methylene-1:3:3-trimethylindoline, stirred in acetic anhydride with sodium nitrite, gives a greenish-yellow basic dye,



The acetamidoindoline derivative yields an orange-red.

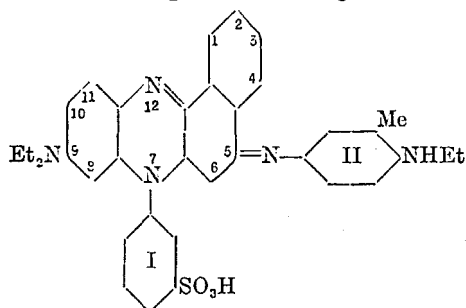
C. HOLLINS.

Manufacture of new dyes and dye intermediates of the dinaphthylene dioxide series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,017, 12.10.28).—1:1'-Dinaphthylene 2:8':8:2'-dioxide is converted by halogenation, preferably in trichlorobenzene, into its di-, tetra-, or deca-chloro-derivative or its di- or tetrabromo-derivative. The products are pigment colours.

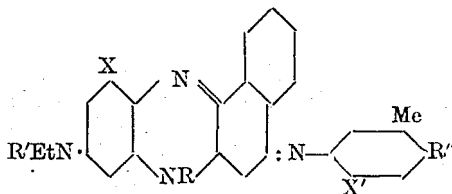
C. HOLLINS.

Manufacture of acid dyes of the phenonaphthasafranine series. J. R. GEIGY A.-G. (B.P. [A] 284,615, [B] 297,441, and [C] 300,549, [A] 31.1.28, Ger., 31.1.27, [B] 21.9.28, Ger., 21.9.27, and [C] 9.11.28; Ger., 9.11.28. Addns. to B.P. 265,986; B., 1928, 441).—(A) The process of the prior patent is applied to isorosinduline-5-sulphonic acids containing no acid substituents in positions 10 and 11. For example, 9-diethylamino-7-*m*-sulphophenyliso-

rosinduline-5-sulphonic acid is condensed with 5-aminoethyl-*o*-toluidine-4-sulphonic acid to give



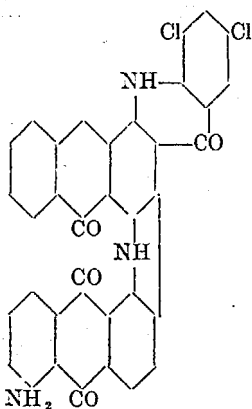
which is a greenish-blue wool dye, fast to light and alkali; the 7-*p*-sulphophenyl compound is similar. (b) Monosulphonic acids of the type shown, but carrying a sulphonic group in nucleus II only are sulphonated, e.g., with 30% oleum at 15°, whereby further sulphonic groups are introduced into the same nucleus or into another external nucleus. Thus the phenonaphthasafraanine carrying a 9-benzylethylamino-group, a 7-phenyl group, and at 5- the *p*-diamine group (figured), gives on sulphonation a disulphonic acid dyeing wool in greenish-blue shades fast to light, alkali, and fulling. [c] Instead of the aryl group attached to the azine nitrogen in position 7 in the dyes of the prior patents an alkyl group is now used. Examples are described in which (a) R =



R' = Et, R'' = NH₂Et, X = X' = SO₃H (reddish-blue); (b) R = CH₂·CH₂·OH or CH₂·CO₂Et, R' = Et, R'' = NHMe, X = X' = SO₃H (blue); R = R' = Et, R'' = NH₂Et, X = SO₃H, X' = H (reddish-blue); R = Et, R' = CH₂·C₆H₄·SO₃H, R'' = OH, X = H, X' = SO₃H (violet). (Cf. also B.P. 284,614 and 285,486; B., 1928, 781; 1929, 674.) C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL.

From I. G. FARBENIND. A.-G. (B.P. 323,543, 5.10.28).—The benzamidocarbazole-acridones obtained by the process of B.P. 305,082 (B., 1929, 277) are treated with sulphuric acid to remove the acyl group: e.g., 1:3:7-trichloroanthraquinoneacridone is condensed with 1-amino-5-benzamidoanthraquinone in 20% oleum to give the carbazole-acridone, water is added, and the temperature is allowed to rise to 100–120°, with production of a grey-black vat dye (annexed formula). The product from 1-amino-4-benzamidoanthraquinone gives bluish-grey shades. C. HOLLINS.



Production of anthraquinone vat dyes. BRIT. ALIZARINE Co., LTD., and P. P. BEGHIN (B.P. 324,084, 18.8.28. Cf. B.P. 260,588; B., 1928, 225).—1-Nitroanthraquinone-2-carboxylic acid is heated with an arylamine, especially β-naphthylamine, in nitrobenzene at 200° in presence of boric acid to give the anthraquinoneacridone in one operation. C. HOLLINS.

[Manufacture of dry finely-divided vat] dyes [of the anthraquinone series]. R. TONKIN, J. S. WILSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 324,002, 9.7.28).—Aqueous pastes of anthraquinone vat dyes are dried below 50° at ordinary or reduced pressure. During or after the drying dextrin is preferably incorporated, or the dried powder may be ground with soap. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. SOC. CHEM. IND. IN BASLE (B.P. 304,245, 17.1.29. Switz., 17.1.28. Addn. to B.P. 279,479; B., 1929, 165).—2:2'- or 3:3'-Dibenzanthronylsulphonic acids are fused with alkali and alkylated to give grey vat dyes. C. HOLLINS.

Manufacture of nitrogenous vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,079, 15.10.28. Addn. to B.P. 318,180; B., 1930, 51).—The products, probably mixed secondary or tertiary amines, of the prior patent are carbazolised by treatment with an acid or alkaline condensing agent (aluminium chloride, alcoholic alkali). Examples resulting in green, brown, and grey to black vat dyes are described. C. HOLLINS.

Manufacture of vat dye mixtures which give direct black dyeings. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 323,478, 22.9.28).—The grey-black vat dye made by alkaline treatment of an aminodibenzanthrone is mixed with an equal or smaller weight of a vat dye obtained by the action of alkali on nitrodibenzanthrone, or of hydroxylamine on dibenzanthrone, or by condensing 1:5-diaminoanthraquinone with 2 mols. of a 2-chloroanthraquinone. Blue-black to neutral black dyeings are obtained. C. HOLLINS.

Manufacture of compounds having an affinity for cotton. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 323,710, 15.1.29. Addn. to B.P. 313,636; B., 1929, 672).—The process of the prior patent is applied to nitroarylaminesulphonic acids in which the amino-group is nitrobenzoylated. The resulting nitro-azo or nitro-azoxy-compounds are subsequently, or in the same operation, reduced to amino-azo or amino-azoxy-compounds which may be diazotised on the fibre and developed. Examples are *p*-nitrobenzoic 4-nitro-2-sulphoanilide and *p*-nitrobenzoic 4-nitro-6-sulpho-α-naphthylamide, reduced with sodium sulphide and alkali, diazotised on the fibre, and developed with phenylmethylpyrazolone. C. HOLLINS.

Manufacture of azo dyes [pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 297,362, 19.9.28. Ger., 19.9.27. Addn. to B.P. 210,081; B., 1925, 310).—A 2:3-hydroxynaphthoic arylamide is coupled on the fibre with diazotised arylamides of 3-amino-4-alkoxybenzoic acids carrying in the 6-position

an alkoxyl group or halogen. Examples of diazo-components are: 3-amino-4:6-diethoxybenzanilide, 6-chloro-3-amino-4-methoxybenzanilide, and 3-amino-4:6-diethoxybenzoic *m*-chloroanilide. The shades obtained are bluish-reds. C. HOLLINS.

Manufacture of azo dyes [pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 302,251, 12.12.28. Ger., 12.12.27).—A dihalogenated *o*-anisidine, *e.g.*, 4:5- or 4:6-dichloro-*o*-anisidine, is diazotised and coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. Good light-fastness is claimed.

C. HOLLINS.

Manufacture of azo dyes insoluble in water [pigments and ice colours]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 323,937, 23.1.29).—2:3:4-Trichloroaniline is diazotised and coupled in substance or on the fibre with a bisacetoacetyl derivative of benzidine, tolidine, dianisidine, etc. [Stat. ref.] C. HOLLINS.

Manufacture of strongly basic azo dyes. J. R. GEIGY A.-G. (B.P. 295,032, 1.8.28. Ger., 6.8.27).—Unsulphonated azo dyes containing primary or secondary amino-groups are treated first with chloroacetyl chloride and then with pyridine or with a di- or tri-alkylamine. The basic groups may alternatively be similarly introduced into the components before coupling. The products give dyeings on tannin-mordanted cotton very fast to light. *p*-Nitroaniline \rightarrow β -naphthol is reduced, chloroacetylated, and boiled with pyridine to give a red for tanned cotton silk, and leather. *Pyridoacet-p-nitroanilide chloride*, m.p. 267°, obtained similarly from *p*-nitroaniline, is reduced, diazotised, and coupled with phenylmethylpyrazolone for a yellow; the *m-nitro-compound*, m.p. 185°, gives on reduction, diazotisation, and coupling through *m*-toluidine with β -naphthol a pure red. *Dimethylaminoacet-m-nitroanilide hydrochloride*, m.p. 236°, *chloroacet-p-nitroethylanilide*, m.p. 144–146°, *pyridoacet-p-nitroethylanilide chloride*, m.p. 216°, *4-nitro-N-chloroacetyldiphenylamine*, m.p. 114–115°, and *4-nitro-N-pyridoacetyldiphenylamine chloride*, m.p. 203–204°, are described, together with a large number of azo dyes in which the pyridoacetyl group is present in the diazo or the coupling component. C. HOLLINS.

Monoazo pyrazolone dye. K. H. HOOVER, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,741,418, 31.12.29. Appl., 9.10.25).—1:2:4-Aminonaphtholsulphonic acid is diazotised and coupled with 1-*m*-sulphonyl-3-methyl-5-pyrazolone or 1-(6-chloro-3-sulphophenyl)-3-methyl-5-pyrazolone for yellow-brown wool dyes becoming blue-red on chroming. Other first components are 1:2:6- and 2:1:4-aminonaphtholsulphonic acids and the 1:2:3:6-disulphonic acid. C. HOLLINS.

Manufacture of [azo] dyes containing metal. Soc. CHEM. IND. IN BASLE (B.P. 297,687, 24.9.28. Switz., 24.9.27).—Azo dyes are precoppered by treatment with a solution of cupric hydroxide in aqueous alkali containing glycerol or other polyhydric alcohol, phenol, tannin, lignin derivatives, etc. Other metals, except chromium (cf. B.P. 186,635; B., 1922, 934 A), *e.g.*, cobalt, may be similarly introduced. C. HOLLINS.

Disazo dye and its production. E. F. HITCH, H. JORDAN, and A. O. BRADLEY, Assrs. to E. I. DU PONT

DE NEMOURS & Co. (U.S.P. 1,739,032, 10.12.29. Appl., 18.1.27).—A monoaminoazo compound containing no phenolic groups is nitrobenzoylated, reduced, diazotised, and coupled with a non-phenolic arylamine, and the product is acylated to give a dye for cotton, silk, and wool. If a nitrobenzoyl chloride is used for the final acylation, the product may be reduced and the resulting dye may be diazotised and developed on the fibre. Examples are: *G*-acid \rightarrow *m*-toluidine, *p*-nitrobenzoylated and reduced, \rightarrow *m*-toluidine, benzoylated (even yellow on cotton-silk), or acetylated (redder yellow), or *p*-nitrobenzoylated and reduced (orange with β -naphthol, yellow with phenylmethylpyrazolone, on the fibre); sulphanic acid \rightarrow *m*-toluidine, *p*-nitrobenzoylated and reduced, \rightarrow Cleve acid, *p*-nitrobenzoylated and reduced (orange-brown with β -naphthol on the fibre); *G*-acid \rightarrow Cleve acid, *p*-nitrobenzoylated and reduced, \rightarrow *m*-toluidine, benzoylated (yellow). C. HOLLINS.

Treatment of azo dyes [removal of *N*-nitroso-groups from aminodiphenylamine ice colours]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 323,128, 12.1.29. Addn. to B.P. 313,865; B., 1929, 711).—Couplings of *N*-nitroso-*p*-diazodiphenylamines with 2:3-hydroxynaphthoic arylamides on the fibre are treated with a boiling solution of sodium sulphite containing a compound capable of oxidation to a quinone or quinonimine, *e.g.*, quinol, *o*- or *p*-aminophenol, or pyrogallol, to remove the nitroso-group. C. HOLLINS.

Manufacture of colour lakes and pigment dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,120, 19.10.28).—Quinizarin-2:5- or -2:6-disulphonic acid or 4-amino-1-hydroxyanthraquinone-2:8-disulphonic acid is converted into its aluminium lake. The quinizarindisulphonic acids are made from quinizarin-6- or -5-sulphonic acid by the action of bisulphite. C. HOLLINS.

Manufacture of *m*-substituted benzoylamino-anthraquinones [vat dyes]. K. H. MEYER, H. HOPFF, and A. KRAUSE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,748,240, 25.2.30. Appl., 3.11.25. Ger., 16.2.25).—See B.P. 264,561; B., 1927, 326.

Derivatives of dinaphthylene dioxide (B.P. 323,806 and 298,640). Anthraquinone derivatives (B.P. 322,576 and 323,026). Substituted anthanthrones (B.P. 323,750).—Diazoamino-compounds (B.P. 324,041).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Preservation of fishing nets by treatment with copper soaps and other substances. II. W. R. G. ATKINS (J. Marine Biol. Assoc., 1930, 16, 583–588).—A continuation of previous work (B., 1928, 255). The most efficient preservation for a single treatment is provided by either (a) 1 lb. of copper soap dissolved in 1 gal. of petrol and added to 1 lb. of tar, or (b) the same, using "Cuprinol" instead of the copper soap. Boiling with cutch, drying, boiling again, and then soaking for 15 min. in a 1% solution of copper sulphate containing excess of ammonia is also satisfactory.

C. W. GIBBY.

Composition of Philippine bagasse. A. VALENZUELA and A. P. WEST (Philippine J. Sci., 1929, 40, 275—

281).—Samples of Philippine bagasse were found to contain about 45% of α -cellulose by Cross and Bevan's method, 20% of pentosans, and 15% of lignin by Philipps' method. Preliminary experiments on the nitration of the bagasse and of the α -cellulose from it could not be made to proceed much beyond the stage of mononitro-cellulose (4.26% N).
J. H. LANE.

Measuring the stress-strain relation of wet textiles with application to wet rayons. H. A. HAMM and R. E. STEVENS (U.S. Bur. Stand. J. Res., 1929, 3, 927—936).—Photographs and a brief description are given of an apparatus for testing the strength of textiles totally immersed in water at various temperatures. Rayon tested in this machine shows considerable decrease in strength by wetting. The bath temperature has little effect on the breaking strength of regenerated cellulose rayon, but cellulose acetate rayon loses its strength with rise of temperature and its elongation due to wetting and its stretch at break increase.

A. R. POWELL.

Influence of methyl alcohol on adsorption of alkalis by cellulose. V. I. SHARKOV (J. Appl. Chem., Russia, 1929, 2, 579—583).—Adsorption of sodium or potassium hydroxide remains constant, although swelling decreases, as the amount of methyl alcohol present increases from 0 to 40%; at 50%, adsorption of potassium hydroxide is reduced to one third. Adsorption of potassium hydroxide from pure methyl alcohol depends on the moisture content of the cellulose. Cellulose (originally containing 5.51% H_2O) dried for 15 hrs. at 120—130° adsorbed practically none, whilst after drying for 4 hrs. at 100—105° it absorbed 1.7% of its weight of potassium hydroxide in 1 hr. from a 0.414% solution.

CHEMICAL ABSTRACTS.

Fractional precipitation of acetone-soluble cellulose acetate. H. J. ROCHA (Kolloidchem. Beih., 1930, 30, 230—248).—Technical cellulose acetate (zellite) has been separated into three fractions (denoted A, B, and M) by progressive addition of water to the solution of the substance in acetone. The A-fraction forms a highly viscous solution in acetone and consists of cellulose acetate with a relatively high content of inorganic impurities and some colouring matter. The M-fraction is chemically pure cellulose acetate, precipitated in a crystallised form, and the B-fraction is characterised by low viscosity. The tensile strength of films of pure cellulose acetate is proportional to the viscosity of the solution from which the film is prepared, and the B-fractions give the poorest films. The viscosity is not additive when two fractions are mixed, but is, in general, reduced. The tensile strength of films of cellulose acetate containing particles of large size can be increased to an optimum value by adding a small amount of a fraction containing small particles. The viscosity of the system cellulose acetate-acetone-water has been investigated.

E. S. HEDGES.

Avoiding losses of solvent in the [acetate] silk industry and methods of solvent recovery. E. SCHWARZ (Chem.-Ztg., 1930, 54, 121—122, 162—164).—Noteworthy losses of volatile solvents such as acetone and alcohol occur in the filling and emptying of storage and mixing vessels. These are reduced by connecting

vessels so that the saturated air remains in a closed system, but more completely so by passing all displaced air through scrubbers. A recovery system is also essential to recovery of solvent vapours during spinning, unless the thread is projected from the nozzles into the solution. Methods of recovery may operate either by condensation, absorption, or adsorption. Condensation methods are more suited to vapour-air mixtures of higher concentration than those met with in acetate silk manufacture. The condensate, owing to the presence of moisture and other impurities, requires redistillation. Absorption may be effected with cresol, sulphuric acid, or water; the second reagent is nowhere used at the present time. The Cheminova process employing cresol is described. This should give a recovery of 95—98%. Losses of cresol carried forward are minimised by the provision of cooling and settling space. Such losses, though small, are not negligible. The cresol also gradually resinifies, and must be redistilled from time to time. Water washing, unlike cresol washing, does not produce any lowering of the solvent vapour pressure. For good recovery the solutions obtained must be very dilute, and the principal expense is steam for concentration. Adsorption may be effected by activated carbon or silica gel. Comparative operating costs of the various methods are discussed.

C. IRWIN.

Alkali recovery from pulp liquors. C. L. WAGNER (Ind. Eng. Chem., 1930, 22, 122—127).—Previous attempts to burn sprayed pulp liquor for the recovery of alkali have failed owing to the use of parallel-current spraying and incomplete combustion of volatile hydrocarbons. The author uses a vertical, circular furnace wider in the upper part and with a soapstone lining. The top and the gas flue are water-cooled. The gases are passed by a fan through a scrubber and then heat a water-tube boiler. Primary and secondary air supplies are arranged to enable the gases produced in the lower part of the furnace to be burned in the upper part. The spray is from the upper part of the furnace. In the soda process the residue reaches the sloping hearth in a dry and almost completely carbonised condition. The primary air then burns the carbon and melts the soda. In the sulphate process a pool of "smelt" is maintained in the bottom of the furnace. The primary air is introduced into the fuel bed above this level, and the conditions are therefore suitable for the reduction of sulphate to sulphide in the pool. Make-up saltcake is added to the fuel. Vertical gas velocities should not exceed 10 ft./sec. and turbulence must be avoided. Permissible temperatures, heat values, and capacities are discussed. The process is at work on the large scale and the amount of steam produced closely approaches the theoretical.

C. IRWIN.

Experimental production of currency paper in the Bureau of Standards paper mill. M. B. SHAW and G. W. BICKING (Bur. Stand. J. Res., 1929, 3, 899—926; cf. B., 1927, 247).—In the manufacture of a high-grade rag paper either sodium hydroxide or lime may be used in the cooking process; the former, however, gives a somewhat softer paper of slightly better printing quality, due to the greater efficiency with which it removes the impurities from the rags and to the greater

solubility of the sodium salts formed as compared with the calcium salts. Gradual increase in roll pressure during heating is essential for obtaining high folding strength in the finished paper. The greatest strength is obtained with a mixture containing 75% of linen and 25% of cotton, but the 50 : 50 mixture is almost as strong and of somewhat better printing quality. Paper with a high content of linen is firmer and freer from "fuzz," but an all-linen paper is too hard for currency use. Paper with a high cotton content has a smaller tensile strength but a greater bearing strength than that with a high linen content. Sizing of the surface is essential for good printing results by the wet-intaglio process; the best sizing bath contains 4% of glue and 0.25% of formaldehyde as a preservative, and it is best used at 55°. Commercial tests on the above lines have resulted in the production of a much improved paper for currency use, which will withstand many more than the specified 3500 double folds without breaking.

A. R. POWELL.

pH control. MAGNUS.—See I.

See also A., Mar., 363, Microscopical examination of wool fibres (BURGESS and RIMINGTON).

PATENTS.

Treatment of cotton, woollen, and other textile yarns. D. COCKBURN (B.P. 324,974, 7.11.28).—The yarn is passed through a valve chamber, having an aperture which can be varied according to the thickness of the threads, into an impregnating chamber containing viscose, and thence through an outlet valve chamber, also adjustable, to the coagulating bath.

F. R. ENNOS.

Separation of wool fat and the like from wool-scouring wash. J. and C. E. THOMSON (B.P. 304,718, 7.12.28. N.Z., 25.1.28).—The wash is centrifuged in a separator bowl having a bulge, which extends round its periphery opposite the edges of the rotating discs and has passing through it a number of jets or openings protected by baffles. The wool fat and water are separated at the centre and sides of the bowl, respectively, while the slush of dirt and water passes through the jets in the bulge.

F. R. ENNOS.

Manufacture of artificial filaments or threads. BRIT. CELANESE, LTD., H. DREYFUS, and R. P. ROBERTS (B.P. 324,899, 25.7.28).—Cellular or tubular filaments of organic derivatives of cellulose are produced by extrusion into a cell in which a sufficient supply of heat is concentrated near the point of extrusion to prevent a lowering of the temperature by rapid superficial evaporation of the solvent; the heat concentration may be effected by means of collector devices similar to those described in B.P. 300,998 (B., 1929, 203).

F. R. ENNOS.

Manufacture of rayon [artificial silk]. MANVILLE JENCKES Co., Assees. of I. B. MERRIMAN (B.P. 301,026, 15.11.28. U.S., 23.11.27).—The cake of thread formed in the pot-spinning process is rewound under light tension on to a perforated tube to form a compacted package containing a small proportion of thread and a relatively large proportion of water and coagulants. It is then washed, subjected to the action of finishing or

dyeing solutions which are forced through the package under pressure, and dried.

F. R. ENNOS.

Esterification of cellulose. RUTH-ALDO Co., INC., Assees. of H. L. BARTHELEMY (B.P. 303,135, 3.8.28. Fr.; 29.12.27).—Cellulosic material is softened by treatment with at least 50% of its weight of hot vapours of glacial acetic acid, and then is acetylated in at least four stages by additions of acetic acid, acetic anhydride, and a catalyst (sulphuric acid together with a halogen in the earlier stages to ensure against formation of cellulose sulphate) in such quantities that in the earlier stages the reaction proceeds smoothly with only a limited rise in temperature after each addition, which is not made until the reaction due to the previous one has practically finished. In the later stages additions are made only after the partially acetylated product has attained the necessary conditions of homogeneity, the bulk of the acetic anhydride being added in the last stage.

F. R. ENNOS.

Manufacture of composite sheet material. WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 304,722, 9.1.29. Ger., 25.1.28).—One or more sheets of regenerated cellulose (e.g., from viscose) capable of swelling in water are combined with one or more sheets of a cellulose derivative (celluloid) which does not swell with water, so that no two sheets of the former are in juxtaposition. The individual sheets are joined together by an agglutinant, which is finally rendered insoluble in water, e.g., gelatin hardened with formaldehyde or a condensation product of urea and formaldehyde.

F. R. ENNOS.

Production of paper. L. MELLERSH-JACKSON. From MATHIESON ALKALI WORKS (B.P. 324,547, 15.2.29).—Chlorine, either in the form of chlorine water or a solution of hypochlorite, is added to the pulp preferably in the mixing box in just sufficient quantity for the white water to show a residual chlorine content of 0.5—1 p.p.m. as indicated by the o-tolidine test. Sterilisation of the stock is thereby effected and the white water, being free from slime-forming organisms, can be satisfactorily coagulated, e.g., with alum at pH 5.5—6.5. The short fibres and coagulated colloidal matter are separated and, being sterile, may be stored for future use. The clear back-water is returned to the mill.

D. J. NORMAN.

Production of paper pulp and paper products. M. M. SEREBRIANY (B.P. 324,527, 28.1.29).—Fibrous material, e.g., wood, bark, sawdust, straw, leather waste, is dried to a moisture content of 4—6%, ground to a fine powder, and, after sifting, is used with other fibrous material and fillers for the manufacture of pasteboard or may be used alone, in which case it is first swollen in water, preferably at 60—80°, with the addition of 20—30% of common salt. Alternatively, 6—10% of a mixture of turpentine with benzine or petroleum (optionally containing resin in solution) may be used as the swelling agent, the resulting mass being subsequently moulded. When wood bark is used part is soaked in brine until an approximately equal weight of salt has been absorbed and is then ground in an edge-mill until "greasy," and is finally mixed with dry ground

bark. Alternatively, some of the bark may be steamed under pressure. D. J. NORMAN.

Manufacture of paper, sheets, boards, or laminated products. BRIT. CELANESE, LTD. (B.P. 301,428, 29.11.28. U.S., 29.11.27).—Cellulose esters or ethers, preferably cellulose acetate prepared and precipitated under such conditions that it assumes a fibrous character, are mixed with vegetable, animal, or mineral fibres in a beating engine and the resulting stock is run on a paper or board machine. Thus 5—50% of cellulose acetate on the weight of finished paper may be used, and fillers, colouring agents, sizing agents, and high-boiling solvents for cellulose acetate may be added. The sheets thus obtained are subjected to heat and pressure, or, alternatively, are brushed with a volatile solvent which is subsequently allowed to evaporate. The products find application as electrical insulators or in the manufacture of wheels, gears, etc. D. J. NORMAN.

Esterification of cellulose materials. B. WYLAN and J. THOMAS, ASSRS. to SCOTTISH DYES, LTD. (U.S.P. 1,748,689, 25.2.30. Appl., 30.1.29. U.K., 26.1.28).—See B.P. 310,556; B., 1929, 594.

Means for production of variegated or parti-coloured yarns [by weaving]. H. P. GREG (B.P. 325,710, 6.3.29).

Paper pulp beating machines, hollanders, refiners, etc. A. E. REED & Co., LTD., and C. D. SHELDON (B.P. 325,609, 3.12.28).

Manufacture of fibre board from pulp. DOWNING-TOWN MANUFG. Co. (B.P. 325,740, 15.4.29. U.S., 24.12.28).

Decorated fabrics (B.P. 324,211). Rubber-coated fabrics (B.P. 299,321).—See XIV. Activation of proteases (B.P. 324,651).—See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of indigo blue serges. D. CARTER (J. Soc. Dyers and Col., 1930, 46, 44—47).—The effect of the condition of the vat on shade, penetration, and fastness to rubbing is briefly discussed. The presence of spinning oils due to faulty scouring, particularly of woollen materials, is a frequent cause of unsatisfactory fastness to rubbing. On the other hand some constituents of inferior-quality oils give rise to oxidation products which are extremely difficult to remove in scouring. Water containing dissolved iron is liable to give dyeings of a duller and greener shade after the indigo vat has been worked a few days. The correction of stained or unevenly dyed pieces and the modification of the shade of dyed material is described, and the effect of the addition to the dye vat of various inorganic salts, glue, organic solvents, Aktivin (toluene-*p*-sulphon-chloroamide), or "solution salt" on the stability of the vat and on the shade and fastness of the resulting dyeings are mentioned. N. CHAPPELL.

Action of light on dyed fabrics. HALLER and G. ZIERSCH (Z. angew. Chem., 1930, 43, 209—215).—The action of light on various cotton fabrics dyed with 38 different dyes of various chemical groups has been examined and the results are tabulated and discussed in detail. Although there was relatively little difference

in the fastness of the colours on the different cottons, Egyptian cotton was distinctly the best in this respect. Mercerisation appreciably reduced the rate of bleaching of the fabric and considerably reduced the loss in strength undergone by the dyed fabric on exposure to light, except in the case of fabrics dyed with basic dyes, which in all cases lost practically the whole of their strength under the conditions of the test. The effect of treating the dyed fabric with various chemicals before exposure to light has also been investigated and sodium metaphosphate, sodium dextrosemaphosphate, and thiocarbamide have been found to be especially useful in stabilising the dyed fabric towards the action of light.

A. R. POWELL.

Dip-volume relationships in laundry washing machines containing water or detergent solutions, together with fabric. R. G. PARKER, J. N. VOWLER, and A. J. V. UNDERWOOD (J. Text. Inst., 1929, 20, T 401—410).—The problem of determining the volume of liquid in a rotary washing machine under any given set of conditions, in order that the concentrations of detergents used may be accurately adjusted, has been investigated by measuring the "standard dip" (depth of liquid in the cage after running and then allowing the cage to remain stationary for 30 sec.) produced by known volumes of water in various machines, both unloaded and loaded with fabric to different degrees of loading. From the observations, expressions are deduced by means of which the volume of liquid present in washing machines (within certain limiting sizes) may be calculated from the machine dimensions, the standard dip, and the degree of loading. B. P. RIDGE.

pH control. MAGNUS.—See I.

PATENTS.

Production of dyeings and prints. I. G. FARBEN-IND. A.-G. (B.P. 299,069, 20.10.28. Ger., 20.10.27).—Cotton is printed or padded with an arylthioglycollic acid carrying an *ortho* carboxylamide group, *e.g.*, 5-chloro-2-amidocarbonyl-*m*-tolylthioglycollic acid, with the addition of caustic alkali, and the colour (bluish-red) is developed by oxidation, *e.g.*, with ferricyanide or calcium oxychloride, finally acidified, rinsed, and soaped. A sodium formaldehydesulphoxylate resist may be employed. C. HOLLINS.

Dyeing of textile materials with vat dyes ["triethanolamine" as assistant]. E. I. DU PONT DE NEMOURS & Co. (B.P. 295,025, 27.7.28. U.S., 4.8.27).—Hydroxylated aliphatic amines, especially "triethanolamine," are added as solubilising agents to the vat dye-bath. C. HOLLINS.

Dyeing of chamois leather with sulphur and vat colours. CHEM. WORKS, FORMERLY SANDOZ (CHEM. FABR. VORM. SANDOZ) (B.P. 303,523, 5.1.29. Ger., 5.1.28).—Chamois leather which has been pretreated with an oxidising agent (perborate, hypochlorite, Chloramine-T) takes up sulphide and vat dyes so quickly that the alkali present has not time to damage the leather. The colours are developed on the leather without the necessity for further oxidation, but an after-treatment with chromic or nitrous acid gives bright shades. C. HOLLINS.

Improving the dyeing capacity of hydrated cellulose. I. G. FARBENIND. A.-G. (B.P. 298,491, 8.10.28. Ger., 6.10.27).—In the process of B.P. 278,684 (B., 1929, 242) for strengthening hydrated cellulose silk, the dyeing capacity is improved by adding to the strengthening bath a soluble salt, *e.g.*, sodium chloride, especially if a weak bleach treatment follows. A mordant may be added to the strengthening bath.

C. HOLLINS.

Dyeing of fibres consisting of cellulose mono- or di-acetate. G. B. ELLIS. From CHEMICAL WORKS, FORMERLY SANDOZ (B.P. 323,011, 16.8.28).—The products of direct acetylation of cellulose without destruction of the fibrous structure are dyed, while still in the swollen state due to the acetylation, with ordinary direct cotton dyes, vat dyes, or acid dyes containing not more than one sulphonic group. If the fibre has been dried it may be swollen again by treatment with acetic acid.

C. HOLLINS.

Coloration of materials containing cellulose derivatives. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 323,180, 22.6.28).—The cellulosic constituent in fibres containing also cellulose esters is mordanted with tannin and tartar emetic under such conditions as to leave the esters unmordanted. The amount of mordant may be arranged so that basic colours are taken up equally by both constituents of the fabric.

C. HOLLINS.

Coloration of cellulose [ester and ether] derivatives. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 323,792, 5.10.28).—Acetate silk etc. is dyed with (preferably unsulphonated) azo dyes derived from nitroarylamines containing two or more linked aryl residues. Examples are: 2:4-dinitro-4'-aminodiphenylamine \rightarrow phenol (yellow); 3-nitro-4-aminodiphenyl ether \rightarrow α -naphthylamine (bluish-red); 2:2'-dinitrobenzidine \rightarrow 2 mols. of *m*-toluidine (orange); 4-nitro-4'-aminodiphenyl \rightarrow *m*-phenylenediamine (orange-brown); 5-nitro-*o*-tolidine \rightarrow 2 mols. of α -naphthylamine \rightarrow 2 mols. of 2:3-hydroxynaphthoic acid (black); 3:3'-dinitro-4:4'-diaminodiphenylmethane \rightarrow 2 mols. of *m*-tolylenediamine (golden-brown).

C. HOLLINS.

Dyeing of regenerated cellulose rayon. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 322,773 and 322,824, 12.9.28).—Level greenish-blue to violet shades are obtained on viscose silk by the application of azo dyes derived (A) from a 4-amino- or 4:4'-diaminodiphenylamine coupled with 1:8-aminonaphtholsulphonic acids; *e.g.*, 4:4'-diaminodiphenylamine with 2 mols. of S-acid (blue), or with H-acid and N.W.-acid (royal blue), or with 2S-acid and β -naphthol (reddish-blue); 4-aminodiphenylamine with 2S-acid (sky-blue); 4:4'-diamino-2-methoxydiphenylamine with H-acid and N.W.-acid (greenish-blue); 4:4'-diaminodiphenylamine-2-sulphonic acid with H-acid and *m*-phenylenediamine (blue-black); (B) from a 4:4'-diaminodiphenylamine coupled with 2:8-aminonaphtholsulphonic acids, *e.g.*, with 2 mols. of γ -acid (reddish-blue), or with salicylic acid and phenyl-J-acid (reddish-blue), or with N.W.-acid and γ -acid (reddish-blue).

C. HOLLINS.

Treatment of textile materials. BRIT. CELANESE, LTD. (B.P. 295,043, 3.8.28. U.S., 4.8.27).—A mottled or

printed effect due to differences in lustre may be produced on fabrics which are composed of or contain thermoplastic derivatives of cellulose, and which have been completely delustred or are capable of having their lustre increased, by damping the fabric in the required design and then ironing the whole at 100° or above. By suitably adjusting the moisture content and/or the temperature of ironing, any degree of lustre up to the maximum may be obtained.

D. J. NORMAN.

Manufacture of a spreading paste for proofing textiles. P. MEYERSBERG (B.P. 295,660, 16.8.28. Austr., 16.8.27).—A rubber latex is converted into a semi-coagulated, stiffened state by the addition of solutions of weak organic acids, especially resin or grease acids (*e.g.*, colophony, resin oil, tall oil, elain, stearic acid, linseed oil acids), in an organic solvent (colza oil, mineral oil). Preferably alkali sufficient to form a soap with the organic acid is added, and castor oil may be used as a stabiliser. An example is: 16% latex (65 pts.), sodium hydroxide (d 1.34, 1 pt.), whiting (20 pts.), castor oil (5 pts.), cresol (0.25 pt.), colophony (4.5 pts.), colza oil (4.5 pts.).

C. HOLLINS.

Manufacture of (A) waterproof, (B) embossed waterproof, fabrics. BRIT. CELANESE, LTD. (B.P. 296,450—1, [A, B] 1.9.28. U.S., 2.9.27).—(A) Open-mesh fabrics, such as georgette or muslins made from organic derivatives of cellulose, may be satisfactorily coated with rubber or other waterproofing compositions. (B) Fabrics composed of or containing thermoplastic derivatives of cellulose are embossed and then treated, preferably on the underside, with a waterproofing composition, particularly rubber-containing solutions.

D. J. NORMAN.

Proofed or treated cloth and its manufacture. C. E. GODDARD (B.P. 325,034, 29.11.28).—Woollen cloth is first waterproofed by treatment with a solution of rubber or of a cellulose ester and is then given several coats of cellulose acetate in admixture with a suitable plasticiser, *e.g.*, castor oil, and a pigment. After each coat the cloth is partially dried and passed between smoothing rolls. The product is suitable for use as printers' blankets.

D. J. NORMAN.

Pressing of textile materials. BRIT. CELANESE, LTD. (B.P. 295,035, 4.8.28. U.S., 4.8.27).—To avoid increasing their lustre during finishing, fabrics containing delustred cellulose acetate are ironed or calendered either in the dry condition at about or above 100°, or first at below 100°, to remove moisture, and then at about or above 100°.

F. R. ENNOS.

Preparing finished [cellulose] acetate silk by the dry or evaporative method. N. V. NEDERL. KUNSTZIJDEFABR. (B.P. 301,017, 15.10.28. Holl., 23.11.27).—The silk is spun from the usual solutions to which 1—5% of a vegetable or animal oil, or mixture of both, having m.p. 20—40° (preferably 30°) has been added. Suitable oils include olive oil, butter fat, beef suet, coconut oil, lard, bone oil, and ground-nut oil.

A. J. HALL.

Impregnation of fibrous materials. J. Y. JOHN-SON. From I. G. FARBENIND. A.-G. (B.P. 322,768, 10.9.28).—Heavy-metal (copper, iron, manganese, lead)

salts of oxidation products of paraffin wax, montan wax, etc. are used for impregnation and preservation of wood, paper, cardboard, cotton, wool, silk, etc. in conjunction, if desired, with dyes, preserving agents, soaps, resins, bituminous oils, driers, dispersing agents, etc. *E.g.*, wood is treated with the copper salt of oxidised paraffin wax, phenol-formaldehyde resin, linseed oil, solvent naphtha, lead naphthenate, or cobalt linoleate, tar oil, and a little nitrobenzene (as deodorant).

C. HOLLINS.

Protection of wool, fur, hair, etc. against textile pests. I. G. FARBENIND. A.-G. (B.P. 298,538, 9.10.28. Ger., 10.10.27).—The material is treated with salts of fluoroboroacetic acid or other complex compound of boron fluoride with an organic acid. C. HOLLINS.

Dyeing machines. I. MITCHELL (B.P. 325,696, 23.2.29).

Sulphonated organic compounds (B.P. 323,788).
Diazoamino-compounds (B.P. 324,041).—See III.
Rayon silk (B.P. 301,026).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Thermal aspect of liquid-phase sulphuric acid production. P. PARRISH (Chem. Met. Eng., 1930, 37, 93—95).—A Gaillard-Parrish chamber plant has operated for 3 years at 3.0—3.5 cub. ft./lb. of sulphur/24 hrs. It has a capacity of 1200 tons of 65% acid per month and works on a sodium nitrate consumption of 3% on sulphur burned. It consists of one Glover tower, three tower chambers (unpacked, each 18 m. high and 7 m. diam.), and two Gay-Lussac towers. Each tower chamber is fed with 600 gals. of chamber acid per hr. by means of turbo-dispersers. The circulation through the Gay-Lussac towers is about six times the daily make. The heat of formation and heat of dilution of sulphuric acid being known, together with the make in each unit, the heat balances for each unit are calculated. It is found that in the chambers 19% of the heat entering and generated is removed by the acid dispersed. The gases enter the first chamber at 100°, but the temperature of the lead walls rarely exceeds 68°. If heat removal were the only factor it is calculated that this plant might be expected to work satisfactorily at not less than 5.67 cub. ft./lb. of sulphur/24 hrs. The better actual result is due to liquid-phase working. C. IRWIN.

Utilisation of chalk and pyrite residues from the manufacture of sulphuric acid. I. A. ALEK-SANDROV (Neft. Choz., 1929, 17, 193—197).—Polemical.

CHEMICAL ABSTRACTS.

Comparison of methods for the extraction of phosphoric acid from phosphorites and Thomas slags. B. SKOPINZEV (Udobren. Uroshaj, Russia, 1929, 37—39; Chem. Zentr., 1929, ii, 2361).—Extraction with aqua regia is applicable to the analysis of phosphorites, but not of Thomas slag; extraction with nitric and sulphuric acids gives higher values. A. A. ELDRIDGE.

Simplified soda process with fixation of ammonia. W. GLUUD and B. LÖPMANN (Z. angew. Chem., 1930, 43, 190—194).—This modification of the Solvay process works throughout with solutions saturated

with respect to ammonium chloride. Another salt, *e.g.*, sodium nitrate, is added which increases the yield of the two products and also their purity. Thus 1 litre of a solution containing 20% of sodium nitrate and saturated with sodium chloride and ammonium chloride will decompose 100 g. of ammonium bicarbonate. This salt is added to the solution at 16—17°, gradually with stirring, the sodium bicarbonate filtered off, and sodium chloride added to the warmed filtrate. This is cooled to 14° with separation of ammonium chloride. Experiments on a larger scale showed that not more than 10% of sodium nitrate need be added. The loss of this was 1% of the ammonium chloride produced. Other sodium or ammonium salts can be used equally well. An experimental works' scale plant in Holland is described. In this the sodium bicarbonate produced is calcined and converted into caustic soda. The sodium bicarbonate produced contains about 0.2% NaCl and the ammonium chloride about 5% (NaCl + NaHCO₃). The ammonium bicarbonate used is prepared from ammonia from coke-oven sources without any special purification. Rotary filters for the sodium bicarbonate have been added. C. IRWIN.

Determination of ammonia in synthetic ammonia solution. EGELING (Chem.-Ztg., 1930, 54, 195).—A special form of weight pipette, provided with taps above and below the bulb, is employed. The pipette is weighed, evacuated, and a quantity of the ammonia solution is admitted by opening the lower tap after connecting the pipette with a glass tube dipping under the liquid. After reweighing the pipette its contents are run out through a glass tube dipping into water in a graduated flask; the pipette is then rinsed well with water and the contents of the flask are titrated as usual. The method has the advantage that the whole of the operations take place without exposure of the liquid to air, hence errors due to volatilisation of ammonia are obviated. H. F. HARWOOD.

Determination of sodium hypophosphite. M. FRANÇOIS and (Mlle.) L. SEGUIN (J. Pharm. Chim., 1930, [viii], 11, 193—197).—A rapid and accurate method for the determination of sodium hypophosphite is described, based on the reduction of mercuric chloride to mercurous chloride: $\text{NaH}_2\text{PO}_2 + 4\text{HgCl}_2 + 2\text{H}_2\text{O} = 2\text{Hg}_2\text{Cl}_2 + \text{H}_3\text{PO}_4 + \text{NaCl} + 3\text{HCl}$. Sodium hypophosphite reduces mercuric sulphate to mercury only when the former is present in excess; in the presence of excess mercuric sulphate, mercurous sulphate is formed (cf. Maquenne and Roux, A., 1899, ii, 381).

C. W. SHOPPEE.

Culinary rock-salt. H. ZELLNER (Chem.-Ztg., 1930, 54, 193).—Analyses of over 200 samples of commercial culinary rock-salt showed that on an average it contained 98.89% NaCl, the remainder being sulphates of calcium, magnesium, and sodium together with a little potassium chloride, water, and insoluble matter. The material is consequently just as suitable for use in culinary processes as the artificially prepared salt crystals generally employed. Detailed figures of the results of 100 analyses accompany the paper. H. F. HARWOOD.

Ferric alumina. A. R. MOBERG and E. M. PART-RIDGE (Ind. Eng. Chem., 1930, 22, 163—164).—Ferric

alumina is a colloidal sol of hydrous alumina peptised by the addition of ferric chloride. The hydrous alumina is prepared by exactly neutralising aluminium chloride with sodium aluminate. After washing, the precipitate is mixed with ferric chloride in the desired proportions. By adjusting these, precipitation can be effected at any p_H value likely in water treatment and in spite of any inhibitive colloids present. It is in consequence much more convenient than aluminium sulphate for the treatment of water contaminated by fluctuating proportions of industrial effluents. C. IRWIN.

Determination of silica in phosphate rock. W. L. HILL and K. D. JACOB (J. Assoc. Off. Agric. Chem., 1930, 13, 112—117).—Presence of fluorine in phosphate rock reduces the silica, as determined by the ordinary method, by 0.5—2.7%, as compared with that given by the modified Berzelius method (B., 1929, 1015). Increased time of acid digestion slightly lowers the insoluble material, but does not affect the silica figures.

K. V. THIMANN.

Rare earths. XXXIV. Spectrographic determination of impurities therein. P. W. SELWOOD (Ind. Eng. Chem. [Anal.], 1930, 2, 93—96).—Neodymium solutions containing known amounts of different impurities have been examined spectrographically. A drop of the concentrated solution was placed on the crater of the positive graphite electrode and the resulting spectrum photographed, using a Hilger quartz spectrograph. Manganese, magnesium, calcium, silicon, or aluminium could be detected when present to the extent of 0.001%, barium, bismuth, or beryllium to 0.01%, and samarium or gadolinium to 0.1%, the lines of the last two elements being so close to those of neodymium as to render identification difficult; 0.1% of lanthanum present in yttrium compounds could also be detected by the above method. H. F. HARWOOD.

Determination of carbon dioxide in carbonates. W. W. SCOTT and P. W. JEWEL (Ind. Eng. Chem. [Anal.], 1930, 2, 76—77).—A simple form of apparatus for the determination of carbon dioxide in carbonates by the loss of weight method is described. The results are accurate to 0.1—0.2%, but the method is not suited for use with basic carbonates or with carbonate of lead. H. F. HARWOOD.

Rapid determination of nitrogen peroxide in nitrogen peroxide-air mixtures. C. W. WHITTAKER, F. O. LUNDSTROM, and A. R. MERZ (Ind. Eng. Chem. [Anal.], 1930, 2, 15—18).—The method is based on the fact that between 150° and 160° nitrogen peroxide is completely dissociated into the dioxide, whilst the further decomposition into nitric oxide and oxygen is inappreciable. The gas is measured in a jacketed pipette, monobromobenzene (b.p. 156.2°) being used as the heating agent, and the mineral oil ("Nujol") as the confining liquid. After measurement of the volume the gas is passed through concentrated sulphuric acid to absorb the nitrogen peroxide, and the residual gas is measured in a gas pipette at the ordinary temperature. The results obtained for the nitrogen peroxide present agree satisfactorily with those given by the gas-balloon method. The apparatus used is fully described, and necessary precautions in its use are indicated.

H. F. HARWOOD.

Determination of small amounts of hydrogen peroxide and of ozone. N. ALLEN (Ind. Eng. Chem. [Anal.], 1930, 2, 55—56).—The various methods proposed for the detection of hydrogen peroxide and ozone are discussed, and their application to the determination of these substances has been studied. Hydrogen peroxide is best determined by the bleaching of a very dilute acid solution of potassium permanganate containing magnesium sulphate; 1 pt. in 10 million can thus be detected. The relative oxidising power of ozone on potassium iodide was found to be the same whether in neutral or alkaline solutions, 1 mol. of ozone liberating 1 mol. of iodine in each case, whilst in acid solutions 1.25 mols. were liberated. Small amounts of ozone may be determined by passing the ozonised oxygen through a dilute alkaline solution of potassium iodide, acidifying, and adding starch solution, the colour produced being compared with that given by a standard iodine solution; 0.000001 g. of ozone can thus be detected. Benoist's statement (B., 1919, 285 A) that 2 mols. of ozone react with 1 mol. of fluorescein could not be confirmed, a ratio of 11 to 1 being always obtained. The discharge of the green colour of the dye by ozone forms a sensitive test for the latter, but is not well adapted for quantitative purposes. H. F. HARWOOD.

Procedure for determining total nitrogen by conversion into nitric acid. E. M. EMMERT (J. Assoc. Off. Agric. Chem., 1930, 13, 146—147; cf. A., 1929, 1062).—Very rapid heating with sodium chlorate oxidises the nitrogen without risk of explosion from the formation of chlorine peroxide. The nitrate is then distilled and determined colorimetrically. The method is suitable for soil or for plant tissue. K. V. THIMANN.

Refrigeration and the saltpetre industry. WARNECKE.—See I. **Alkali recovery from pulp liquors.** WAGNER.—See V. **Baking powders.** WEIL.—See XIX. **Saline lake waters.** PANTELEIMONOV.—See XXIII.

See also A., Mar., 286, **Fractional precipitation** (RUFF and ASCHER). 304, **Synthesis of ammonia** (BREWER and WESTHAVER). 305, **Preparation of amorphous boron and borides** (ANDRIEUX). 307, **Preparation of phosphorescent substances** (SHIROV). 308, **Preparation of crystalline lead dioxide** (SIMON). **Preparation of manganese dioxide** (KATO and MATSUHASHI). 309, **Pyrophoric iron and ammonia synthesis** (FINZEL). 310, **Determination of halogen** (HELLER). **Determination of bromides and iodides** (HOFMANN; TOMÍČEK and JÁNSKY). 311, **Determination of sodium hydroxide in presence of sodium carbonate** (POETHKE and MANICKE).

PATENTS.

Manufacture of silica and like adsorbent gels [of low apparent density]. SILICA GEL CORP., Assees. of G. C. CONNOLLY and E. B. MILLER (B.P. 303,138, 14.8.28. U.S., 30.12.27).—The hydrogel is treated for 36—120 hrs. with hot water at 80—40°. The product, the apparent density of which increases as the temperature of the water falls, is activated, e.g., by heating at 315° for 3 hrs. The gels produced are hard, highly active, and heat resistant; they are relatively pure and highly adsorptive. S. K. TWEEDY.

Conversion of ammonium thiocyanate into ammonium sulphate and sulphur. C. J. HANSEN, Assee. of H. KOPPERS A.-G. (B.P. 314,972, 12.11.28. Ger., 6.7.28. Addn. to B.P. 309,565; B., 1930, 323).—The thiocyanate is heated in chrome-nickel steel vessels with ammonium thiosulphate and sulphuric acid; the two latter chemicals react to yield sulphurous acid, sulphur, and ammonium sulphate. The acidic ammonium sulphate mother-liquors obtained as in the process of the prior patent may be employed in place of the sulphuric acid of that process. S. K. TWEEDY.

Production of solid calcium nitrate. C. C. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 325,364, 25.1.29).—Solid calcium nitrate is precipitated by adding nitric acid to a concentrated solution of the salt. Preferably, a saturated calcium nitrate solution containing nitric acid is adjusted to a temperature at which solid anhydrous calcium nitrate is in equilibrium with the acid solution, and then a calcium salt together with an equivalent amount of nitric acid are added. The nitric acid may be formed *in situ* by leading in oxides of nitrogen, e.g., the products of ammonia oxidation, and if chalk be added simultaneously at an equivalent rate the process becomes continuous. The crystals are centrifuged off and washed with a suspension of chalk in calcium nitrate solution of such strength that the adherent acid is just neutralised. S. K. TWEEDY.

Production of calcium formate. F. W. SEFTON-JONES. From LONZA ELEKTRIZITÄTWERKE U. CHEM. FABR. A.-G. (B.P. 322,909, 22.12.28).—Carbon monoxide and lime are made to react at 160° under 80 atm. pressure in an aqueous calcium formate solution, saturated at 15°. On cooling, calcium formate crystallises out. C. HOLLINS.

Dehydrating substance. H. ISOBE, Assr. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,740,351, 17.12.29. Appl., 25.7.24. Jap., 1.2.24).—The product comprises inorganic adsorbent material, e.g., acid clay, impregnated with 10–20% of calcium chloride. L. A. COLES.

Manufacture of a calcium magnesium chloride product. W. R. COLLINGS and J. J. SHAFER, Assrs. to DOW CHEM. CO. (U.S.P. 1,738,492, 3.12.29. Appl., 7.3.28).—An aqueous solution containing 0–2 mols. of magnesium chloride per mol. of calcium chloride, e.g., residual brine from the purification of sodium chloride, is added continuously to a solution saturated with respect to one or more magnesium calcium chlorides, e.g., with respect to tachyrite ($2\text{MgCl}_2 \cdot \text{CaCl}_2 \cdot 12\text{H}_2\text{O}$) and to $\text{MgCl}_2 \cdot 2\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$, and the solution is evaporated under conditions of temperature and pressure such that the chlorides crystallise out in a ratio about the same as that in which they are present in the original solution. After superficial drying, non-caking, hydrated crystals are obtained. L. A. COLES.

Production of ferricyanides. J. SCHRÖTER (B.P. 314,956, 28.6.29. Ger., 5.7.28).—Ferrocyanides are treated with oxygen, or gases containing oxygen, at raised pressure and temperature in presence of a weak acid or of acid in a quantity suitable for neutralisation of the metal equivalent liberated by the oxidation, the oxidation of the solution being completed by electrolysis and the solid salt being separated by concentration

and crystallisation. Calcium ferrocyanide is preferably employed, and lime then separates on the cathode and can be used for neutralising the free acid formed by periodic reversal of the polarity of the electrodes. The calcium carbonate mud produced during the pressure oxidation may also be utilised in the anode space for neutralisation of free acid. Double salts containing calcium ferrocyanide may also be used. S. K. TWEEDY.

Manufacture of cupric cyanic and thiocyanic compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 323,802, 10.10. and 1.12.28).—Ammonia is added to copper sulphate solution in amount less than that required to redissolve the precipitate; on treatment with a soluble cyanide or thiocyanate there is formed a complex cupric aminocyanide or diamminothiocyanate. An organic amine (ethylenediamine, pyridine) may be added to a mixture of copper sulphate solution and a soluble cyanide or thiocyanate with a like result.

C. HOLLINS.

Separation of aluminium chloride from other metal chlorides. IMPERIAL CHEM. INDUSTRIES, LTD., N. BENNETT, and H. DODD (B.P. 325,440, 17.5.29).—Aluminium chloride and phosgene combine to form a liquid in which metal chlorides are insoluble and which is stable at ordinary temperature, but is decomposed at 60° into aluminium chloride and phosgene. Aluminium chloride may be prepared from bauxite or the like by treatment with excess of phosgene. S. K. TWEEDY.

Manufacture of catalysts for oxidation of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,234, 8.11.28).—A solution containing one or more neutral salts of metals (e.g., of metals such as aluminium, tin, zinc, forming oxides stable towards hydrogen below 600°; or of iron, cobalt, or nickel), acidified sufficiently to repress hydrolysis and prevent the formation of base-exchange materials, is treated with a large excess of a solution of a salt of an inorganic oxyacid (e.g., an alkali phosphate or borate) capable of precipitating the neutral metal salt, this solution containing caustic alkali in such quantity that a slight excess of alkali remains in the solution after the precipitation. The dried catalyst thus obtained is advantageously brought into contact with a small quantity of corrosive gas (e.g., halogen, hydrogen halide, nitrosyl chloride). S. K. TWEEDY.

Manufacture of active silica. F. STOEWENER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,748,315, 25.2.30. Appl., 4.3.26. Ger., 8.12.24).—See B.P. 270,040; B., 1927, 481.

Production of solid mixtures of alkali hypochlorite and alkali chloride. A. OPPE (U.S.P. 1,748,897, 25.2.30. Appl., 27.2.26. Ger., 23.3.25).—See B.P. 249,510; B., 1927, 42.

Mothproofing material. M. G. MINAEFF, Assr. to LARVEX CORP. (U.S.P. 1,748,579—80, 25.2.30. Appl., [A] 29.11.27, [B] 11.10.28).—See B.P. 301,421; B., 1929, 1012.

VIII.—GLASS; CERAMICS.

Effect of non-plastics (grog) on the shrinkage and porosity of fireclay in the unfired state. S. R. HIND and F. WHEELER (Trans. Ceram. Soc., 1930, 29,

10—29).—Linear measurements of contraction are shown to be unreliable as a basis for the study of essentially voluminear relations. In 30% mixtures decrease in the size of grog increased the ultimate (dry) porosity slightly and the amount of tempering water considerably. The volume of the additional water was proportional to the square root of the surface factor. The effect of porous and non-porous grog on the ultimate porosity was investigated. With non-porous grog porosity decreased in a linear manner with increasing grog content up to 60%, after which it increased to that given by the packing density of the grog alone. With porous grog only a slight decrease occurred up to 60%. A method of plotting contraction data is given, from which the linear contraction for any percentage of grog and initial consistency can be read off directly. Contraction increases with grog-content if the percentage of tempering water is maintained constant. The volume contraction of mixtures of ordinary working consistency is proportional to the percentage of clay present. Examination of the shrinkage-drying curves showed that grog arrests the contraction of clay, the porosity between the grog particles being increased, although the porosity of the mixture as a whole may show a decrease. Porous grog becomes saturated if sufficient time is allowed for maturing, followed by remixing. The clay particles do not penetrate into the pores of porous grog; porous grog mixtures require a longer time to dry than mixtures containing non-porous grog. F. SALT.

Sodium peroxide-carbon fusion for the decomposition of refractories. G. G. MARVIN [with W. C. SCHUMB] (J. Amer. Chem. Soc., 1930, 52, 574—580).—The applicability of the process as a method of decomposing refractory materials has been studied. Experience with 15 highly refractory materials, including both minerals and artificial products, indicates that, when properly carried out, this method is more complete, more rapid, and easier of manipulation than are the usual methods. Nickel crucibles may be employed, and show no loss in weight on repeated use. A mixture of 150 g. of sodium peroxide and 10 g. of sugar carbon is most suitable, and 1 g. of the specimen is mixed with 15 g. of this mixture without bringing any into contact with the crucible wall. Details are given of the method of firing and cooling the crucible, and of the subsequent treatment of the product. The unfused residue from 1 g. of the sample need not exceed 2 mg. and, consisting of undecomposed particles of the original sample, may be subtracted from the weight used. J. W. SMITH.

Burner design for furnaces. SEIL and others.—See II. **Refractory mortars.** PHILIPP.—See IX.

See also A., Mar., 285, **Optical properties and softening of glass** (TAMMANN and HARTMANN). 289, **Sedimentation of clay suspensions** (BOUTARIC and ROY). 315, **Weathering of felspar** (TAMM). **Dehydration and rehydration of kaolin** (SCHACHTSCHABEL).

PATENTS.

Process and apparatus for manufacturing pottery or ceramic articles. J. DENGLER (B.P. 325,613, 4.12.28).

Annular kilns (G.P. 457,413). **Drying apparatus** (U.S.P. 1,741,981).—See I.

IX.—BUILDING MATERIALS.

Multi-stage process for burning Portland cement clinker. R. D. PIKE (Ind. Eng. Chem., 1930, 22, 148—152).—Trials on a working scale, in which cement was burned in stages in two calciners in parallel followed by a rotary kiln, indicated that greater heat economy resulted from passing the hot gases from the rotary kiln direct to waste-heat boilers and a potash recovery plant rather than to the calciners. As the gases are delivered to the boiler at about 960° the latter may be relatively small and of high rating as compared with waste-heat boilers attached to the usual type of rotary kiln. The potash recovery plant dealt with the total gases from ten kilns for more than four years. The gas was passed downward through a spray tower in which the greater part of the dust was removed, though an essential feature was that the potash fume, instead of being wetted by the sprays, was carried on by the cooled and humidified gases to Cottrell precipitators of plate type. Potash was precipitated in flocculent form on to a moving belt which conveyed it through rollers to consolidate the light material containing 31—32% K_2O as sulphate. The slurry from the towers was settled, decanted, and returned to the kiln. The saving of this process over the usual rotary-kiln practice was estimated at 345,000 B.Th.U. per barrel of clinker. C. A. KING.

Refractory mortars. O. PHILIPP (Chem. Fabr., 1930, 86—88).—A mortar for refractory brickwork must have sufficient adhesion when first applied and also must have, after firing, a porosity no more than that of the brickwork, so that slags are not formed. The first requirement is satisfied by the use of fireclay or, if refractory oxides are used instead, by the addition of a binder such as sodium silicate, dextrin, etc. The second requirement demands that the working temperature shall lie above the sintering temperature of the mortar, but below the softening point. In many cases it is necessary to lower the sintering temperature by addition of salt, sodium silicate, asbestos, etc. Refractory mortars for very high temperatures are usually composed largely of the material of the brickwork finely ground and mixed with a cement such as bentonite. Another cement addition is a mixture of starch, powdered glass, and crushed quartz.

C. IRWIN.

Corrosion of [copper] open-valley flashings. K. H. BEIJ (U.S. Bur. Stand. J. Res., 1929, 3, 937—952).

—Corrosion, resulting in a narrow line of holes or cracks immediately under the edge of the overlying roofing, has frequently occurred in copper flashings, particularly open-valley flashings on buildings with shingle roofs situated in large towns near the seaboard. This type of corrosion appears to be an accelerated attack due to differential aeration of the water retained by capillarity between the roofing and the flashing after a fall of rain. The presence of sulphur dioxide and sodium chloride greatly increased corrosion, although the action of either alone is relatively slow. A porous, absorbent, roof material, such as wood shingles, materially increases the rate of corrosion, whereas with slate shingles corrosion is slow as the roof dries quickly. The trouble may be cured by inserting a

smooth hardwood strip between the roofing and the flashing.

A. R. POWELL.

Comparative efficiencies of the components of creosote oil as preservatives for timber. F. H. RHODES and F. T. GARDNER (Ind. Eng. Chem., 1930, 22, 167—171).—Disintegrated wood pulp was suspended in water, collected on a filter, pressed into a pad, and dried. This was saturated with 10 c.c. of a solution of the preservative in ether, dried, and placed in a Petri dish on top of a malt-agar culture of *Fomes annosus* covered with a large watch-glass, the whole being set in a basin of water and incubated at 25°. Complete inhibition of growth was assumed when none occurred within a month. A sample of creosote was separated into bases, acids, and neutral oils, each of which was divided into 10 fractions according to b.p. and the toxicity of each was determined. The neutral-oil fractions were fully as toxic as the tar acids of similar b.p., whilst the bases were less so. Fungicidal power decreased with increasing b.p. above 300° in each case. If the Petri dish was not water-sealed marked loss of creosote through evaporation occurred. The high-boiling compounds if present reduce this loss below the average of the fractions taken separately. Tar acids were much less rapidly volatilised than neutral oils, owing to more thorough adsorption. The results above given as to evaporation losses were confirmed by vapour pressure determinations. Analysis of the fractions by the differential cryoscopic method in benzene solution indicated that the best material contained relatively large proportions of naphthalene and diphenyl together with appreciable quantities of the higher-boiling fractions. Fluorene and phenanthrene have only slight germicidal value, but their permanence increases the effective power of the more volatile components.

C. IRWIN.

Road tar. COOKE.—See II.

PATENTS.

Oven or kiln for baking pulverulent materials such as limestone, cement, etc. J. BELLAY (B.P. 324,858, 4.4.29. Addn. to B.P. 309,177; B., 1930, 191).—The kiln operates as is described in prior patent, but is rectangular in cross-section, and contains flat, rectangular retorts heated for the production of water-gas merely by the heat radiating from materials under treatment; the discharge of the products is effected through rotary, slotted drums through which passes a current of air serving to heat the boiler.

H. ROYAL-DAWSON.

Cementitious product and its manufacture. G. A. WITTE, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,737,904, 3.12.29. Appl., 6.6.27).—Portland cement (2—4 pts.) ground to ordinary fineness (90% through 200-mesh) is mixed with 1 pt. of the cement of a much greater degree of fineness, to produce a high early strength when used.

C. A. KING.

Hydraulic binding agents. CHEM. FABR. GRÜNAU, LANDSHOFF, & MEYER A.-G., Assees. of C. HINRICHSSEN (B.P. 301,509, 30.11.28. Ger., 1.12.27).—The setting of hydraulic cements is accelerated by the addition of a mixture of calcium chloride and hydrochloric acid

and/or an easily hydrolysable chloride, e.g., chlorides of titanium, tin, iron, antimony. Aluminium chloride may replace part of the hydrochloric acid or other chlorides.

C. A. KING.

Reclamation of [used] plaster of Paris. M. I. AISCHE (B.P. 324,374, 26.7.28).—Hydrated plaster from, e.g., potters' moulds is washed with water or suitable chemical reagents to remove impurities and calcined at 90—500°, with or without the addition of gypsum or other strengthening agents.

C. A. KING.

Manufacture of solid bodies from disintegrated alabaster, gypsum, marl, etc. BINDPHAST PRODUCTS, LTD., and J. A. GREENE (B.P. 324,320, 18.10.28).—The pulverised materials are mixed with 15—25% of a 33% suspension in water of equal parts of magnesium chloride and calcined magnesite, together with about 5% of zinc oxide, alum, or sodium silicate. The mass is then moulded to the desired shape.

C. A. KING.

Manufacture of porous brick and the like. H. A. STOCKMAR (U.S.P. 1,740,631, 24.12.29. Appl., 20.6.27).—Refuse (clay, shale, etc.) from coal washeries is ground, mixed with carbonaceous fuel, and moulded into bricks which are burned by the combustible matter contained therein.

C. A. KING.

Manufacture of building bricks, flagstones, etc. A. CHEVRON (B.P. 324,812, 1.2.29).—Cement (20 pts.) is added to moistened clay (80 pts.), made into a paste, and the whole is thoroughly mixed in a kneader and moulded under pressure.

H. ROYAL-DAWSON.

Colouring of stone artificially. G. FUGMANN (B.P. 324,864, 19.4.29).—The stone, especially crystalline stone, e.g., marble, is coloured by impregnating it with a liquid made by adding esters of organic hydroxy-acids, or cellulose esters, to metal compounds dissolved in organic solvents; the solvent is then removed by heat, and the mineral colour precipitate produced as usual.

H. ROYAL-DAWSON.

Composition construction or heat-insulating material [cellular cement]. W. K. NELSON, Assr. to INSULEX CORP. (U.S.P. 1,748,614, 25.2.30. Appl., 11.5.25).—See B.P. 301,359; B., 1929, 130.

Drying cylinders for road-making materials such as granite, sand, and clinker. C. G. HODGSON, and MILLARS' MACHINERY CO., LTD. (B.P. 325,582, 22.11.28).

Apparatus for (A) drying or cooling, (B, C) treatment, of [crushed] stone, slag, etc., for production of road-paving materials. S. L. THORNE, and TILBURY CONTRACTING & DREDGING CO., LTD. (B.P. 325,489—325,491, [A—C] 15.11.28).

Utilisation of waste heat (U.S.P. 1,741,663).—See I. Insulating material (B.P. 324,109).—See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Comparative properties of wrought iron made by hand-puddling and by the Aston process. H. S. RAWDON and D. A. KNIGHT (U.S. Bur. Stand. J. Res., 1929, 3, 953—992).—In the Aston process pig iron from the cupola is desulphurised by the addition of

sodium carbonate to the ladle and then blown in a Bessemer converter, the purified metal being poured into a bath of molten slag of correct composition and the resulting "ball" squeezed into bloom and rolled as usual. Pipe made of this material has been compared with that made from wrought iron produced by the old hand-puddling process, the chemical composition of the irons being almost identical. The carbon content of the Aston iron was much more uniformly distributed than that of the hand-puddled iron and "carbon streaks" were absent. The former had a slightly lower tensile strength and greater ductility than the latter, but otherwise there was little to choose between the mechanical properties of the two irons. The resistance to corrosion, machinability, and welding properties were the same in both cases; thus Aston iron is equal in every respect to hand-puddled iron and can safely be used for the manufacture of pipe to meet all the standard specifications.

A. R. POWELL.

Malleable iron: short-cycle anneal. I. R. VALENTINE (Metals and Alloys, 1929, 1, 233—234).—The influence of time and temperature on the absorption of Fe_3C into solid solution was investigated. The temperature of maximum precipitation of temper carbon is 700—750°. A new method of annealing is described.

CHEMICAL ABSTRACTS.

Peculiar crystal growth in iron and copper and its cause. R. KÜHNEL [with E. NESEMANN] (Z. Metallk., 1930, 22, 53—54).—Two cases of extraordinarily coarse crystal growth in mild steel and one case in firebox copper are described and the cause of trouble (incorrect heat and mechanical treatment) is briefly explained.

A. R. POWELL.

Effect of tensile overstrain on the magnetostriction of steel. J. S. RANKIN (J. Roy. Tech. Coll., Glasgow, 1930, 2, 173—187).—Changes in the magnetostriction of steel which had been overstrained to varying degree have been plotted from observations by an oscillating-valve ultramicroscopic method. High-carbon steels showed less magnetostriction than soft low-carbon steels, but the effect of hardening a steel by tensile overstrain was to increase the magnetostriction considerably, though this change in extension does not depend simply on the amount of tensile extension. From consideration of results in conjunction with the work of Webster (Proc. Roy. Soc., 1925, 109A) it is suggested that when a rod of iron is overstrained the axes of a large proportion of the constituent crystals veer into the direction of the longitudinal axis of the rod, and it is necessary only that a majority of the grains be so arranged to account for the increase in magnetostriction.

C. A. KING.

Creep of steel under simple and compound stresses. R. W. BAILEY (Engineering, 1930, 129, 265—266, 327—329).—Limiting creep stresses as measured at present are not likely, even if they do exist, to have practical importance in the development of high-temperature steam plant. The examination of a tube from a superheater working at 340° and 195 lb./in.² indicated the spheroidisation of cementite which resulted in a reduction of creep resistance. In 0.9% carbon steel the reduction in stress for a given rate of creep was

about 25%, a less value being expected in steels of lower carbon content. Similar structural alteration is likely to be accelerated at higher working temperatures. In view of the probability that creep continues at stresses far below those at which at present measurement is possible, a creep rate of 10^{-8} tensile strain per hour being regarded as of material importance, steels at initial steam temperatures and working stresses must not be classed as permanent and elastic material. Annealing of tubes before use is advisable as annealed steel requires a temperature at least 50° above that of the normal metal for spheroidisation to occur in the same time. Similar results were obtained from creep tests at 540° in air and steam, and oxidation of machined steel was not localised under medium and low stresses. Torsional tests showed that creep by shearing stresses at a plane was not influenced by the normal stress on that plane, and from tests on lead pipes under internal pressure creep may be expected in the direction of the diameter only unless there is superimposed axial stress.

C. A. KING.

Determination of carbon in high-melting alloys, using the high-frequency induction furnace. G. F. SMITH and G. L. HOCKENYOS (Ind. Eng. Chem. [Anal.], 1930, 2, 36—38).—The determination of carbon in tungsten steels, stellites, and similar refractory alloys is greatly facilitated by carrying out the combustion with the aid of a high-frequency induction furnace in place of the usual resistance furnace. The sample (1 g.) surrounded by a weighed coil (6 g.) of soft iron of low (0.01%) carbon content is placed in a combustion thimble of alundum and clay. The whole is placed in a combustion tube of pyrex glass, set vertically in a water-cooled coil of flattened copper tubing. A 3-kw. mercury-arc converter is employed as the high-frequency induction equipment. The sample is burned in oxygen in the usual manner, combustion being started by the arc formed on the coil of soft iron when the power is applied. The method offers the advantages that complete combustion even of refractory alloys is readily attained, and that the sample used may be in the form of chips or very coarse turnings. Full details are given of the complete apparatus employed.

II. F. HARWOOD.

Effect of depolarisation on speed of rusting. F. TÖDT (Korrosion u. Metallschutz, 1929, 5, 169—174; Chem. Zentr., 1929, ii, 2257).—Corrosion by oxygen depolarisation at the cathode surface of local elements is measurable; that found gravimetrically accords with that determined by measurement of the polarisation current at a platinum surface in combination with iron. The corroding iron surface appears to consist of local elements composed of active and passive iron. The practical significance of the experiments is discussed.

A. A. ELDRIDGE.

Splayed molten cadmium coatings in gasoline storage tanks. L. PESSEL (Ind. Eng. Chem., 1930, 22, 119—121).—Corrosion tests were run for 60 days with pieces of steel plate coated with (a) zinc, and (b) cadmium immersed in petrol, to which was added distilled water, sea-water, sulphur, or an organic lead compound with distilled water. The liquids were agitated daily with air. The plates were then photo-

graphed and the sludge was weighed. Uncoated plates showed heavy corrosion and pitting; zinc-coated plates produced a considerable amount of sludge from the zinc, though the steel was undamaged; cadmium-coated plates showed some corrosion with sea-water, but none with the other liquids. The lead compound apparently increases the corrosiveness of petrol. It is considered that cadmium spraying of storage tanks may be worth while in spite of the high price of the metal.

C. IRWIN.

Influence of initial state on variations in hardness resulting from cold-working of certain metals and alloys. GUICHARD, CLAUSMANN, and BILLON (Compt. rend., 1930, 190, 468—470; cf. B., 1930, 331).—Using the method previously described, the relation between deformation (D) and hardness has been investigated for the same metals unannealed. Silver, copper, and copper-nickel alloys gave identical results. Unannealed silver-copper alloys showed increased hardness throughout, the excess decreasing from about 60% for $D = 0$ to 20—35% for $D = 90$; the excess increases as the percentage of copper decreases.

C. A. SILBERRAD.

Kinetics of the eutectoidal decomposition of γ -bronzes. O. DAHL (Z. Metallk., 1930, 22, 48—52).—Various bronzes having a composition within the ($\alpha + \gamma$)- and ($\alpha + \delta$)-ranges have been quenched from above the eutectoid transformation line at 520° and the mechanism of the changes which occur on annealing at 150—400° has been studied by hardness-time and electrical resistance-time measurements. At 220—300° the resistance at first increases rapidly to a maximum, then decreases sharply to an ill-defined minimum, and finally increases slowly and regularly; at 150° only the first increase is observed, and at 400° this increase does not occur at all. The first increase in resistance is attributed to the beginning of sub-microscopic precipitation of α , the subsequent decrease in resistance to the separation of α from the γ -solid solution, and the final increase to the eutectoidal decomposition of the remaining γ into ($\alpha + \delta$). Ageing at the lower temperatures results, therefore, in the formation of a supersaturated γ -phase, which is unstable at higher temperatures and which cannot be obtained by quenching alone. During the precipitation of α in the early stages age-hardening of the alloys occurs to a small extent. Similar phenomena have been observed in a β -aluminium bronze with 11% Al.

A. R. POWELL.

Technical analysis of manganese bronzes. G. SEUFERT (Chem.-Ztg., 1930, 54, 155—156).—The separations of manganese by hydrogen peroxide in ammoniacal solution and by persulphate in acid solution have been examined. In the electrolysis for copper the lead peroxide separated at the anode contains some manganese, from which it is freed by redissolving and again electrolysis. From the combined liquors manganese and iron are thrown down free from zinc, nickel, etc. by double precipitation with hydrogen peroxide in ammoniacal solution, and the manganese is separated by the persulphate method.

S. I. LEVY.

Effect of lead on the ($\alpha + \beta$)- β equilibrium in a 60/40 brass (Muntz metal). L. R. VAN WERT

(Met. and Alloys, 1929, 1, 200—205).—When the alloy (Pb 0.04—1.76, Fe 0.02—0.03%, Sn nil) was quenched in ice water after 0.5 hr. at 750°, lead seemed to confer no added solvent powers on the β -solution; the solubility of α in β is decreased. After 0.5 hr. at 800° no undissolved α -brass was found in the alloy of lowest lead content, but considerable residual α -brass was found in the alloy of highest lead content. When the quenching temperature was 850°, the low-lead brass showed only β -brass; the high-lead alloy still contained α -brass. The presence of lead increases the precipitation of the α -phase from the supersaturated solution formed by quenching.

CHEMICAL ABSTRACTS.

Possibilities of production of radium and vanadium from carnotite. H. A. DOERNER (Ind. Eng. Chem., 1930, 22, 185—189).—The carnotite of Colorado and Utah was worked first for vanadium, afterwards for radium, but the practice is now discontinued. Production costs are discussed and it is concluded that the mechanical concentration of low-grade ore offers the best prospect of cheaper radium production, especially if an ore containing at least 1% U_3O_8 can also be treated for vanadium. The carnotite forms undesirable incrustations on sand grains, which must be removed by roasting. A metal trough with a double row of agitating paddles and fed with jets of compressed air is effective in separating carnotite dust from sand. A concentrate so prepared may be treated with sulphuric, hydrochloric, or nitric acid, but each method involves difficulties. The use of sulphuric acid might be possible if the large excess of acid were recovered by distillation. The alternative method of removing the gangue, principally silica, by alkali fusion is too expensive for a dust concentrate, and removal with hydrofluoric acid also appears doubtful. In the event of carnotite being again worked for vanadium it is urged that radium slimes should be stored for future use.

C. IRWIN.

Preparation of pure electrolytic nickel. I. Elimination of copper from nickel-copper electrolytes. C. G. FINK and F. A. ROHRMAN (Amer. Electrochem. Soc., June, 1930. Advance copy. 14 pp).—The effects of various factors on the current efficiency of copper deposition from solutions of nickel and copper sulphates during electrolysis for 1 hr. have been studied. A high ratio of copper to nickel, high acidity, low current density, high temperature, and vigorous stirring lead to a high efficiency of copper deposition. From a well-stirred N-nickel sulphate solution containing 1 g./litre of copper at 55°, electrolysis at 1 amp./dm.² for a time theoretically sufficient to deposit all the copper actually deposited over 90% of it. The principles underlying the separation and their application to the refining of commercial nickel are discussed.

H. J. T. ELLINGHAM.

Electrolytic cadmium deposits as a rust preventative. B. PLANNER and M. SCHLÖTTER (Z. Metallk., 1930, 22, 41—47).—The grain size of cadmium deposits obtained from various acid and alkaline plating baths with and without addition of colloids has been investigated. Satisfactory deposits are obtained from cyanide baths containing 0.175 g.-mol./litre of cadmium cyanide and 0.57 g.-mol./litre of potassium cyanide, using 50—

100 amp./m.² at 40–60°; addition of 5 g./litre of caffeine reduces the grain size appreciably, but sodium silicate has no action whatever. The current yield is raised by rise of temperature but lowered by increase of current density in alkaline baths. In acid baths the current yield increases with rise of temperature and with the current density, but the grain size of the deposit also increases; hence it is advisable to work with current densities of 100–150 amp./m.² at 40°. The grain size also decreases with increasing mol. wt. of the acid used, being coarse with perchloric, medium with hydrofluosilicic, and fine with phenolsulphonic acid. Bright, lustrous, but very brittle and poorly adherent deposits of cadmium are obtained at 0–15° with high current densities (700–800 amp./m.²) from acid baths containing organic colloids. A cadmium deposit of only 22 g./m.² is stated to protect iron from corrosion in the salt-spray test more efficiently than a zinc deposit of 400–500 g./m.² A. R. POWELL.

Cadmium plate. R. HOFFELT (Korrosion u. Metallschutz, 1929, 5, 176–180; Chem. Zentr., 1929, ii, 2259).—The use of cadmium plate for culinary utensils is considered. In presence of acids, e.g., acetic, considerable amounts of cadmium are dissolved. For most domestic utensils cadmium-plated iron is, however, specially suitable; cadmium-tin plate is recommended.

A. A. ELDRIDGE.

Photo-electrometallurgy. R. W. DRIER (Ind. Eng. Chem., 1930, 22, 153–157).—The possibility of separating minerals or other materials on the basis of colour or lustre was tried by conveying screened particles on a moving belt under a highly illuminated area. Reflected light from certain particles actuated a photo-electric cell, and the current produced was relayed to operate a picking-up device which separated the particles of high reflecting power. In this way 60% of the silver in a mixture of silver and copper was separated, the silver to copper ratio being improved from 1 in 100 to 1 in 10. Separation of a mixture of babbitt metal and brass was less successful as the indices of reflection were too close, but by treating the mixture with hydrogen sulphide a wider difference was obtained. Separation of other minerals was effected, but the principal difficulty lies in the mechanical design of the separating device. C. A. KING.

Change of volume of cast iron during solidification. K. HONDA, T. KASE, and Y. MATSUYAMA (Sci. Rep. Tôhoku, 1930, 18, 699–714).—See B., 1930, 14.

p_H control. MAGNUS.—See I. Burner design for furnaces. SEIL and others.—See II. Corrosion of copper. BEIJ.—See IX. Measurement of corrosion. GOLLNOW.—See XI. Determination of cobalt. HEIM.—See XIII.

See also A., Mar., 284, **X-Ray analysis of system nickel-bismuth** (HÄGG and FUNKE). **System lead-germanium** (BRIGGS and BENEDICT). **System silver-copper-zinc** (UENO). 285, **Ternary system Cu-Sn-Sb** (TASAKI). 299, **Electrolysis of alumina** (WASILEWSKI and MANTEL). 304, **Electrodeposition of chromium** (MÜLLER and ESSIN). 305, **Electrolytic preparation of metals from fused borates** (ANDRIEUX). 309,

Pyrophoric iron (FINZEL). 311, **Reagent for sulphide minerals** (WILKE-DÖRFURT).

PATENTS.

Hot-blast cupolas. (A) C. D. BARR and W. D. MOORE, (B) W. D. MOORE, Assrs. to AMER. CAST IRON PIPE CO. (U.S.P. 1,740,886 and 1,740,900, 24.12.29. Appl., [A] 24.3.28, [B] 15.11.27. Renewed [A] 9.11.29).—(A) The hot gases from just above the melting zone are withdrawn from the furnace through an annular space formed by a suspended tubular apron spaced from the wall and having pipes or flues leading from it to a point in the cupola above the charging door. In this way deposition of sulphur from the gases on to the descending charge is avoided. (B) The cylindrical body of the furnace is built up of a number of vertical, hollow sections with a transversely curved shape arranged with their edges abutting, and each being independently connected to a bustle pipe and to a tuyère to conduct air through the section from a blower to the furnace. This construction admits of ready replacement of worn parts without dismantling the whole furnace. A. R. POWELL.

Reverberatory furnace. W. F. SKLENAR, and BRIT. REVERBERATORY FURNACES, LTD. (B.P. 324,339, 22.10.28).—In a reverberatory furnace with a hollow bed and a combustion chamber above, the tapping hole is above the normal level of the metal. From the other end of the bed a vertical shaft is provided with a charging door, above which pipes may be inserted to preheat the air required for combustion, or the shaft may be utilised for heating ladles of metal after tapping. C. A. KING.

Operation of an open-hearth furnace. W. TRINKS (U.S.P. 1,741,002, 24.12.29. Appl., 22.6.23).—Air ports are arranged symmetrically on either side of a central gas port, the normal air supply meeting the gas at the mouth of the gas port. From each air port a narrow duct leads air into the gas port and a jet of compressed air is used to increase the velocity in these ducts. At the outgoing end of the furnace the jet of compressed air is maintained to prevent flue gases passing through the secondary air ducts. C. A. KING.

Open-hearth furnace structure and its operations. R. B. KERNOHAN, J. S. LOCHHEAD, and W. TRINKS (U.S.P. 1,741,025, 24.12.29. Appl., 1.10.21).—In an open-hearth furnace having a centrally disposed gas port and an air port above, small air ducts branch from the rising air-main into the gas duct. Jets of compressed air in these subsidiary ducts increase the velocity of the air current. C. A. KING.

Open-hearth furnace. R. B. KERNOHAN and J. S. LOCHHEAD (U.S.P. 1,741,024, 24.12.29. Appl., 29.10.20).—Regenerated air at a pressure of about 1.5 in. water-gauge is introduced into an open-hearth furnace through a port centrally situated, near the end of which port producer gas is also admitted to effect better mixing and a more concentrated flame to be directed on to the bed of the furnace. Slag pockets are provided in conjunction with the port. C. A. KING.

Open-hearth furnace. S. TREVERTON, Assr. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,741,666, 31.12.29. Appl., 8.9.27).—The walls of the furnace are stepped

so that the lining may be renewed with loose material at its natural angle of repose. The buckstaves are cranked to avoid large masses of idle brickwork filling.

B. M. VENABLES.

Multiple-hearth roasting furnace. A. E. WHITE. From NICHOLS COPPER CO. (B.P. 324,471, 5.12.28).—In addition to hollow rabble arms the vertical, central shaft of a shelf roasting furnace is provided with sockets into which heat-absorbing arms may be fitted as required to cool certain hearths. Air which has circulated through the rabble or the cooling arms may be conducted down an annular space in the shaft to heat similar arms over the cooler hearths lower down the furnace.

C. A. KING.

Roasting and agglomeration of fine ore or roasting residues. SOC. POUR L'ENRICHISSEMENT ET L'AGGLOMERATION DES MINERAIS (B.P. 307,708, 11.3.29. Ger., 10.3.28).—A furnace for roasting fine ores consists of a horizontal, endless, chain-grate conveyor totally enclosed so that uniform suction may be applied under the length of grate on which the material is roasted. The grate is stationary during the actual roasting period, and is moved only during the charging and discharging operations. Ore may be roasted direct on the grate or on a bed of protective material, and is ignited by means of burners arranged above the grate.

C. A. KING.

Continuous process and furnace for the thermal treatment of metals etc. INFRA, ASSEES. OF V. SORREL and L. A. LAFONT (B.P. 306,446, 2.10.28. Fr., 20.2.28).—An electrically heated, inclined, tube-furnace is provided at one point in its length with either natural or electro-magnets for the purpose of retaining magnetic materials within the furnace so long as they remain magnetic. When demagnetised by a sufficient duration of heating the material falls through the furnace by gravity either into an auxiliary chamber for further heat-treatment or into any suitable cooling medium.

C. A. KING.

Annealing and heat-treating furnaces. A. T. KATHNER (B.P. 320,841 and 320,865, 16.4.28).—The furnace comprises a heating chamber and a cooling chamber in horizontal alinement, the cross-sectional area of the latter being less than that of the heating chamber and its length one third greater than that of the heating chamber. The tops of both chambers are composed of a number of arched sections which can be individually raised or removed for admitting cooling air or for cleaning the conveyor mechanism which passes along the floor of both chambers (cf. U.S.P. 1,669,902; B., 1928, 527).

A. R. POWELL.

Treatment of steel castings or other metals. G. HERRMANN and L. ZERZOG (B.P. 325,307, 26.11.28).—After annealing, a casting is cooled uniformly and rapidly by either blowing or sucking air through the hollow travelling hearth on which the casting rests, thereby causing a distribution of cool air around the metal. The hearth may be built up of blocks having both horizontal and vertical air-passages.

C. A. KING.

Heat-resisting [iron] alloy. OESTERR. SCHMIDT-STAHLWERKE A.-G. (B.P. 305,047, 28.1.29. Austr., 28.1.28).—The alloy comprises iron with 10–25% Cr,

0–10% Al, 1–10% Cu, 0.8–2.5% C, and the usual small amounts of manganese, sulphur, and phosphorus. A preferred composition is 23.5% Cr, 2% Al, 3.5% Cu, 1.1% Si, 0.35% Mn, and 2.25% C. A. R. POWELL.

Preparation of blende for sintering. SOC. ANON. LA NOUVELLE MONTAGNE (B.P. 325,455, 27.6.29. Belg., 5.1.29).—The ore is moistened with zinc sulphate solution, preferably obtained by the action of sulphuric acid on the flue dust of zinc ore roasting furnaces or on zinciferous residues, and the mass is passed through an extrusion press to obtain it in a vermicular form for roasting.

A. R. POWELL.

Non-corrodible alloy. H. ANDRÉ (B.P. 310,962, 23.4.29. Fr., 5.5.28).—An alloy containing 20–25% (23%) Si and 80–75% (77%) Ni is claimed.

H. ROYAL-DAWSON.

Light-metal alloy. J. WEISS (B.P. 309,538, 18.3.29. Austr., 12.4.28).—Alloys suitable for the pistons of internal-combustion engines contain aluminium together with 12–18% Cu, 0.1–5% Mg, and 0.1–5% Ni; 0.05–8% Fe may also be present.

H. ROYAL-DAWSON.

Magnetic alloy. W. S. SMITH and H. J. GARNETT (U.S.P. 1,748,311, 25.2.30. Appl., 17.7.28. U.K., 27.7.26).—See B.P. 279,549; B., 1928, 20.

Smelting of [zinc] ore and other zinc-containing materials. W. JOB, ASSR. to AMER. LURGI CORP. (U.S.P. 1,748,450, 25.2.30. Appl., 28.6.26. Ger., 2.7.25).—See B.P. 268,201; B., 1927, 415.

Treatment of lead-zinc sulphide ores, mattes, etc. E. A. ASHCROFT, ASSR. to MAGNESIUM CO., LTD. (U.S.P. 1,748,748, 25.2.30. Appl., 28.6.23. U.K., 2.6.23).—See B.P. 216,695; B., 1924, 637.

Method of performing [internal] brazing, soldering, and similar operations. UNIQUE & UNITY CYCLE CO., LTD., and J. MUNN (B.P. 325,647, 1.1.29).

Tube-mill (U.S.P. 1,741,604).—See I. **Corrosion of copper flashings.** BEIJ.—See IX. **Electric welding** (G.P. 460,615). **Magnetic materials** (B.P. 323,222).—See XI.

XI.—ELECTROTECHNICS.

Measurement of the corrosion of metals. G. GOLLNOW (Chem.-Ztg., 1930, 54, 110).—The Tödt "corrosimeter" (cf. Köhler, A., 1929, 528, 1034) measures the "oxygen depolarisation current" developed in a cell in which one pole is the sample to be tested and the other a platinum foil immersed in aqueous solution. The author has modified this apparatus by providing for two test samples, either of which can be switched into circuit. Comparative results can thus be accurately obtained with elimination of all constant sources of error. The arrangement is also convenient for determining the variation of corrosion with surface area.

C. IRWIN.

Determination of the volatile wood acids corrosive to lead cable sheath. R. M. BURNS and B. L. CLARKE (Ind. Eng. Chem. [Anal.], 1930, 2, 86–88).—An investigation of cases of corrosion of lead cable sheath in creosoted Douglas fir conduit revealed the presence of acid vapours which were eventually traced

to the wood. A method has been developed for the extraction and determination of these wood acids, consisting in the condensation of the volatile constituents of wood sawdust removed under reduced pressure, and titration of the condensate, using a modified differential potentiometric electrode (cf. Burns and Campbell, B., 1929, 561). Acidity data have been obtained for various woods, and a correlation is attempted between these acidities and the observed corrosive character of the woods. The volatile acid content of western yellow pine, Douglas fir, and spruce was high; of southern yellow pine, much less; whilst of hemlock and cedar, it was very low or non-existent. H. S. GARLICK.

Mica insulation. J. M. MACAULAY and D. CARSON (J. Roy. Tech. Coll., Glasgow, 1930, No. 2, 161—173).—Contrary to previous observations (*ibid.*, 1927, No. 4), no change was detected in the insulating property of mica when a freshly split surface was exposed in a dry vacuum, in clean dry air, or in air-free water vapour until a high degree of saturation was attained. Freshly peeled mica in a moist atmosphere conducts, but may be rendered temporarily insulating by heating to 15° above atmospheric temperature or by a reduction in the moisture content of the atmosphere. An old surface shows conductivity at 200°, increasing to 400°, and on cooling reacts similarly to a freshly split surface. The effect is considered to be due to a temporary layer (water vapour and dust) attracted to a new surface, and perhaps also to a permanent layer removed only by a considerable degree of heating. These conducting surface layers, whether temporary or permanent, are not to be regarded as simple liquid electrolytes owing to the strong forces of cohesion between the surface layer and the mica.

C. A. KING.

Determination of carbon in alloys. SMITH and HOCKENYOS. **Beryllium.** MARCHAL. **Electrolytic nickel.** FINK and ROHRMANN. **Cadmium deposits and rust prevention.** PLANNER and SCHLÖTTER. **Photo-electrometallurgy.** DRIER.—See X. **Electrolytic iron oxide pigment.** BRUCKNER.—See XIII.

See also A., Mar., 281, **Ruthenium as superconductor** (McLENNAN). 299, **Electrolysis of alumina** (WASILEWSKI and MANTEL). 304, **Electrodeposition of chromium** (MÜLLER and ESSIN). **Electrolytic preparation of amorphous boron, borides, and metals** (ANDRIEUX). 315, **High-frequency field** (RHEINBOLDT and HESSEL). 335, **Electrolytic reduction of nitriles** (OGURA). 337, **Electrolytic reduction of 2-nitro-4'-hydroxyazobenzene** (ITOMI).

PATENTS.

Electric induction furnace. E. F. NORTHRUP (B.P. 313,044, 10.5.29. U.S., 5.6.28).—An exploring coil connected to a source of current is arranged in or around the refractory wall of the receptacle for molten metal, and a protecting or warning device, preferably actuated by change in the resistance of the coil circuit, is inserted in that circuit, and is operative on a fault occurring in the refractory permitting leakage of molten metal. J. S. G. THOMAS.

Electric induction furnace. SIEMENS & HALSKE A.-G. (B.P. 301,894, 7.11.28. Ger., 8.12.27).—The

furnace space is enclosed within an induction coil of solid material, preferably made of cylindrically-wound, multiple-crossed, copper bands or ribbons insulated from each other, the coil being carried by rod-shaped insulators and immersed in oil. J. S. G. THOMAS.

[Rotary, heat-treatment] electric furnace. BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 324,609, 28.1.28. Cf. B.P. 314,775; B., 1929, 726).—The rotary, work-preheating conveyor of the prior patent is enclosed partly within a stationary insulating casing and partly within the stationary heating chamber of a furnace structure which normally is secured to the insulating casing, but is adapted to be separated therefrom and withdrawn clear of the conveyor, to afford access to the interior of the heating chamber. J. S. G. THOMAS.

[Automatic regulation of] electric induction heating installations. P. VIRY (B.P. 302,235, 7.12.28. Fr., 12.12.27).—Automatically operated mechanical parts of furnaces are mechanically driven from the converter group generating the heating current. In cases in which the speed of the converter groups cannot be controlled, the supply voltage is suitably regulated by hand to a value determined by reference to two scales graduated, respectively, to indicate frequencies and temperatures, and a co-operating scale of volts. J. S. G. THOMAS.

Electric welding process. E. SCHROEDER (G.P. 460,615, 16.10.23).—The welding is effected by high-frequency induction from a coil surrounding the parts to be joined; if necessary a solder may be used.

A. R. POWELL.

Electric accumulator plates. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 324,163, 20.11.28).—The lead paste is prepared with glycol, in place of glycerin. Thus a mixture composed of 100 pts. of lead dust (containing about 65% PbO), 2.5 pts. of finely-powdered barium sulphate, and 20 pts. of dilute sulphuric acid, is kneaded with 3 pts. of ethylene glycol and filled into a grid-skeleton. J. S. G. THOMAS.

[Cathodes for] secondary electric cells. L. A. LEVY, and ALMEIDA ACCUMULATORS, LTD. (B.P. 324,415, 30.10.28).—Cathodes for use in secondary electric cells in which zinc is deposited at the cathode during charging are prepared by immersing the cathode base, preferably of iron, in a bath of molten zinc-mercury alloy. Thus a sheet of Armco iron is degreased and immersed in a molten zinc amalgam composed of 4 pts. of electrolytic zinc and 5 pts. of mercury at 370°. J. S. G. THOMAS.

[Selenium] cells which are sensitive to infra-red rays. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 311,662, 13.5.29. Ger., 11.5.28).—Extremely thin layers of selenium and heavier metals, e.g., tellurium or bismuth, are deposited by cathodic sputtering upon a support and then thermally treated. The thickness of the sensitive layer is not considerably greater than the depth of penetration of the radiation. J. S. G. THOMAS.

Arrangement for treating liquids with ultra-violet rays. PATENT-TREUHAND GES. F. ELEKTR.

GLÜHLAMPEN M.B.H., and K. WIEGAND (B.P. 324,503, 7.1.29).—Fine streams of liquid (especially milk) fall vertically through a shaft or passage surrounded by an upright-glow gas-discharge tube, so that the streams do not come into contact with the inner wall of the discharge tube, and are subjected to radiation on all sides. J. S. G. THOMAS.

Manufacture of vacuum tubes. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of T. S. FULLER (B.P. 300,265, 22.10.28. U.S., 11.10.27).—An alloy of aluminium and an oxidisable metal, *e.g.*, barium or strontium, is introduced into the tube prior to sealing, and is heated to such a temperature that the oxidisable metal volatilises, leaving the aluminium substantially unaffected. The volatilised metal is subsequently deposited as a thin film upon the wall of the tube and is used for absorbing gas. [Stat. ref.]

J. S. G. THOMAS.

[Gas-filled] electric incandescence lamps. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. R. FONDA (B.P. 312,695, 31.5.29. U.S., 31.5.28).—Destructive arcing from a burnt-out filament is prevented by arranging arc-quenching substances, *e.g.*, the nitrate or sulphate of copper, nickel, or barium, in the path of the arc from the filament to the base of the lamp. The volatilisation of these substances causes such a rise in the voltage necessary to maintain the arc that the arc is extinguished. J. S. G. THOMAS.

[Electrical] insulating material. BINDPHAST PRODUCTS, LTD., and J. A. GREENE (B.P. 324,109, 18.10.28).—A mixture of pulverised base material, *e.g.*, slate, slate waste, quarry and colliery refuse, from which metallic impurities have been removed, together with magnesium chloride, calcined magnesite, and, if desired, a small proportion of alum and/or asbestos is moulded and dried. J. S. G. THOMAS.

Manufacture of insulated plates of magnetic material for electrical purposes. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of J. E. GROSS (B.P. 300,911, 19.11.28. U.S., 19.11.27).—Insulating coatings consisting of an aqueous solution of sodium silicate (*d* 1.15—1.3) and glycerin, and/or carbohydrate compounds, *e.g.*, sugar, together with, if desired, a filling material, *e.g.*, talc, mica, aluminium oxide, are applied to plates of magnetic material and baked at 120—150°.

J. S. G. THOMAS.

Magnetic materials [nickel-cobalt-iron alloys]. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 323,222, 24.9.28).—Alloys containing 20—54.5% Ni, 20—40% Fe, and 15—50% Co, with up to 10% Mo, W, V, Ta, Mn, Zr, Cu, or Si are claimed. A preferred composition is 45% Ni, 25% Co, 23% Fe, 7% Mo, and 0.5% Mn. The alloy is heated at 1100° for 1 hr., cooled to 600° during 3 hrs., then to 400° over a further 2 hrs., reheated at 600°, and cooled to the ordinary temperature at a rate of 5—25°/sec. (cf. B.P. 273,638; B., 1928, 97).

A. R. POWELL.

Electric induction furnace. W. ESMARCH, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,748,706, 21.11.28. Ger., 8.12.27).—See B.P. 301,894; preceding.

Electrolyte for lead accumulators and method

of using the same. M. KUGEL (U.S.P. 1,748,485, 25.2.30. Appl., 16.8.27. Ger., 6.11.26).—See B.P. 280,197; B., 1928, 864.

Apparatus for generating Röntgen rays. SIEMENS-REINIGER-VEIFA GES. F. MEDIZIN. TECHN. M.B.H. (B.P. 314,369, 20.11.28. Ger., 26.6.28).

Electrodes [anode] for electric-discharge devices. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of M. A. ACHESON (B.P. 307,053, 24.1.29. U.S., 2.3.28).

Conductivity of fluids (B.P. 325,277).—See I. **Oil-purifying apparatus** (U.S.P. 1,741,756).—See II. **Liquid hydrocarbons from ethylene** (B.P. 295,705).—See III. **Laminated products** (B.P. 301,428).—See V. **Ferricyanides** (B.P. 314,956).—See VII. **Thermal treatment of metals** (B.P. 306,446).—See X.

XII.—FATS; OILS; WAXES.

Method of determining the mol. wt. of higher saturated fatty acids and its application to the determination of lignoceric acid in mixtures containing peanut butter. J. GROSSFELD (Z. Unters. Lebensm., 1929, 58, 209—261).—The higher fatty acids are precipitated as the lead soaps from the alcoholic solution of soaps from 10 g. of the fat and the precipitate is repeatedly crystallised from 150—200 c.c. of hot alcohol. The free fatty acids, purified by dissolution in alcoholic potash and reprecipitation, are dissolved in neutral alcohol, the solution is carefully neutralised to phenolphthalein by means of 0.5*N*-alcoholic potash, and an equal volume (50 c.c.) of neutral ether is added to the warm solution. If a finely-crystalline precipitate forms on cooling, only negligible quantities of acids higher than stearic acid are present; increasing amounts may give a flocculent or a gelatinous precipitate. After 2 hrs. a further 100—150 c.c. of ether are added, and several hours later the precipitate is filtered into a crucible, washed with ether, sucked dry, dried (in air, then at 60°, and finally at 100—105°), cooled, and weighed. The bulk of the dry potassium salts, accurately weighed, is dissolved in a warm mixture of alcohol and acetic acid in quantities dependent on the weight of the potassium salt (tabulation given). To the solution chloroform and, dropwise with shaking, aqueous 20% perchloric acid are added in quantities also to be derived from the table. The flask is corked and its contents are allowed to cool. The precipitate is filtered off in a tared asbestos-kieselguhr Gooch crucible previously dried at 120—150°, washed with a saturated solution of potassium perchlorate in 1:1 alcohol-ether, dried in a steam-oven, cooled, and weighed. The potassium chlorate value (*k*) is calculated from the formula: $k = \{100C(w + s)\}/W$, where *w* is the weight of potassium perchlorate, *s* a correction for the solubility of potassium perchlorate, *C* a factor derived from a table prepared experimentally, using pure stearic acid, and *W* the weight of potassium salts used. The mean mol. wt. (*M*) of the fatty acids is derived from $M = (13,856/k) - 38.09$ and the lignoceric acid content from $L = 16.63 \times (39.54 - k)$ or $L = 6.81(42.98 - k)$, the former being used when $k < 37.16$, the latter when $k > 37.16$. It is assumed that only salts of stearic, lignoceric, and

arachidic acids are present, and that the ratio of lignoceric acid to arachidic acid is constant. These calculations may be obviated by the use of tables provided. Peanut oil is conveniently determined in mixtures of fats by the content of lignoceric acid. The method described gives correct results within 0.04% of lignoceric acid or 2–4% of peanut oil. Lignoceric acid is absent from cacao butter, lard, suet, butter, hazel-nut oil, walnut oil, almond oil, apricot-kernel oil, illipé butter, and karité fat. With certain modifications the method can be used for determining the various higher fatty acids in presence of each other and for preparing pure fatty acids. The behenic acid content (B) of hardened train oil can be determined from the formula $B = 14.99(42.98 - k)$.

W. J. BOYD.

Constitution of rice-husk fat. E. DE'CONNO and L. FINELLI (Annali Chim. Appl., 1930, 20, 26–29).—The percentage composition of the rice husk used was: moisture 10.57, protein 13.50, fat 15.13. The fat had m.p. 24–25°, acid value 101.25, saponif. value 189.125, glycerin 4.92, Reichert value 5.2, Hühner value 95.125, iodine value 100.77, unsaponifiable matter 1.415. The fat contains 39% of liquid, and 61% of solid, glycerides. The acids of the former class consist mainly of oleic acid, together with about 12% of linoleic acid; those of the solid glycerides are 42.7% of a C_{20} acid, m.p. 75°, probably arachidic acid; 39.65% of a C_{18} acid, m.p. 69° (? stearic acid), and 11.4% of a C_{16} acid, m.p. 62° (? palmitic acid).

T. H. POPE.

Fat analysis. A. HEIDUSCHKA and J. MÜLLER (Arch. Pharm., 1930, 268, 145–147).—Light petroleum extraction should be used to determine the unsaponifiable matter in the product of hydrolysis of fats of unknown constitution only after the results have been checked by ether extraction. Thus the former method gives 1.5 and the latter 3% of unsaponifiable matter in laurel fat (cf. B., 1930, 291), the deficiency in the first instance being due to the non-extraction of myricyl alcohol. The value of distillation in a high vacuum as a method for the quantitative separation of fatty acid mixtures is discussed.

H. E. F. NOTTON.

Effect of method of rendering on the refractive index of fats. H. W. BLOCK (J. Assoc. Off. Agric. Chem., 1930, 13, 142–146).—The refractive index is very slightly increased on heating for 6 hrs. or more in air at 100°, but is otherwise independent of the method of rendering.

K. V. THIMANN.

Sulphur-treated and blown linseed oil. A. HOLLANDER (Farben-Ztg., 1930, 35, 998–1000).—It is shown that sulphur treatment reduces the tendency to swell in water of linseed oil to a degree comparable with stand oil, and under certain experimental conditions the water-resistance of tung oil is attained. Sulphur in "sulpho-oils" is combined by association and is not taken up at the double linkings, the iodine value being maintained or increased slightly. By examination of the oil after coagulation with iodine and chloroform, it is established that the sulphur is irregularly distributed in the oil molecule. Owing to its "condensed" state (as evinced by water-resistance) a "sulpho-oil" does not dry as quickly as the oil from which it is derived,

in spite of its iodine value, and its oxidative degradation is retarded. Its behaviour compares with that of a stand oil rather than of a blown oil, and valuable oils are obtained by "vulcanising" blown oils.

S. S. WOOLF.

Detection and determination of tung oil. A. GÉRARD (Ann. Falsif., 1930, 23, 28–30).—The n_D , optical dispersion, and hexabromide test allow of the identification of tung oil in mixtures of oils, and Bolton's modification of Worstall's test is used in conjunction with these values. The proportion of the oil in a varnish is most satisfactorily found by the von Reibnitz test (B., 1928, 936) with iodine; Scheiber's stannic chloride test and Goldsmith's test (B., 1927, 118), which makes use of sodium nitrite in the presence of sulphuric acid, may also be employed.

D. G. HEWER.

Castor oil and ricinoleic acid. A. HEIDUSCHKA and G. KIRSTEN (Pharm. Zentr., 1930, 71, 81–91).—An exhaustive qualitative and quantitative examination of a castor oil was undertaken, full experimental details of which are given. The percentage composition of the oil was found to be: ricinoleic acid 82, α -linoleic acid 1.4, oleic acid 6.8, dihydroxystearic acid (m.p. 142.5°) 1.3, stearic acid 3.4, glyceryl residue (C_3H_5) 4.1, unsaponifiable matter 0.3. No evidence could be obtained for the existence of any other saturated acids, β -linoleic, or linolenic acids. By cold saponification of the oil and fractional crystallisation of the precipitated barium salts from alcohol, pure ricinoleic acid (m.p. 3°) was obtained having iodine value 85.39 (calc. 85.1) and acid value 188.25 (calc. 188.1). The acid remained clear and transparent even after long keeping. On vacuum distillation it yielded a constant-boiling fraction at 233–234°/14 mm. which was shown to be a mixture of liquid decomposition products (cf. Böeseken, A., 1927, 1169), and on analysis gave 77.10% C and 11.39% H, approximating to that of an acid $C_{18}H_{32}O_2$ (cf. Krafft, A., 1888, 1270; Mangold, A., 1894, i, 492), with which, however, it cannot be identified since the iodine and neutralisation values of this acid (191.1, 200.2, respectively) differ widely from the corresponding values obtained for the distillation product (141.4, 176.7). Free ricinoleic acid was found to have no purgative effect; the action of castor oil is possibly a consequence of the formation of nascent ricinoleic acid in the digestive tract.

E. LEWKOWITSCH.

Butter fat in ice-cream. CHASE and KING. Iodine in butter, oil, etc. MCCLENDON and others.—See XIX.

See also A., Mar., 292, Sodium oleate and zoomarate (HIROSE and SHIMOMURA). 317, Illipene, and high alcohols in illipé butter (TSUJIMOTO). 319, Kanyl alcohol; a new alcohol in liver oil of "Tarabakani" (TSUJIMOTO). 321, Synthetic glycerides (AVERILL and others). 322, Transformation of fatty acids into hydrocarbons (PETROV). 362, Fats of Japanese birds (KOYAMA). Unsaturated fatty acids of human liver (MÜLLER).

PATENTS.

Autonomous apparatus for neutralising and bleaching heated oleaginous liquids under vacuum.

E. BATAILLE (U.S.P. 1,748,274, 25.2.30: Appl., 6.10.24. Fr., 30.4.24).—See B.P. 233,345; B., 1926, 448.

Treatment of fatty materials (F.P. 553,338 and Addn. F.P. 32,762).—See II. **Wool fat from wash liquors** (B.P. 304,718).—See V. **Paste for proofing textiles** (B.P. 295,660).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Experimental determination of brightness—film thickness curves of wet paints. D. L. GAMBLE and A. H. PFUND (Ind. Eng. Chem. [Anal.], 1930, 2, 63—66).—The chief weakness in existing procedures for the determination of such curves lies in the film-thickness measurement, *e.g.*, by the micrometer gauge. An optical method based on the channelled spectrum phenomenon was developed, the thickness of a wedge of wet paint being measured in a modified Pfund cryptometer. A photometer of the Macbeth type was used in making brightness measurements. Existing mathematical expressions for brightness—film thickness relationships were investigated and their limitations are indicated. Curves and data for typical white pigments are given.
S. S. WOOLF.

Measurement of the levelling properties of paints and enamels. G. S. HASLAM and L. D. GRADY, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 66—69).—Levelling is mainly a function of the flocculation of the pigment and to a lesser extent of the surface forces of the paint which govern flow after the high shearing forces of application have been removed (and which are masked in normal consistency determinations). The du Noüy tensiometer was used for measuring these levelling forces, and results obtained as in measuring surface tension (although it is shown that properties other than surface tension are actually being recorded) were found to agree with practical trials by a painter. Comparison with consistency readings, as determined by five types of apparatus, shows the superiority of the tensiometer in this connexion, differentiation between paints with the same "flow constants" being possible.

S. S. WOOLF.

Growth-inhibitive and preservative value of poisonous paints and other substances. J. H. ORTON (J. Marine Biol. Assoc., 1930, 16, 373—452).—Investigations have been carried out on the efficiency of paints and protective coatings of various kinds in preventing the growth of marine organisms. The properties and composition of an ideal anti-fouling paint are discussed.
C. W. GIBBY.

Determination of cobalt in driers, japans, and alloys. O. HEIM (Ind. Eng. Chem., [Anal.], 1930, 2, 38).—The material (10 g.) is oxidised with sulphuric acid and 30% hydrogen peroxide and the excess acid expelled. The solution is diluted, neutralised with ammonia, and made slightly acid with hydrochloric acid. Zinc oxide is then added to the solution at 50° until just a trace remains undissolved (a few drops of ferric chloride being previously added if iron be absent) and the liquid filtered. The filtrate is evaporated to 20 c.c., transferred to a separating funnel, and 30 g. of ammonium thiocyanate are added. The cobalt is

removed by repeated extraction with ether and amyl alcohol (9:1), the extract shaken with 20 c.c. of 10% sulphuric acid, and the cobalt in solution determined electrolytically. Fischer's method may also be employed to determine the cobalt. In this a cold 2% solution of 3:5-dimethylpyrazole is added to the almost neutralised solution containing the cobalt, followed by 5 c.c. of 0.5*N*-sodium hydroxide. The purple precipitate of the cobalt compound is collected on a Gooch crucible, washed with cold water, dried at a low temperature, and weighed as $(C_5H_7N_2)_2Co$.
H. F. HARWOOD.

Electrolytic production of Turkey-red iron oxide pigment. W. H. BRUCKNER (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.).—Experiments are made to find the conditions under which a fine-grained iron oxide precipitate of Turkey-red colour can be produced by electrolytic means. Solutions of sodium hydroxide, chloride, or sulphate, or mixtures of these substances, at various concentrations were electrolysed in a diaphragm cell using iron or steel anodes of various qualities and employing various current densities. The best results were obtained by using a wrought-iron anode in a diaphragm cell containing sodium sulphate solution (40—60 g./litre) at 30—40° with a current density of 20—25 amp./ft.² The anolyte and catholyte are continuously withdrawn, mixed together, and, after filtering off the precipitate, the solution is returned to the cell. The precipitate is dried and heated for 5 min. at 575—625°. The desired colour was not obtained if sodium chloride was used as the electrolyte, and in any case traces of impurity in the anode material and the conditions of calcination greatly affect the colour of the product.

H. J. T. ELLINGHAM.

Microscopical measurements for the determination of particle size of pigments and powders. E. J. DUNK, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 59—62).—Particles ranging from colloidal dimensions to sieve sizes may be counted and measured by a combination of settling, micro-projection, and ultramicroscopy. Mounting, focussing by remote control, particle counting, and calculation of results are discussed, and the necessary apparatus is described in detail.
S. S. WOOLF.

Use of protective colloids in colorimetric determination of certain metals as lakes of dyes. W. E. THRUN (Ind. Eng. Chem. [Anal.], 1930, 2, 8—9).—A solution of gum arabic or "starch glycerite" (preparation described) prevents the flocculation of many lakes formed by aluminium, magnesium, and other metals, and may be advantageously employed in the colorimetric determination of these latter. The addition of "starch glycerite" in the proportion of 1 c.c. to 12 c.c. of solution also increases the intensity of the colour produced in the case of the curcumin lake of beryllium (*cf.* A., 1928, 386) and renders the test more sensitive.

H. F. HARWOOD.

New application of the Abbé refractometer in the analysis of lacquer thinners. J. D. JENKINS (Ind. Eng. Chem. [Anal.], 1930, 2, 127—128).—The Abbé refractometer dispersion-scale readings for common aliphatic solvents and diluents average 41.4, whilst the aromatic hydrocarbons approximate to

35.6, the value for toluene (with, however, a more marked variation). Since the large majority of lacquer thinners contain toluene as the only aromatic hydrocarbon, the dispersion-scale reading of mixtures can be taken as a straight-line function of the volume composition. The presence of higher homologues of toluene can be detected by odour and evaporation characteristics, in which case the refractometer reading should be checked by a determination of the residue insoluble in 75% sulphuric acid. Petroleum hydrocarbons must be determined separately.

S. S. WOOLF.

Petroleum hydrocarbons as diluents in lacquers. O. R. BRUNKOW (Ind. Eng. Chem., 1930, 22, 177—178).—The factors which influence the use of naphthas as diluents for lacquers are discussed and a comparison is made, from the point of view of cost, between toluene and a mixture of toluene and naphtha as diluents. It is concluded that it is practicable to replace half the toluene by naphtha when the former costs 40—45 cents/gall.

H. INGLESON.

Composition of gum-resin. P. BOBROV (J. Appl. Chem., Russia, 1929, 2, 405—427).—Gum-resin produced in the Vyatka district contained (%): ether-insoluble residue 0.92—0.99, acidic substances 43.4—47.5, phenolic substances 6.00—8.17, neutral substances 38.1—37.8. Incomplete data concerning the composition of the fractions are recorded.

CHEMICAL ABSTRACTS.

Delicate reaction of colophony and resin acids. F. MICHEL (Chem.-Ztg., 1930, 54, 182—183).—Mineral oils, fats, or similar substances which are insoluble in alcohol are extracted with warm 50% alcohol and the alcoholic solution is evaporated to dryness. The residue is extracted with hot water, thoroughly washed and dried, and dissolved in 3 c.c. of chloroform. The resulting solution is treated with 5 c.c. of 65—67% sulphuric acid and, after vigorous shaking, acetic anhydride is added drop by drop to the upper chloroform layer. If colophony or a resin acid is present a beautiful violet colour is produced in this layer and, after again shaking, the dye formed passes into the sulphuric acid layer to which it imparts a red colour which has a characteristic spectrum with absorption bands at λ 580 and 511.

A. R. POWELL.

Tung oil. GÉRARD.—See XII. **Podophyllum resin.** WARREN.—See XX.

See also A., Mar., 289, Molybdenum-blue (DUCLAUX and TITÉICA).

PATENTS.

Manufacture of an artificial resin from ammonia and carbon monoxide. H. SPINDLER (F.P. 637,051, 29.10.26).—A mixture of carbon monoxide (2 mols.) and ammonia (2 mols.) is heated at 150—400° under 1000 atm. pressure in the presence of a copper, nickel, or iron catalyst, whereby the formaldehyde and carbamide formed condense with the production of a resin.

A. R. POWELL.

Manufacture of titanium-containing compounds. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH, Assrs. to NAT. METAL & CHEM. BANK, LTD. (U.S.P. 1,748,429, 25.2.30. Appl., 4.4.29. U.K., 1.10.27).—See B.P. 309,051; B., 1929, 529.

Drying apparatus (U.S.P. 1,741,981).—See I. **Dyes and intermediates** (B.P. 324,017). **Azo-dye pigments** (B.P. 302,251 and 323,937). **Colour lakes** (B.P. 324,120).—See IV. **Rubber-coated fabrics** (B.P. 299,321).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spray-drying of latex in Sumatra. H. DE LEEUW (Chem. Met. Eng., 1930, 37, 100—102).—Latex contains approx. 28% of rubber hydrocarbon, 0.3—0.7% of ash, 1—2% of protein, 1—2% of quebrachite, and 0.15—0.35% of reducing sugar. The rubber globules are 0.0005—0.003 mm. in diam. The Hopkinson method of spray-drying is considered to avoid a number of variables which are necessarily present in coagulation with acetic acid or alum. The latex is poured upon a disc rotating at high speed in the top of a drying chamber supplied with air at 177°. The flakes of dried rubber are removed at intervals and pressed. Sprayed rubber has a moisture content of 0.3%, the time required for curing is shorter, and the vulcanised product resists ageing better. The optimum tensile strength of the latter is also somewhat higher than that of the usual grades of rubber.

C. IRWIN.

Plastometer. KARRER and others.—See I.

PATENTS.

Production of reversible [rubber] latex compositions. DUNLOP RUBBER CO., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 324,375, 26.7.28).—Substantially reversible compositions of plastic consistency containing as little as 3% of water are obtained from aqueous dispersions of rubber or similar materials by careful evaporation in the presence of protective colloids, e.g., salts of a fatty acid with an alkali metal or an organic base, and one or more non-coagulant hygroscopic substances, e.g., polyhydric alcohols or their derivatives. These compositions can be rendered irreversible with respect to re-dispersion by water by mechanical treatment such as extrusion or kneading.

D. F. TWISS.

Extraction of rubber from rubber latex. AKTIEB. SEPARATOR (B.P. 303,895, 7.1.29. Swed., 13.1.28).—After the removal of concentrate from rubber latex by centrifuging, the residual impoverished latex, on account of impurities present, yields by coagulation an inferior rubber. This disadvantage is avoided by submitting the impoverished latex to purification in a suitable centrifuge before coagulation; this purification process can be assisted by preliminary addition of a substance, e.g., an electrolyte, capable of converting part of the colloidal or semi-colloidal impurities into a separable condition.

D. F. TWISS.

Manufacture of rubber. DEWEY & ALMY CHEM. CO., and D. M. STEVENS (B.P. 324,287, 19.3.29).—Rubber, while still in aqueous dispersion, is treated with an active-oxygen vulcanising agent, e.g., *m*-dinitrobenzene, *s*-trinitrobenzene, benzoyl peroxide, or potassium permanganate. The rubber obtained by evaporation or coagulation has the salient characteristics of vulcanised rubber except for the absence of sulphur. Treatment of latex with trinitrobenzene (3% on the

weight of rubber) for 60 min. at 95° yields rubber with tensile strength 1800—1900 lb./in.² and elongation 750%.

D. F. TWISS.

Manufacture of polymerisation products of diolefines [artificial rubber]. J. Y. JOHNSON and A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,004, 14.8.28 and 29.1.29).—In the polymerisation of diolefines in the presence of alkali metals the presence of hydrocarbons which do not take part in the reaction, e.g., butylene, cyclohexane, benzene, turpentine, or gasoline, is advantageous. These additional hydrocarbons do not act merely as diluents, but also influence the rate and course of the polymerisation; the greater the proportion of diluent the greater is the proportion of the polymerisation product soluble in benzene and the lower is the viscosity of the solutions obtained.

D. F. TWISS.

Vulcanising [of rubber]. F. W. FARR (B.P. 324,545, 13.2.29).—In order to promote uniform vulcanisation by sulphur chloride throughout a mass of rubber, the latter is perforated, reduced into small pieces, or mixed with fibrous material so as to facilitate penetration of the vulcanising fluid to all parts; the mass is then compressed while vulcanisation occurs. The method is suited to the repair of cuts in motor tyres.

D. F. TWISS.

Vulcanisation of synthetic rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,489, 27.12.28).—Polymerisation products of diolefines, e.g., of butadiene, are vulcanised by small proportions of selenium (e.g., 3% or less), with or without sulphur, in the presence of a suitable accelerator such as piperidylthiocarbamic acid or tetramethylthiuram disulphide. The products have great resistance to wear by friction.

D. F. TWISS.

Treatment of rubber and like material. GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 305,572, 8.11.28. U.S., 7.2.28).—An aromatic nitrosoamine, e.g., phenyl- β -naphthyl nitrosoamine or dinaphthyl nitrosoamine, is used as an antioxidant or age-resister for rubber.

D. F. TWISS.

Manufacture of thread and the like rubber. F. C. JONES (B.P. 324,186, 13.12.28).—A suitable rubber mixture is extruded through holes of suitable dimensions into a hot aqueous solution of an alkaline polysulphide which effects vulcanisation; the threads pass preferably first into an aqueous solution, e.g., of glycerin or soap, which prevents them from sticking together.

D. F. TWISS.

Decorative treatment of [india-rubber or] waterproof fabrics. J. MANDLEBERG & Co., LTD., and J. LLOYD (B.P. 324,211, 4.1.29).—The proofed material has a pattern produced thereon by embossing; a coloured rubber mixture is then applied by spreading, its consistency being such that it is taken up by certain edges or borders of the sunk or embossed pattern so as to produce a coloured surround with a shadow-like effect.

D. F. TWISS.

Vulcanisation accelerators (B.P. 323,512).—See III. **Paste for proofing textiles** (B.P. 295,660). **Waterproof fabrics** (B.P. 296,450).—See VI. **Activation of proteases** (B.P. 324,651).—See XVIII.

XV.—LEATHER; GLUE.

Determination of labile sulphur in gelatin and proteins. S. E. SHEPPARD and J. H. HUDSON (Ind. Eng. Chem. [Anal.], 1930, 2, 73—75).—It is pointed out that different proportions of labile sulphur may be obtained from the same sample by variations in the method employed. The details of a procedure worked out to determine the sulphur which forms silver sulphide in presence of ammoniacal silver chloride solution are described.

S. I. LEVY.

pH control. MAGNUS.—See I.

See also A., Mar., 382, **Tanning materials in leaves of *Carpinus betulus* at different times** (NIETHAMMER).

PATENTS.

Manufacture of [bleached] glues, gelatins, and the like. A. R. JAHN (B.P. 324,461, 7.3.29).—The glue etc. liquor concentrated to 15—45% of solids, is churned with sodium thiosulphate (2% or less on the weight of glue) for 10 min. at a temperature between the jelling point and 60°. The product contains minute bubbles which assist dissolution when the glue etc. is used. Ammonium carbonate may be added to the liquor to assist bubble formation, and borax as a preservative. These bubbles may be removed if necessary by slow churning at 90°. Liquid glues, e.g., starch products, may be similarly treated. E. B. HUGHES.

Manufacture of plastic compositions [from casein]. E. P. CARPENTER, Assr. to AMER. MACHINE & FOUNDRY Co. (U.S.P. 1,740,573, 24.12.29. Appl., 7.7.27).—A mixture of casein ground to 60-mesh with sufficient dilute hydrochloric acid to give an acidity of 10° is heated at about 60° to cause the casein to form a homogeneous plastic mass, which is washed in a 20% to saturated magnesium solution to remove impurities and cloudiness; the product is dried at about 40°, ground to 90-mesh, sprayed with water, and moulded and dried by compression and heat. L. A. COLES.

Emulsions (B.P. 323,720).—See III. **Dyeing of charmois leather** (B.P. 303,523).—See VI.

XVI.—AGRICULTURE.

Rheology of soil pastes. G. W. S. BLAIR (J. Rheology, 1930, 1, 127—138).—A plastometer is described for investigating the behaviour of pastes of soil or clay and water while flowing through narrow glass tubes. The data obtained from this instrument are useful in agricultural practice, since there is a close correlation between shearing strength of a sample and the pull required to draw a plough through the particular type of soil. The plastometer can also be used to study the effect of fertilisers on the physical structure of the soil. It has been found necessary to introduce a new constant into the flow equation, without which serious errors occur in estimating the viscosity of very thin pastes and the mobility of thicker ones. It is not possible to present a complete equation to relate the rate of flow to the shearing stress independent of capillary dimensions, but certain constants of the material can be obtained having independent dimensions which are closely related to the properties of the soil.

E. S. HEDGES.

Nature of hydrolytic acidity in soil. T. ARND [with W. SIEMENS and W. HOFFMANN] (*Z. Pflanz. Düng.*, 1930, 16A, 65—79).—Published work on the nature and determination of hydrolytic acidity in soils is critically discussed. Examination is recorded of the apparent acidity indicated by treatment of soil with various neutral salt solutions. True hydrolytic acidity is that remaining after exhaustive treatment of the soil with neutral salt solutions and the removal of active and exchange acidity. Recorded hydrolytic acidity values depend on the p_H value of the neutral salt solution used in their determination. The cause of hydrolytic acidity cannot be ascribed to a particular form of association of hydrogen ions with the acidoid particles of soil, whereby they are exchanged only with the cations of hydrolytically alkaline salts, but, in common with neutral salt decomposition, results from the exchange of hydrogen ions with cations of both neutral and hydrolytically alkaline salts. This exchange is a surface phenomenon, and therefore depends on the degree of dispersion of the soil particles. Sodium acetate by increasing dispersion records higher values for hydrolytic acidity than flocculating neutral salts.

A. G. POLLARD.

Relation between degree of dispersion and nature of the exchange bases of soil. I. VAJNA (Mezőg. Kutat., 1929, 2, 303—319; *Chem. Zentr.*, 1929, ii, 1963).—The degree of dispersion increases with the hydration of the cations. If the exchange bases are bivalent the degree of dispersion is increased. The International (Washington) Method A is not recommended for determining the original degree of dispersion of a soil.

A. A. ELDRIDGE.

The phosphate question [in soils]. III. Influence of phosphoric acid on the growth of plants. O. ARRHENIUS (*Z. Pflanz. Düng.*, 1930, 16A, 94—101; cf. B., 1929, 787).—Curves representing the relationship between crop yields and increasing phosphate content of the nutrient solutions in sand cultures were sigmoid in type. Corresponding relationships between the rate and extent of phosphorus intake and the growth period of plants are recorded and discussed from the point of view of fertiliser treatment.

A. G. POLLARD.

Phosphoric acid content and reaction conditions of the soil in various countries. C. DREYSPRING and C. KRÜGEL (*Superphosphate*, 1929, 2, 181—188, 201—210, 221—230).—The root-soluble phosphoric acid (Neubauer) of soils from Poland, Tunisia, Algeria, the Cameroons, Finland, and Czechoslovakia was determined.

CHEMICAL ABSTRACTS.

Reaction and phosphate content of soil. F. TERLIKOWSKI (*Rocz. Nauk. Roln. Leś.*, 1929, 22, 427—434; *Chem. Zentr.*, 1929, ii, 1963).—In general, soils having p_H below 6.0 are poor in active phosphorus compounds. In other groups no definite relation was observed, but most of the soils containing at least 15 mg. of P_2O_5 per kg. have an alkaline reaction.

A. A. ELDRIDGE.

Reaction and lime question [in soils]. R. THUN (*Z. Pflanz. Düng.*, 1930, 16A, 79—94).—Numerous soil analyses are recorded, showing chalk content, p_H value, and hydrolytic and exchange acidities. The relation-

ships between acidity, the availability of soil phosphate and potash, and plant growth are discussed. Close consideration of the lime condition of soils is of paramount importance in modern agricultural practice.

A. G. POLLARD.

Fixation of iodine in soils. J. BECK (*Z. Pflanz. Düng.*, 1930, 16A, 57—65).—The iodine content of a number of mineral soils examined varied directly with the proportion of clay. Examination of the mechanical soil fractions of soils showed the iodine to be largely concentrated in the clay fraction of mineral soils, and in the organic matter of humus soils. The iodine content of soils poor in clay and humus does not increase appreciably as a result of continuous manuring with seaweed.

A. G. POLLARD.

Balanced fertilisers and Liebig's law of the minimum. W. THOMAS (*Science*, 1929, 70, 382—384).—The work of Lagatu and Maume, showing that the absorption of nitrogen, phosphorus, and potassium by the vine does not follow Liebig's law, is discussed. These results show that whatever the effect the absence of an element from a fertiliser has on yield, it is due not to a depression, but to an increase in absorption of the other elements and the lack of nutritional balance which results.

L. S. THEOBALD.

[Fertilising] effect of surophosphate, nitrophos, and phosphorite compared with that of superphosphate and Thomas slag. M. GÓRSKI and J. KROTOWICZÓWNA (*Rocz. Nauk. Roln. Leś.*, 1929, 22, 139—152; *Chem. Zentr.*, 1929, ii, 1962—1963).—The effect of superphosphate (100) was greater than that of Thomas meal (90), and much greater than that of surophosphate or Polish phosphorite. The results depended largely on the nitrogenous fertiliser.

A. A. ELDRIDGE.

Citrate-soluble phosphoric acid in colloidal phosphate. J. B. MARTIN and E. C. SHOREY (*J. Assoc. Off. Agric. Chem.*, 1930, 13, 133—136).—Colloidal phosphate, if present, passes into the ammonium citrate extract, but may be flocculated by addition of sodium chloride.

K. V. THIMANN.

Use of crude [ammoniacal] gas-liquor as a fertiliser. K. H. RAUPP (*Gas- u. Wasserfach*, 1930, 73, 230—232).—Field trials indicate the value of ammoniacal liquor (1—4% NH_3) as a fertiliser. No injury to arable crops resulted from the use of diluted liquor. On meadow land applications of liquor at full strength caused a temporary discoloration of the grass, but considerably increased hay yields were obtained.

A. G. POLLARD.

Method for differentiating natural guano from its substitutes. L. PINTO (*Z. Pflanz. Düng.*, 1930, 16A, 111—120).—The presence of guanine in natural guano serves to distinguish it from synthetic preparations. Guanine in guano may be determined by means of metaphosphoric acid in alkaline extracts, but precipitation with silver nitrate from nitric acid extracts is much more sensitive.

A. G. POLLARD.

Effect of heavy application of phosphoric acid on the yield of oats. M. KÓRCZEWSKI and F. MAJEWSKI (*Rocz. Nauk. Roln. Leś.*, 1929, 22, 213—244; *Chem.*

Zentr., 1929, ii, 1963).—The inorganic phosphate in the straw and roots corresponded with the concentration in the soil; phosphoric acid fertilisation only slightly affected the percentage of organic phosphoric acid in the seed. The highest nitrogen content occurred with the lowest and highest applications of phosphorus. The nitrogen yield was fairly constant. The injurious effect of heavy applications of phosphate is not direct, but is due to disturbance of the chemical-physiological equilibrium. A. A. ELDRIDGE.

Resorption of phosphoric acid and potassium by cereal plants. J. PÁZLER (Z. Zuckerind. Czechoslov., 1930, 54, 297—302).—Comparison is made with barley, rye, oats, and wheat of the resorption of phosphates and potassium in Neubauer experiments. In spite of frequent cases of low germination and tendency to injury by fungal diseases, rye is considered the most suitable of the cereals for this purpose.

A. G. POLLARD.

Influence of light on the resorption of potash and phosphates in Neubauer experiments. A. NĚMEC and M. GRAČANIN (Z. Pflanz. Düng., 1930, 16A, 102—110).—Experiments with rye in Neubauer tests showed that the intake of phosphate was smaller, and of potash greater, in strong light than in weak. Comparison of growth in daylight with that in light of various colours showed that the resorption of potash was decreased by green light and increased by red and blue light. Coloured light in all cases reduced the intake of total ash constituents. Results with phosphate were less definite. A. G. POLLARD.

Influence of the potash concentration in the culture medium on production of carbohydrates in plants. G. JANSSEN and R. P. BARTHOLOMEW (J. Agric. Res., 1930, 40, 243—261).—From determinations of potassium, sugars, dextrin, starch, and hemicellulose in six crop plants grown in soil, sand, and water cultures, it is concluded that the relation between potassium and carbohydrate in the plants fluctuates greatly, and that a high percentage of sugars and starch is not necessarily associated with a high percentage of potassium. In water cultures it was found that between concentrations of 0 and 5 p.p.m. potassium was taken up in amounts in excess of the plant requirement of potassium for sugar and starch elaboration. The total weight of sugars and starch showed good correlation with the percentage of potassium in the plant. E. HOLMES.

Nitrogen content of, and distribution in, leguminous plants during growth. H. WOZAK (Forts. Landw., 1929, 4, 485—488; Chem. Zentr., 1929, ii, 2689).—Experimental results for various leguminous plants are tabulated. A. A. ELDRIDGE.

Effect of various modes of cultivation on nitrate formation in soil. A. V. SABASCHNIKOV (Forts. Landw., 1929, 4, 625—632; Chem. Zentr., 1929, ii, 2718).—A review of work carried out at Russian experiment stations. A. A. ELDRIDGE.

Lack of catalyst and bacterial content of soil in relation to the fertilising action of calcium cyanamide. W. KUBIENA (Forts. Landw., 1929, 4, 617—

622; Chem. Zentr., 1929, ii, 2718).—Most cultivated soils are poor in catalysts bringing about the decomposition of calcium cyanamide; sandy soils, poor in electrolytes and colloids, are unable to decompose the compound, and also contain relatively few bacteria. The fertiliser is unsuitable for soils lacking humus.

A. A. ELDRIDGE.

[Culture of] sugar beet. F. F. MATZKOV, A. Y. KOKIN, S. I. KOKINA, P. NENKO, A. A. VETUKHOVA, and B. A. PANSIN (Nauch. Zapiski Sakh. Prom., 1929, 8, 297—326).—Maximal root and chlorophyll production and maximal sugar content are obtained during 10-hr. days. In continuous light the energy of leaf assimilation decreases, whilst that of respiration increases. On decrease of the number of leaves to one third the root weight and sugar content remain normal. The sugar content of the beet is not increased by purely chemical stimulants. CHEMICAL ABSTRACTS.

Insecticidal properties of cryolite and barium fluosilicate. S. MARCOVITCH and W. W. STANLEY (Ind. Eng. Chem., 1930, 22, 121).—These materials are less soluble than is sodium fluosilicate and are safer on foliage. They have given good results against the Mexican bean beetle when used as a spray at the rate of 1 lb. per 50 gals. of water, or as a dust at 6 lb. per acre. Fish oil was used to increase the adhesiveness of the dust. C. IRWIN.

Soils at Cameron's Highlands. J. H. DENNETT (Malayan Agric. J., 1930, 18, 20—29).

Production of ammonium oxalate for use as fertiliser. W. DOMINIK (Rocz. Nauk. Roln. Leś., 1929, 22, 169—181; Chem. Zentr., 1929, ii, 1962).

Effect of various doses of phosphorite in presence of various nitrogenous fertilisers. B. VOVK (Rocz. Nauk. Roln. Leś., 1929, 22, 89—131; Chem. Zentr., 1929, ii, 1963).

pH control. MAGNUS. Consistency of plastic materials. SCHOFIELD and BLAIR.—See I. Determining total nitrogen. EMMERT.—See VII. Sugar palm. MILSUM and DENNETT.—See XVII.

See also A., Mar., 385, Copper as an element necessary to plants (QUARTAROLI). Phosphorus-containing constituents of rye embryos (KOEHLER).

PATENTS.

Sprayer [for insecticidal solutions etc.] LOWEL SPECIALTY Co. (B.P. 297,729, 16.7.28. U.S., 26.9.27).

Formaldehyde derivatives. (B.P. 299,064). Diazo-amino-compounds (B.P. 324,041).—See III.

XVII.—SUGARS; STARCHES; GUMS.

Ash and electrical conductivity of syrups and molasses derived from sugar cane. F. W. ZERBAN and L. SATTler (Ind. Eng. Chem. [Anal.], 1930, 2, 32—35).—The authors' formula (B., 1928, 422) gives results within about 0.5%, and generally within 0.25%, if the following modifications are made. On account of the high amount of ash, the concentration of the solution is reduced to 0.5%, and the sugar concentration may be corrected by adding 4.5% of pure cane sugar. If this is omitted the formula becomes $0.001640(9.13K + 1935$

— K_1) for raw-sugar products. For cane syrups $0.001640 \times (9.13K + 2047 - K_1)$ gives better results. For refinery products lower factors have been found, and there are grounds for associating this with the removal of weak bases by the char.

F. E. DAY.

Colloidal state of substances in products of manufacture of beet sugar. S. S. KUTZEV (Zhur. Sakhar. Prom., 1929, 3, 297—306).—Colloids are preferably coagulated by freezing or by neutralising with hydrochloric or sulphuric acid. Partial decolorisation may be due to adsorption of colouring matters by the coagulated colloids. Beet syrup, when vigorously shaken with benzene, carbon tetrachloride, or ether, forms an emulsion which on separation forms a gel on the surface of the unmixed solutions. The colloids extracted by emulsion, after settling for 1—2 months, retain 6—9% of molasses. Basic lead acetate does not precipitate all the optically active colloids. Titration with standard soap solution is inexact in the presence of colloids which can form a foam.

— CHEMICAL ABSTRACTS.

Technical and economical basis of the conversion of wood into sugars. M. NAPHTALI (Z. angew. Chem., 1930, 43, 215).—Dried waste wood gives a yield of 60—70% of its carbohydrate content in sugar and a yield of 30% of its weight in lignin when treated on the counter-current principle with concentrated hydrochloric acid. The operation is carried out in prodorite stoneware vessels and the resulting liquor is filtered through porous stones built into the vessels. The filtered syrup is sprayed through hot gas oil, which separates most of the hydrochloric acid and water, leaving a mixture of oil and syrup, which is heated to expel the oil for use again. The resulting syrup is dried in a spray dryer and the dilute acid recovered from the oil evaporator and dryer is treated with hydrogen chloride to bring it up to its original concentration for use again. Acetic acid is also recovered from this dilute acid liquor in amount equal to that obtained by the destructive distillation of the wood. The sugars obtained by the above process are suitable only for animal fodder or for the production of alcohol by fermentation.

A. R. POWELL.

Determination of the sugar content of carbonatation sludge. G. VAVRINECZ (Z. Zuckerind. Czechoslov., 1930, 54, 302—304).—Numerous methods for the preparation and clarification of sugar extracts from carbonatation sludge are compared. Results vary considerably. Extraction by shaking with water required 6—48 hrs. for completion, due, it is suggested, to the slow liberation of adsorbed sugar.

A. G. POLLARD.

Determination of lævulose with cupro-potassium carbonate solution. H. A. SCHUETTE and J. N. TERRILL (J. Assoc. Off. Agric. Chem., 1930, 13, 93—98).—The copper-lævulose equivalents for the Soldaini-Ost solutions are given, reduction for 2.5 hrs. at 48.9° being recommended. Filtration should be delayed for 48 hrs. to allow precipitation of the colloidal cuprous oxide formed.

K. V. THIMANN.

Determination of dextrose in the presence of lævulose. D. T. ENGLIS and W. J. BYER (Ind. Eng.

Chem. [Anal.], 1930, 2, 121—122).—The rates of reduction of iodine by dextrose and dextrose-lævulose (1:4) mixture have been examined. In a solution buffered by borate to p_H 10.6, dextrose gave the theoretical reduction at 60 min., after which further reduction proceeded very slowly. In carbonate solution at p_H 11.5, the theoretical reduction was reached in 10 min., and in 120 min. was exceeded by about 4%. In presence of the above proportion of lævulose the reduction in 60 min. at p_H 10.6 and in 10 min. at p_H 11.5 was increased by about 4%. After about 130 min. this excess became about 6% at p_H 10.6 and 30% at p_H 11.5. It is concluded that borate has no protective effect on the lævulose and that the different rates of reduction are determined by the alkalinities.

F. E. DAY.

Determination of starch syrup and starch sugar in presence of sucrose and invert-sugar. C. I. KRUISHEER (Z. Unters. Lebensm., 1929, 58, 261—281; cf. B., 1926, 963).—From determinations of reducing power and lævulose content (1) before inversion, (2) after weak inversion with 3% hydrochloric acid for 10 min. at 68—70°, and (3) after stronger inversion with 3% hydrochloric acid for 1 hr. on the boiling water-bath, by means of a number of simple equations it is possible to determine sucrose, invert-sugar, and starch syrup in presence of one another. The method is suitable for the examination of marmalades, syrups, and sugar products. The method of Schoorl (B., 1929, 952) for determination of reducing power and that of Kolthoff (B., 1923, 467 A) for determination of lævulose are suitable.

W. J. BOYD.

Measurement of the tenacity (Ergiebigkeit) of starches, especially of potato starch. PARLOW (Z. Spiritusind., 1930, 53, 14—15, 56).—Detailed methods are described for the determination of the tenacity of starches by the Lawaczek viscosimeter and by a simpler and cheaper form of viscosimeter devised by the author. The latter apparatus is an improved form of that of Stern, and has the advantage that the starch solution is prevented by an arrangement from falling in drops from the lower end of the capillary through which it flows. An unbroken flow of starch solution is obtained, and the surface tension effect which increases the time of flow is excluded. The viscosity readings are not directly proportional to the tenacity owing to the viscosity increasing more with the higher concentrations than corresponds to the higher content of starch in solution. The values obtained are not absolute, and both types of viscosimeter require to be standardised by the normal starch of Wolff, which can be obtained from the Research Institute of the Starch Industries. The tenacity of the normal starch is taken as 100%, and, according to the measurements made, a starch showing values below 120% is of inferior quality, 121—150% is normal, and above 150% is considered excellent.

C. RANKEN.

p_H control. MAGNUS.—See I. Philippine bagasse. VALENZUELA and WEST.—See V. Culture of sugar beet. MATZKOV and others.—See XVI. Detection of hydroxymethylfurfuraldehyde in honey. WEISS. Honey and honey cakes. KRUISHEER. Saponins and sugar wares. LODÉ.—See XIX.

See also A., Mar., 300, Velocity of inversion of sucrose (TAKETOMI).

PATENTS.

Glue products (B.P. 324,461).—See XV. Maize products (B.P. 324,702).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Hop-drying in the Saaz and Hallertau districts.

A. H. BURGESS (J. Inst. Brew., 1930, 36, 97—98).—The kilns, which are of the "Linhart" type, have three floors superimposed above a bottom floor, which is in the form of a drawer with a perforated iron bottom, and which is furnished with small wheels to permit its withdrawal from the kiln. The hops are loaded to a depth of 4 in. and dried for 1 hr. on the top floor. They are then dropped to the under floor for a similar interval, and the top floor is reloaded with fresh hops. At the end of the second hour the hops gravitate a step lower, and are eventually removed from the kiln in the bottom floor, the whole process, from loading to removal, having taken about 4 hrs. The temperature of the drying air is in the region of 38° between the top and second floors. The hot air may either pass directly from the heater through the hops, or may be led into flues and admitted beneath any, or all, of the four layers of hops. A "Linhart" kiln with a floor area of 162 sq. ft. will dry about 1000 imperial bushels of green hops in 24 hrs.

C. RANKEN.

Comparative brewing trials with new and commercial varieties of hops. W. T. SMITH and A. J. COSBIE (J. Inst. Brew., 1930, 36, 91—96; cf. B., 1928, 941).—The hybridisation of Continental and Oregon hops with English male hops has produced hops of very high preservative power, but has not eliminated entirely the flavours peculiar to the foreign female parent. Such hops are therefore of value as substitutes for foreign hops. The limitations of judging hops by hand examination cannot be overlooked as certain hops, the appearance of which has been unfavourable during the past three years, have consistently shown superior analytical and brewing results.

C. RANKEN.

Extra-compression of hops. T. K. WALKER, J. J. H. HASTINGS, and A. G. ALDOUS (J. Inst. Brew., 1930, 36, 99—104).—The hops were re-pressed 10 weeks after the original packing, and could be compressed to about half the normal volume without mechanical damage. Compressed hops deteriorated more slowly than normally pressed hops, and retained their preservative powers and appearance up to a period of 18 months as well as did normal hops kept in cold store. Extra-compression was also of advantage during cold storage, and the advantage was greater according to the extent of compression. The advisability of extra-compression at the time of packing requires further investigation.

C. RANKEN.

Effect of varying the steeping period of barley.

C. H. JOYCE (J. Inst. Brew., 1930, 36, 131).—The steeped barleys were malted, and the extract of the malt was determined and referred to the original barley. Steeping periods of 65 hrs. in the case of Scotch barley and of 60 hrs. in the case of Danubian barley were too long, and resulted in a decrease in the amount of extract

per quarter of barley, whereas a period of 50 hrs. with the Karachi, and more especially with the very dry Californian Mariout, barley was insufficient, and also resulted in a loss of extract. The malting loss with the Danubian barley was abnormally high. C. RANKEN.

Determination of relative diastatic powers of malt. F. C. SILBERNAGEL (Ind. Eng. Chem. [Anal.], 1930, 2, 31—32).—A "paste" of ordinary potato starch is stated to give more consistent results than soluble starch in Ling's method (B., 1900, 1029).

F. E. DAY.

Determination of liquefying power of malt diastase. S. JÓZSA and H. C. GORE (Ind. Eng. Chem. [Anal.], 1930, 2, 26—28).—The substrate is potato starch paste containing 84.22 g. of dry starch in 2000 g., buffered with acetate (p_H 4.6), the reaction of the mixture being p_H 5.0—5.2. The "paste" is broken down by stirring with a high-speed mixer. A 100-c.c. pipette, water-jacketed, is used for determining the relative viscosities, and should have outflow times of 55—57 sec. for water and 210—250 sec. for glycerin of d_{20}^{20} 1.2138. The time of outflow of the stirred starch paste after straining through a 100-mesh sieve should agree within 10—15 sec. at 21° with that of the glycerin. A curve is constructed by mixing portions of the paste with various proportions of the same paste which has been completely liquefied by the action of malt, and in which the enzyme has been then destroyed by heat, and plotting the outflow times of the mixtures as percentages of the original (diluted with water to equalise starch concentration) against mg. of starch liquefied. In the test 15 c.c. of 2% malt infusion are allowed to act on 150 c.c. of the substrate for 1 hr. at 21°, and the time of outflow is determined. The number of mg. of starch liquefied is read off from the graph, and this, divided by mg. of malt in the extract, gives liquefying power.

F. E. DAY.

Acid substances entering into the composition of wines. L. SEMICHON and M. FLANZY (Ann. Falsif., 1930, 23, 5—19).—The total acidity of a wine is regarded as the quantity of carbon dioxide that 1 vol. of wine sets free from an excess of calcium carbonate, and may be expressed as c.c. of normal liquid, or by the amount of sulphuric or tartaric acid liberating the same quantity of carbon dioxide. The value is found by covering 250 mg. of very finely-ground calcium carbonate with boiling water, adding 20 c.c. of the wine drop by drop and shaking, and finally boiling the mixture for 5 min. The amount of carbon dioxide not attacked is determined, and the quantity liberated by the wine calculated from the value. The method may be regarded as indicating exactly the saturation of all the carboxyl groups of the organic compounds present. Free organic acids are determined as above, salts of organic acids by finding the alkalinity of the ash, and an ester determination would give the esters of the organic acids; the sum of the three values gives the content of total organic acids. The sum of free and combined organic acids is regarded as a new value, giving a picture of the synthesis of organic acids taking place during maturation, physiologically comparable with the synthesis of sugar.

D. G. HEWER.

A special method of wine making. F. OBRÉ (Ann. Falsif., 1929, 22, 595—597).—A method is described by which three grades of wine (grey or rose, red, and press wine) can be obtained from one batch of grapes. The advantages and disadvantages of the method are stated and analyses of three wines made by the method are given. A. SHORE.

Currant wine. J. PINNOW (Z. Unters. Lebensm., 1929, 58, 331—342; cf. B., 1923, 902 A).—Increase in temperature promotes fermentation of the dextrose in the must to a greater degree than that of the *lævulose*. Variations in the proportions of alcohol, glycerin, and succinic acid are ascribed neither to variations in sugar content of the must nor to changes in the temperature of fermentation, but are due to alterations in the character of the yeast on berries of different location and vintage. The ratio of neutral esters of low volatility to total esters of low volatility also varies with the vintage. Ethyl hydrogen succinate and ethyl hydrogen citrate were identified in currant wine. In a 6-year-old wine the former amounted to 16% of the total succinic acid, whereas the neutral ester amounted only to 1% of the total succinic acid. Evidence of the presence of an acid which forms a lactone, probably γ -hydroxyvaleric acid, was obtained. W. J. BOYD.

Detection of added mineral acid in wines by means of the potentiometer. V. MORANI (Annali Chim. Appl., 1930, 20, 30—48).—The p_H of normal wines varies from 2.65 to 3.78 or, with wine of low acidity and high extract, even more. Values below 2.65 are to be attributed to the presence of strong mineral acid. Gradual addition of sulphuric or hydrochloric acid to wine causes rapid lowering of the p_H , this being at first approximately proportional to the amount of acid added. The presence or absence of added mineral acid in the wine may be shown definitely by means of the buffering powers. To five 50-c.c. portions of the filtered wine are added 2, 4, 6, 8, and 10 c.c., respectively, of 0.1N-potassium hydroxide, the p_H of each portion and of the untreated wine being then determined. The buffering powers of the successive intervals of the neutralisation curve are obtained by dividing 4 (the number of c.c. of *N*-alkali added per litre) by the successive increments of p_H . Adjacent numbers in the series of values thus obtained should vary (usually increase) at most by one or two units. Any marked difference, such as four units, renders certain the presence of strong mineral acid. T. H. POPE.

Lactic acid in the determination of the volatile acids of wines. (MME.) LAMBERTI (Ann. Falsif., 1929, 22, 592—595).—The various methods of determining the volatile acids of wines are compared, particularly with regard to the behaviour of lactic acid, which may be present in wine in very variable amounts. The proportion of the lactic acid present which was included in the volatile acids was found to vary according to the method of determination adopted, and the conclusion is drawn that this may be one cause of the fact that the quantity of volatile acids found in any particular wine differs according to the method of procedure used for their determination. A. SHORE.

Determination of alcohol by pycnometer. A. F. FUERST (Ind. Eng. Chem. [Anal.], 1930, 2, 30—31).—The alcohol content of aqueous distillates containing not more than 5% of alcohol may be determined within an accuracy of 0.01%. Details of procedure are given. S. I. LEVY.

p_H control. MAGNUS.—See I. Sugar from wood. NAPHTALI.—See XVII.

See also A., Mar., 372, Malt pectinase (WILLAMAN). Analysis of proteins (HUNTER and DAUPHINEE). 374, Selective fermentation of dextrose and *lævulose* by brewer's yeast (IVEKOVICH). Decreasing rate of fermentation (RAHN). Phosphoric esters of alcoholic fermentation (ROBISON and MORGAN). 384, New enzyme in jack bean (KITAGAWA and TOMIYAMA). Enzymic substance in koji made from rice (ITO).

PATENT.

Activation of proteases. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 324,651, 19.10.28).—The proteases are activated by the addition of thioacids or the salts thereof. Thus papain with added sodium thiosulphate coagulates rubber latex more rapidly than does untreated papain, and completely degums raw silk by degrading the sericin covering, which is not attacked by the inactivated enzyme. C. RANKEN.

XIX.—FOODS.

Nutritive value of cereal breakfast foods. I. Composition and calorific value. J. R. MURLIN, W. R. LINE, H. A. PIPER, and H. B. PIERCE. II. Digestibility *in vitro*. Methods. J. S. CARMAN, H. G. SMITH, G. C. HAVENS, and J. R. MURLIN (J. Nutrition, 1929, 2, 83—90, 91—110).—Analytical values for six prepared foods are recorded. Coefficients of utilisation were: protein 84% (rolled oats), 94% (wheat endosperm); fat 90%; carbohydrates 96—97%. Cooking (boiling) increases the digestibility of the starch less than that of the protein. The simultaneous action of a proteolytic enzyme (trypsin) and a diastase produced much more rapid digestion of starch and protein than either enzyme alone. CHEMICAL ABSTRACTS.

Disinfection of grain and flour with chloropicrin and other volatile substances. P. Y. SALDAU, V. I. POSPELOV, A. D. PETROV, and V. B. ISACHENKO (Trans. State Inst. Appl. Chem., Moscow, 1928, No. 10, 90—107).—Experiments with *Calandra granaria*, *Pachymerus chinensis*, and *Aleurobius farinæ* showed that chloropicrin is 8—10 times as toxic as carbon disulphide, although the latter in a container on the top of the grain or flour may penetrate faster than the former. Chloropicrin depresses the germination of wheat, stimulates that of oats, and has no effect on that of lentils. CHEMICAL ABSTRACTS.

Addition of chemicals to flour in order to increase the volume on baking. H. KALNING (Chem.-Ztg., 1930, 54, 161).—Amongst additions to flour which without generation of carbon dioxide appear to improve the "rising" qualities are calcium chloride, calcium phosphate, potassium bromate, ammonium persulphate, chlorine, benzoyl peroxide, sodium perborate, and manganese sulphate. Most of these substances are

used in very minute quantities, and any excess reverses the effect. They are of little benefit to good-quality flour, but improve that of poorer qualities. Ammonium persulphate can be detected by benzidine solution (blue coloration); benzoyl peroxide by alcoholic guaiacol solution (green coloration). No chlorine is present in flour treated with chlorine.

C. IRWIN.

Baking powders and "mineral raising agents."

L. WEIL (Ann. Falsif., 1929, 22, 601—604).—Chemical yeasts ("levures chimiques") is the official and commercial name under which baking powders are sold in France. The paper is a short review of the acid constituents available for the manufacture of baking powders, and of the chemical changes which take place in them during baking. The suggestion is made that a baking powder containing neither alum nor cream of tartar (the price of which is prohibitive) could be made from sodium pyrophosphate, sodium bicarbonate, and calcium tartrate, and that calcium pyrophosphate could be used as a diluent and would prevent deterioration of the powder in view of the fact that it is able to fix a considerable quantity (35% of its weight) of water.

A. SHORE.

Oxidation-reduction in milk. I. Oxidation-reduction potentials and the mechanism of reduction. II. Choice of an indicator for the reduction test. Reduction of Janus-green-B in milk. H. R. THORNTON and E. G. HASTINGS (J. Bact., 1929, 18, 293—318, 319—332).—The positive limits of the difference in potential between the normal hydrogen electrode and milk lay between +0.2 and 0.3 volt, and the negative limits approximated to -0.2 volt. The "poising" effect of methylene-blue in milk is negligible. Methylene-blue reduces over a satisfactory range of potential and is the most suitable indicator. It is suggested that the "poising" action of Janus-green-B is due to molecular rearrangement or the formation of an intermediate reducing (probably hydrazo-) compound.

CHEMICAL ABSTRACTS.

Comparison of the modified Babcock and the Mojonnier methods for determination of butter fat in ice-cream. E. S. CHASE and F. G. KING (J. Dairy Sci., 1929, 12, 473—480).—In 86% of 754 tests the results agreed within 0.2%. The modified Babcock method is described.

CHEMICAL ABSTRACTS.

Separation of maize starch introduced fraudulently into egg powder. COMTE (Ann. Falsif., 1929, 22, 600).—The high sp. gr. of starches is used as the basis of a method of separating them from other powdered materials. A weighed quantity of the powder is shaken vigorously in a centrifuge tube with a mixture of carbon tetrachloride and ether (3:1 pts. by vol.). The mixture is centrifuged at a slow rate, whereby the starch settles to the bottom, and after washing and air-drying is weighed. The method, with suitable modifications, can be applied to the separation of other powders.

A. SHORE.

Chicory "agglomérés" and their adulteration. L. GOBERT (Ann. Falsif., 1929, 22, 580—591).—"Agglomérés" is the name given in France to powdered chicory which is transformed into artificial granules by the addition of various adulterants and of some agglutinant

such as sodium silicate. Such granules are added to those obtained by roasting and crushing the chicory root. Approximate macroscopic and microscopic methods of analyses of admixtures so produced are given. Comparative analyses of the ash content also accompany the paper. The necessity for revising the analytical standards to which these products must conform in France is emphasised.

A. SHORE.

Source of diastase in honey. G. H. VANSSELL and S. B. FREEBORN (J. Econ. Entomol., 1929, 22, 922—926).

—The amount of pollen in honey is correlated with the diastatic activity, but artificial pollen cultures do not show as high diastatic values as those of normal honeys. The digestive tract of the bee, when free from pollen, contains no diastase. The pollen count should be used as an adjunct to the official diastase test.

CHEMICAL ABSTRACTS.

Diastase in honey. G. H. VANSSELL (J. Econ. Entomol., 1929, 22, 926—929).—For the determination of diastatic activity the proteins in 1 g. of honey are precipitated with alcohol, the solution then being diluted and acidified with acetic acid to p_H 4.4. After addition of soluble-starch solution (0.5 c.c.) the mixture is kept at 36° and the time required for conversion of the starch into sugar is determined. The diastase contents of raw honeys differ widely; excessive heating destroys the diastase.

CHEMICAL ABSTRACTS.

Detection and determination of [hydr]oxy-methylfurfuraldehyde in honey and artificial honey. F. WEISS (Z. Unters. Lebensm., 1929, 58, 320—331).—The honey (10 g.) is rubbed in a mortar thrice with 5 c.c. of ethyl acetate each time, and the solvent is poured off and evaporated while gently warmed in a glass basin, using a hand bellows to remove last traces. The residue is stirred twice with 0.5 c.c. of water and the solution filtered. To the filtrate 5 c.c. of the reagent (a saturated, freshly filtered solution of *p*-nitrophenylhydrazine in 30% acetic acid) are added, the basin and filter being mixed with portions of this. After several hours the crystals are filtered into a tared Gooch crucible dried at 105°, washed with water, and dried for 2—3 hrs. at 105°. The weight of precipitate $\times 0.435$ gives the weight of hydroxymethylfurfuraldehyde. Qualitatively the reagent can be used to detect less than 0.001 g. of the aldehyde. With larger quantities of honey the various quantities are proportionately increased. Ether is unsuitable as solvent for the extraction. In artificial honey 0.03—0.23% of hydroxymethylfurfuraldehyde was found, in pure honey none, and in honey heated to 90—100° small quantities were detected.

W. J. BOYD.

Application of the formol titration to honey. H. A. SCHUETTE and V. TEMPLIN (J. Assoc. Off. Agric. Chem., 1930, 13, 136—142).—The formol titration value of honey is slightly lowered by the addition of syrups, but the amounts involved are too small, and the error of the method too great, for the procedure to be of analytical value. The titration cannot be correlated with the nitrogen content of the honey.

K. V. THIMANN.

Examination of honey and honey cakes. C. I. KRUISHEER (Z. Unters. Lebensm., 1929, 58, 282—300).

—The author's method for determining various sugars in presence of one another (B., 1930, 388) is useful in part for the examination of honey and honey cakes. For genuine honey the minimum value of the ratio {lævulose before inversion (F_1) \times 100}/{dextrose before inversion (G_1)} is 90 and that of the ratio ($F_1 \times 100$)/extract is 43 (cf. Mees, B., 1929, 109). In baking, the amount of dextrin increases, and if lævulose (before inversion) is absent, *i.e.*, if starch syrup, starch sugar, or artificial honey from glucose has been substituted for genuine honey, then considerable lævulose is formed during baking probably owing to the action of alkali on the dextrose. In cakes prepared with honey, invert-sugar, or sucrose, little change in the sugars occurs apart from dextrin formation. Owing to these changes it is not possible to determine the quantity of starch sugar added by the author's method. By the aid of Fiehe's reaction it is possible to determine the nature of the saccharine material added. For this purpose it is necessary to determine the dry weight of the crumb, the content of water-soluble extract, the reducing power (R_1) and lævulose content (F_1) of the extract before inversion, and the reducing power (R_2) after weak inversion with 3% hydrochloric acid for 10 min. at 68–70°. If the sucrose content, $0.95 (R_2 - R_1)$, is above 5%, sucrose has been added. When the ratio $100F_1/G_1$ is less than 90 a starch product has been added. A positive Fiehe reaction indicates that invert-sugar has been used if $100F_1/G_1 > 90$, otherwise it may be due to hydroxymethylfurfuraldehyde present in the starch sugar or syrup added. If the p_H of the extract is above 5, as it usually is, hydroxymethylfurfuraldehyde has not been produced during the baking process. Under these conditions it tends to be destroyed.

W. J. BOYD.

Iron, copper, and manganese content of some common vegetable foods. R. E. REMINGTON and H. E. SHIVER (J. Assoc. Off. Agric. Chem., 1930, 13, 129–132).—The iron, manganese, and copper content of a number of vegetables has been determined. The metals are present in considerably larger quantities in the leaves than in the roots and fruits. The figures for iron and manganese are more variable than those for copper, the amount of which does not exceed 20 pts. per million of dry weight.

K. V. THIMANN.

Vitamins. S. MATHEWS and C. NEWTON (Ga. Agric. Exp. Sta. Ann. Rep., 1928, 33–34).—Cooking in an open kettle reduced the vitamin-C content of turnip greens by 85%, but did not affect the vitamin-A content. Collards contain 33% and Hearts of Gold cantaloupe 10% as much vitamin-C as do turnip greens.

CHEMICAL ABSTRACTS.

Is prohibition of addition of foam-forming saponin-containing substances to sugar wares (*e.g.*, "halwa") justified? A. LODE (Z. Unters. Lebensm., 1929, 58, 311–319; cf. Heiduschka and Zywnew, *ibid.*, 1923, 45, 61).—Samples of "halwa" (sesamé meal) were found to contain 0.02–0.09% of saponin in terms of Saponinum purum albiissimum (Merck). Excessive addition is unlikely owing to the unpleasant taste and effect on the mucous membranes of the mouth and throat, which are pronounced in con-

centrations of saponin otherwise harmless. It is suggested that up to 0.2% should be permitted. Methods are given for the detection and determination of saponin in "halwa" based on the hæmolytic properties of the former. Thus in the determination a series of solutions of increasing dilution is prepared from a neutralised 10–20% solution of the product in 0.85% salt solution, and a drop of erythrocyte suspension is added to 5 c.c. of each. The most dilute members of the series showing complete dissolution of the corpuscles after 2 hrs. and after 18 hrs., respectively, are noted. A similar series of tests is carried out, using solutions of a standard saponin preparation, and by comparison the strength of the "halwa" solution in terms of the standard saponin preparation is found.

W. J. BOYD.

Determination of traces of iodine. III. Iodine in milk, butter, oil, and urine. J. F. McCLENDON, R. E. REMINGTON, H. VON KOLNITZ, and R. RUFÉ (J. Amer. Chem. Soc., 1930, 52, 541–549).—Further adaptations of the previous method (cf. A., 1928, 607; 1929, 413) are described. Foodstuffs are burnt with oxygen in a silica or pyrex glass-tube furnace, the material being introduced continuously by means of an atomiser in the case of oils and by a screw-feed device with solids, the latter being packed into sausage casing to facilitate their introduction. Heat may be applied either externally by gas burners or internally by electrically heated platinum spirals or by "oxy-gas" torches. Urine is freed from urea with urease and burned in the same way in a pyrex tube. The iodine is absorbed in sodium sulphite solution and freed from excess nitrite with hydrazoic acid. The iodide is oxidised with a known amount of nitrite and the iodine extracted with carbon tetrachloride. Using known volumes of both carbon tetrachloride and aqueous layer, the iodine can be determined colorimetrically.

J. W. SMITH.

Determination of citric acid in fruits and fruit products. B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1930, 13, 99–103).—The citric acid is precipitated as the lead salt, decomposed with hydrogen sulphide, and the acid converted into pentabromoacetone with potassium bromide and permanganate, and weighed as such. The method gives results 5% low with pure citric acid, but is accurate in the presence of malic and tartaric acids and is suitable for use with pure acid mixtures or fruit products.

K. V. THIMANN.

Determination of tartaric acid in fruits and fruit products. B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1930, 13, 103–112).—Two methods are described. The fruit product is freed from pectin by precipitation with alcohol, and the tartaric acid precipitated as the lead salt and decomposed with hydrogen sulphide. In the first method the acid is determined gravimetrically as potassium hydrogen tartrate. In the second diammonium citrate, calcium acetate, and ammonium *l*-tartrate are added and the precipitated calcium racemate is determined by oxidation with permanganate at 80°. Both methods are unaffected by presence of citric or malic acid, but give somewhat low results with less than 50 mg. of tartaric acid. The former is the more rapid.

K. V. THIMANN.

Application of Burgess-Parr sulphur photometer to rapid determination of sulphur in foods and biological material. E. W. TOEFFER and P. W. BOUTWELL (Ind. Eng. Chem. [Anal.], 1930, 2, 118—121).—Methods of converting sulphur into a soluble form are discussed. The use of the Burgess-Parr bomb is very suitable where a total weight of sample not exceeding 1 g. may be employed; for larger samples, oxidation by perchloric and nitric acids is recommended. The photometric determination based on the production of colloidal barium sulphate is found to be simple, rapid, and reliable.

S. I. LEVY.

Apparatus for destructive oxidation of organic material in the determination of metals in foods. J. W. BARNES (Ind. Eng. Chem. [Anal.], 1930, 2, 107—108).—Modifications of the usual apparatus for the oxidation by nitric and sulphuric acids are described.

S. I. LEVY.

pH control. MAGNUS.—See I. Utilisation of vegetable residues. LEONE and OCCHIPINTI.—See II. Culinary rock-salt. ZELLNER.—See VII. Cadmium plate. HOPFELT.—See X. Rice-husk fat. DE'CONNO and FINELLI.—See XII. Sugar from wood. NAPHTALI. Determination of syrups. KRUISHEER.—See XVII.

See also A., Mar., 317, Illipene, and higher alcohols in illipé butter (TSUJIMOTO). 368, Comparison of nutritive properties of soya-bean "milk" and cows' milk (Tso).

PATENTS.

Bleaching of flour. N.V. CHEM. IND. VAN HASSELT (CHEM. IND. VAN HASSELT); and N.V. MEELFABR. DER NEDERL. BAKKERIJ (MEELFABR. DER NEDERL. BAKKERIJ) (B.P. 308,253, 22.2.29. Holl., 20.3.28).—Bleaching of flour by oxides of nitrogen is accomplished by addition of solid substances (e.g., nitrosylsulphuric acid or its double compounds with stannic or ferric chloride, admixed, if desired, with silica gel, bleaching earth, etc.) giving nitrous acid on hydrolysis, or by addition of solutions of nitrogen peroxide and chlorine in a suitable solvent, such as glacial acetic acid or benzene.

E. B. HUGHES.

Manufacture of products derived from maize. W. ACTON (B.P. 324,702, 1.11.28).—The usual British method of preparation is modified so that the liquors used for steeping, washing, etc. are worked back into the process without any loss except that due to the drying of the starch, the inhibition of development of putrefactive organisms being attained by keeping the liquor at p_H 4 and at 40—45°. In the neutralisation stage p_H 6.5 is maintained to give the best separation of starch. The liquor from this stage is treated with the acid liquor from the steeping tanks for precipitation of the gluten which is made with the bran into a gluten feed.

E. B. HUGHES.

Extraction and purification of pectin. J. RENNOTTE (F.P. 578,463, 22.5.23).—The pectin in fruit extracts is recovered by addition of alcohol, redissolved in water, and reprecipitated with alcohol; tannin, dye-stuffs, etc. remain in the alcoholic solutions.

A. R. POWELL.

[Apparatus for] freezing fish, meat, and other

foodstuffs. II. G. FAIRWEATHER. From MARITIME FISH CORP., LTD. (B.P. 325,685, 12.2.29).

Treatment of liquids with ultra-violet rays (B.P. 324,503).—See XI.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Sterilisation in pharmaceutical practice. E. DEUSSEN (Arch. Pharm., 1930, 268, 190—203).—A discussion, from the view point of the pharmacist, of published results on the preparation of pure water, the sterilisation of vessels, and the effects of different methods of sterilisation on solutions of alkaloids and other thermolabile drugs. Experiments are described to show that, at temperatures above 70°, distilled water dissolves appreciable quantities of alkali from ampoule glass.

H. E. F. NOTTON.

Comparative study of cinchophen preparations in their action on the elimination of uric acid and the mode of action of cinchophen. T. KAKU (Acta med. Keijo, 1928, 11, 47—66).—The uric acid-eliminating properties of the derivatives methoxylated in the phenyl group are generally stronger than those of non-methoxylated derivatives. The effect of 6- (but not 7-) methyl is to weaken the action; 6:8-dimethyl also weakens it, but 8-methyl increases it.

CHEMICAL ABSTRACTS.

Assay of resin of *Podophyllum*. L. E. WARREN (J. Assoc. Off. Agric. Chem., 1930, 13, 117—128).—The physiological activity of *Podophyllum* resin is not restricted to the portions soluble in alcohol or chloroform, and estimations of these fractions are therefore of no value as an assay. A method for the determination of total resins by extraction with an alcohol-chloroform mixture is described.

K. V. THIMANN.

Colorimetric determination of acriflavine and neutral acriflavine. E. L. HOWES (Ind. Eng. Chem. [Anal.], 1930, 2, 114).—3:6-Diamino-N-methylacridinium chloride ("neutral" acriflavine) and its monohydrochloride may be determined by means of the colour reaction with nitric acid containing nitrogen oxides. Suitable methods of manipulation are described.

S. I. LEVY.

Laboratory testing of germicides and chemotherapeutic agents. O. SCHÖBL (Philippine J. Sci., 1929, 40, 283—289. Cf. B., 1924, 342, 765, 995; 1925, 650).—The author discusses the work of Reddish and Drake (J. Amer. Med. Assoc., 1928, 91, 712) and of Simmons (*ibid.*, 704) on the relative values of mercurochrome-220 and tincture of iodine for skin disinfection. Employing one type of bacteria only (*Staphylococcus*), Reddish and Drake found the bactericidal action of mercurochrome equal to that of iodine, but Simmons, on the basis of tests with six bacterial types and inoculation experiments with anthrax, found iodine to be superior.

J. H. LANE.

Determination of chloral in chloral syrup. M. G. MEILLÈRE (J. Pharm. Chim., 1930, [viii], 11, 145—147).—Quantitative reduction of the chloral occurs when the syrup (5—10 g.), diluted to 75 c.c. with sulphuric acid (18%), is treated overnight with coarse zinc filings (3 g.). The chlorine is determined as silver chloride. Titration of chloral syrup with alkali is

best effected with 0.2N-barium hydroxide (cf. François, B., 1928, 243). H. E. F. NOTTON.

Rapid method for the determination of arsenic in medicinal preparations. C. G. ESCOLAR (Anal. Fis. Quim., 1930, 28, 167—170).—The arsenic is converted into sodium arsenate and determined colorimetrically by addition of sodium molybdate and stannous chloride solutions. The coloration reaches its maximum in about 10 min., and is compared with that produced by a known quantity of arsenic. H. F. GILLBE.

Ceylon citronella oil. A. W. R. JOACHIM (Trop. Agric., Ceylon, 1929, 73, 136—141).—No relationship between the total geraniol content and values of d , n , or $[\alpha]$ was observed. Ceylon and Java citronella oils have, respectively, d 0.898—0.920, 0.882—0.900; $[\alpha]$ -7° to -18° , 0 to -5° ; n 1.4785—1.4900, 1.4640—1.4725; total geraniol 55—64, 80—92%.

CHEMICAL ABSTRACTS.

Utilisation of vegetable residues. LEONE and OCCHIPINTI.—See II. **Commercial ether.** LEFFMANN and PINES.—See III. **Castor oil.** HEIDUSCHKA and KIRSTEN.—See XII.

See also A., Mar., 350, **Local anæsthetics in the pyrrole series** (BLICKE and BLAKE). 351, **Substituted phenylethylbarbituric acids** (BOUSQUET and ADAMS). **Local anæsthetics derived from piperazine** (FOURNEAU and BARRELET). 352, **Components of Chinese drug "Ma Huang"** (KANAO). 353, **Harmala alkaloids** (SPÄTH and LEDERER). **Microchemical reactions for hydrastine and berberine** (WAGENAAR). 370, **Pharmacology of *Allium sativum*** (LEHMANN). **Testing of brucine derivatives** (THOMS). **Determination of salvarsan** (HIRAMATSU). **Toxicity of arsenobenzene compounds** (KIELBASINSKI). 379, **Preparation of two active and specific thymus substances** (NITSCHKE). **Antimony trichloride reaction for vitamin-A** (KARRER and others; NORRIS and CHURCH). 380, **Concentration of vitamin-B₂** (NARAYANAN and DRUMMOND).

PATENTS.

Manufacture of phenyl-oxalyl acetic acid alkyl esters [alkyl phenyloxalacetates]. R. H. PRITCHETT, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,741,877, 31.12.29. Appl., 19.1.29).—Ethyl oxalate reacts with ethyl phenylacetate in dry alcohol in presence of sodium ethoxide to give ethyl phenyloxalacetate.

C. HOLLINS.

Manufacture of bornyl and isobornyl esters. L. SCHMIDT (B.P. 306,385 and 306,387, 10. and 13.8.28. Fr., 18.2.28).—(A) The conversion of pinene into bornyl and isobornyl acetates by means of acetic acid and boron trioxide or boroacetic anhydride proceeds better in two stages, the acetic acid being added after a preliminary esterification with boroacetic anhydride or boric acid and acetic anhydride. (B) Alternatively, the esterification is completed by removing the boron compound and heating the filtrate with another catalyst, e.g., oxalic acid, at a lower temperature. C. HOLLINS.

Quinine compound and its manufacture. W. H. ENGELS, Assr. to MERCK & Co. (U.S.P. 1,741,761, 31.12.29. Appl., 8.11.23).—Quinine sulphate, $Q.H_2SO_4$, is stirred in aqueous solution with a solution of

sodium salicylosalicylate to give a sparingly soluble salt, $Q.2C_{14}H_{10}O_5$, m.p. 86—88°, useful in the treatment of influenza, colds, neuralgia, etc. C. HOLLINS.

Manufacture of compounds of *k*-strophanthidin. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 323,973, 19.3.29).—Either of the two *k*-strophanthidins is converted by acylation (e.g., with acetic anhydride and pyridine) into uniformly more active compounds. The *acetyl* (m.p. 248—249°), *propionyl* (m.p. 239—240°), *α-bromopropionyl* (m.p. 192—193°), *isovaleryl* (m.p. 183—184°), *α-bromoisovaleryl* (m.p. 203—204°), *n-valeryl* (m.p. 212°), *n-hexoyl* (m.p. 205—207°), *isoheptyl* (m.p. 180°), *isoheptoyl* (m.p. 195—197°), *phenylacetyl* (m.p. 207—208°), and *cinnamoyl* (m.p. 254—255°) derivatives are described. C. HOLLINS.

Manufacture of [formaldehyde-bisulphite] derivatives of aminoaryl antimony compounds [stibinic acids]. I. G. FARBENIND. A.-G. (B.P. 298,234, 3.10.28. Ger., 6.10.27).—*p*-Aminobenzenestibinic acid, or partly hydrolysed *p*-acetamidobenzenestibinic acid, is treated with formaldehyde and sodium hydrogen sulphite. C. HOLLINS.

Production and separation of active substances from glands. SOC. CHEM. IND. IN BASLE (B.P. 299,060, 19.10.28. Switz., 20.10.27).—Generative glands are hydrolysed with acid or alkali or are fermented with trypsin, and the lipoid-soluble hormone is extracted with ether or benzene, the water-soluble hormone being then recovered by known means. C. HOLLINS.

Manufacture of [therapeutic] durable soap creams containing highly-concentrated salt solutions. S. WEISSBEIN, Assr. to BÄDER- u. VERKEHRS-A.-G. (U.S.P. 1,748,533, 25.2.30. Appl., 23.6.26. Ger., 16.7.25).—See B.P. 309,012; B., 1929, 443.

Vermicides (B.P. 323,187).—See III. **Healing medium etc.** (U.S.P. 1,739,586). **Medicament** (U.S.P. 1,740,543).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., Mar., 305, **Photochemistry of silver halides** (LESZYNSKI).

PATENTS.

Photographic paper having a transferable gelatin-silver film and processes in connexion herewith. N. POZNIAKOV (B.P. 324,330, 11.9.28).—Before the application of the sensitive emulsion the paper surface is treated with a water-soluble undercoating which is without action on the emulsion, e.g., gum arabic, sugared gelatin, soluble starch. For the preparation of negatives for true-to-scale work, the material, prior to development, is pressed face downwards to make firm contact with a rigid support, e.g., wetted celluloid, then treated with water to remove the base, and finally developed and fixed. [Stat. ref.] J. W. GLASSETT.

Manufacture of photographic surfaces. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 324,381, 26.9.28).—Organic compounds which contain the grouping $>C:C<$ with each carbon atom directly linked to a CO group, and which are converted under the action of light into a stereoisomeric form, are admixed with a

colloid and a catalyst such as a halogen or a compound yielding a halogen in the presence of light, *e.g.*, mercuric bromide, tetrabromoethane, ω -bromoacetophenone, or a perbromide, and coated upon a suitably coloured base or the latter is saturated with the sensitising solution. If neither isomeride is coloured, use is made of their specific reactions to form a coloured substance by esterification, salt-formation, etc., and the image is fixed by removal of the other isomeride or of the catalyst. Examples are given showing the application of (a) *trans*-dibenzoyl ethylene, (b) maleic acid sensitised with bromine vapour, the image being formed from the sparingly soluble silver or mercury salts of fumaric acid, and (c) phenylcitraconic acid catalysed by ω -bromoacetophenone, from which the silver, mercury, or thallium salt of phenylmesaconic acid is subsequently formed and changed to black by a reducing agent or sodium sulphide. J. W. GLASSETT.

Photographic developers. I. G. FARBENIND. A.-G. (B.P. 295,939, 20.8.28. Ger., 18.8.27. Addn. to B.P. 292,950; B., 1929, 925).—*N*- β -Hydroxyethyl-*o*-aminophenol has developing properties similar to those of the *p*-compound. C. HOLLINS.

Treatment of photo-printing materials for the establishing of images thereon. J. HALDEN & Co., LTD., and J. HOLDEN (B.P. 324,048, 9.10.28).—Prints prepared from diazo papers are treated, at any time subsequent to the development with the coupling agent, with an acid solution of an inorganic reducing agent (*e.g.*, sodium metabisulphite, or stannous chloride with a small quantity of tartaric acid), whereby the background is cleared and stabilised and the contrast increased. This treatment is particularly applicable to those cases where the background is strongly coloured by the use of a coupling agent giving a strongly coloured alkaline solution. [Stat. ref.] J. W. GLASSETT.

Colour photography. T. T. BAKER (B.P. 324,043, 8.8.28).—In printing from master colour films the dilution of colours, which results from the overlap in the spectral bands transmitted by the dyes constituting the reseau of the master film, is avoided in the colour positive by either of the following methods: (a) the master film is treated after the first development with a reducing agent, *e.g.*, potassium ferricyanide, which attacks the low densities first, and is then intensified and reversed; or (b) the dyes constituting the elements of the positive reseau are concentrated so that their spectral transmission bands are narrower than those of the theoretical primaries. J. W. GLASSETT.

Production of photographic pictures in natural colours. KALLE & Co. A.-G. (B.P. 299,010, 15.10.28. Ger., 19.10.27).—Two or more differently coloured pictures made by using stable diazo compound as described in B.P. 234,818 (B., 1925, 693) are superposed, or are produced successively on the same base. Suitable diazo compounds are, *e.g.*, those from 6-ethylamino-*m*-toluidine with phenol for yellow, with H-acid for blue, and 1:2:4-aminonaphtholsulphonic acid with resorcinol for red; or 6-ethylamino-*m*-toluidine with H-acid for blue, and 1:2:4-aminonaphtholsulphonic acid with phenylmethylpyrazolone for orange. C. HOLLINS.

[Tri-packs for] photography in natural colours. T. T. BAKER, and COLOUR PHOTOGRAPHS (BRITISH AND FOREIGN), LTD. (B.P. 324,394, 25.10.28).

XXII.—EXPLOSIVES; MATCHES.

Deterioration of mercury fulminate when stored under water. W. T. INGRAHAM (Army Ordnance, 1929, 10, 201—202).—Mercury fulminate (99.56%), stored for 5 years in canvas bags under water in a dark, cool place, gave values as follows: distilled water, 98.26%; sea-water, 82.05%; alkaline water high in carbonates, 95.82%; alkaline water high in sulphates, 97.73%. CHEMICAL ABSTRACTS.

Combustion velocity of smokeless powders. N. YAMAGA (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 60—62; cf. B., 1925, 301).—In assuming the combustion of smokeless powder in a gun or closed vessel to be a reaction between activated molecules, it is considered that there is activation between the molecules at the surface of the powder and those of the products of decomposition. The combustion velocity (*v*) may be calculated theoretically from the equation: $v = (KP/T)e^{-(E/RT)} - (E'/RT)$, *P* being the pressure, *E* the critical energy of the products of the combustion, and *E'* the critical energy of the powder. Experiments with powders of various compositions confirm the correctness of the equation, and give the following values for the constants: $E/R = 9613$, $E'/R = 138$, and $\log K = 1.243$. W. J. WRIGHT.

Dynamites: their repulsive strength, rate of detonation, and poisonous gases evolved. N. A. TOLCH and G. ST. J. PERROTT (U.S. Bur. Mines Rep. Invest., 1929, No. 2975, 16 pp.).—The order of propulsive strength differs according to the basis (weight or bulk). The rate of detonation increases with the rated strength. The order of diminishing production of carbon monoxide is: straight, ammonia, and ammonia-gelatine and gelatine-dynamite. For each type the carbon monoxide produced increases with the rated strength.

CHEMICAL ABSTRACTS.

Plastic study of nitroglycerin-nitrocotton gels. L. C. PRITCHETT (J. Rheology, 1930, 1, 149—156).—A modified form of the Bingham plastometer has made it possible to measure the consistency of mixtures of dynamite nitrocellulose and nitroglycerin. The mobility values are readily determined and the curves give large and quite definite yield values. The "A" nitroglycerin gels have a higher mobility than similar concentrations of "D" nitroglycerin. The yield values for "A" nitroglycerin for a given concentration, time of gelatinisation, and temperature are greater than for the "D" compound, suggesting that nitrocellulose is dispersed more easily in "A" than in "D" nitroglycerin. The yield value increases and the mobility decreases during the process of dispersion of nitrocellulose in nitroglycerin. With low concentrations of nitrocellulose, dispersion approaches completion in 2 hrs. in "D" nitroglycerin at 59°, and only after several months at 38°; at room temperature the process is indefinitely slow. It is concluded that "A" nitroglycerin is the better solvent for nitrocellulose. E. S. HEDGES.

Ignition velocity of gaseous explosive mixtures. TERRES and WIELAND.—See II.

XXIII.—SANITATION; WATER PURIFICATION.

Measuring the toxicity of insect fumigants. A. L. STRAND (Ind. Eng. Chem. [Anal.], 1930, 2, 4—8).—The methods used for establishing relative toxicities are reviewed, and an examination of the commoner fumigants is made on the basis of the concentration required to kill 50% of the test insects after an exposure time of 5 hrs.

S. I. LEVY.

Pollution studies of the upper Mississippi River. F. L. WOODWARD (Ind. Eng. Chem., 1930, 22, 189—192).—The condition of the river from Minneapolis and St. Paul (Minnesota) to La Crosse (Wisconsin), a distance of 160 miles, has been investigated, samples being taken at 24 points. It is shown that the pollution caused by the two cities is perceptible for a distance of 100 miles in winter and 70 miles in summer, the larger winter effect being due to restricted re-aeration caused by ice. For a distance of nearly 50 miles the river is heavily polluted, fish life being practically absent and nuisances frequent. From this point the river gradually recovers and is able to purify itself from any pollution before the next city is reached.

C. JEPSON.

Utilisation of saline lake waters. B. PANTELEIMONOV (Chem.-Ztg., 1930, 54, 153—155).—A brief account of the work on which the exploitation of the water of the Rapa Lake in the Crimea is being developed, and of work undertaken with a view to extraction of magnesium compounds and halogens from other lake waters (cf. B., 1929, 282, 812, 1043).

S. I. LEVY.

Apparatus for the determination of oxygen in water containing suspended slime. KÜCHLER (Chem.-Ztg., 1930, 54, 184).—The apparatus for sampling the water consists of three glass vessels, *A*, *B*, and *C*, clamped to a long iron handle for lowering them into the water. *A* and *B* are of 300 c.c. and *C* of 1 litre capacity. *A* has a conical bottom terminating in a stopcock, *a*, and all are closed with a rubber stopper bored with two holes. *A* is joined to *B* by a glass tube starting just below the stopper in *A* and terminating in the middle of *B*, and *B* is joined to *C* by a glass tube starting 1 cm. below the stopper of *B* and terminating just flush with the stopper of *C*. In the stopper of *A* is a second glass tube closed with a rubber tube and clip, *b*, and in the stopper of *C* is a second glass tube about 1 m. long also closed with a rubber tube and clip *c*. The apparatus is placed below the surface of the water to be analysed and *b* and *c* are opened, suction being applied to *c* until *A*, *B*, and *C* are filled in succession and the water rises in the metre tube. Thus *A* and *B* are thoroughly rinsed by the water which collects in *C*. The clips are closed and the slime allowed to settle; *a* and *c* are then opened and by blowing through *c* the slime in *A* is displaced by clear water from *B* and thus 300 c.c. of clear water are obtained for analysis without loss of dissolved gas or contamination.

A. R. POWELL.

Extinction of ethylene oxide flames. JONES and KENNEDY.—See II. **Ferric alumina.** MOBERG and PARTIDGE.—See VII. **Germicides.** SCHÖBL.—See XX.

See also A., Mar., 302, **Determination of dissolved oxygen in water** (ALSTERBERG).

PATENTS.

Apparatus for the biological purification of sewage. O. MOHR (U.P. 157,153, 20.5.26).—The sewage

is preliminarily treated mechanically and is led through a duct, into which rain-water pipes feed, into a circularly-bent, apertured drain pipe which encircles a ground-water shaft and which is embedded in a biological filter comprising granular layers arranged at varying inclinations with respect to the shaft, the succeeding layers being progressively graded in granule size. The shaft is provided with slits and the water passes through the filter and slits into the shaft. A de-aerating pipe, provided with a throttle, connects the filter with the top of the shaft. The pipes and filter are swept through by air.

S. K. TWEEDY.

Disinfectant, antiseptic, and healing medium. O. GERNGROSS and K. RÜLKE, Assrs. to KALI-CHEMIE A.-G. (U.S.P. 1,739,586, 17.12.29. Appl., 9.3.25. Ger., 27.6.24).—A thiocyanogen derivative, preferably ammonium, magnesium, or an alkali or alkaline-earth thiocyanate, is added as activating agent to organic disinfecting agents, e.g., alcohols, phenols, aldehydes, heterocyclic compounds, terpenes; albuminous substances, saponin, starch, dextrin, etc. may also be present.

L. A. COLES.

Disinfectant, antiseptic, and medicament. O. GERNGROSS and K. RÜLKE, Assrs. to KALI-CHEMIE A.-G. (U.S.P. 1,740,543, 24.12.29. Appl., 26.6.26. Ger., 28.1.24. Cf. U.S.P. 1,606,359; B., 1927, 93).—The product comprises a silver salt having disinfecting properties (potassium argentocyanide) and an excess of an alkali thiocyanate.

L. A. COLES.

Filling for gas-masks. DEUTS. GOLD- U. SILBERSCHNEIDE-ANSTALT A.-G. VORM. ROESSLER (G.P. 460,765, 12.4.25).—The filling comprises an adsorptive medium for odourless poisonous gases combined with one for tear gas, e.g., zinc carbonate or caustic alkali for hydrogen cyanide and adsorptive carbon for trichloronitromethane. The carbon is of relatively low activity and in a thick layer, so that it does not take up much of the cyanide; in consequence its value as an adsorptive for the tear gas is not impaired.

A. R. POWELL.

Continuous softening of water [by the use of zeolites]. C. H. NORDELL, Assr. to PERMUTIT Co. (U.S.P. 1,740,199, 17.12.29. Appl., 15.5.22).—The apparatus consists of a softening tank in which the raw water rises through a descending shower of zeolites, the hardest water thus coming into contact with the more nearly exhausted material. The softened water is withdrawn from the upper part of an inverted bell placed near the top of the tank and the exhausted material together with water to render it sufficiently fluid to run is forced by water pressure into a separate vessel where it is regenerated by the addition of a predetermined volume of saturated brine. The regenerated zeolites are delivered into a washing tank placed above the softening tank and connected thereto by an automatic valve. Water from the softening tank is admitted intermittently and on rising through the descending softening agent removes the unused brine etc. The washed zeolites are admitted at the same time into the softening tank and the cycle is recommenced. The apparatus described is so arranged that the operation proceeds automatically (cf. U.S.P. 1,608,661; B., 1927, 206).

C. JEPSON.

Formaldehyde derivatives (B.P. 299,964).—See III.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 9 and 16, 1930.*

L—GENERAL; PLANT; MACHINERY.

Furnaces for high temperature. R. OTTO (Chem. Fabr., 1930, 41—43).—The various furnaces by means of which temperatures of 2000° to 2600° may be attained in an oxidising atmosphere are described. Zirconia is one of the most important materials for the construction. Mixtures of coal gas and air under pressure and of oxygen and petroleum vapour are employed as sources of heat.

S. I. LEVY.

Component heat exchanges in the evaporative condenser. A. W. SCOTT (J. Roy. Tech. Coll., Glasgow, 1930, No. 2, 268—298).—Consideration of the heat exchange in a condenser involves the heat exchange between steam and cooling surface and the metal to water interchange. At the water-air interchange the factors involve the convective heating by the water of the air and its associated vapour, together with the process of conversion of a portion of the cooling water into vapour at the air temperature. Experimental work with steam conditions ranging from a reduced pressure of 24 in. to atmospheric pressure and a velocity of that of air up to 25 ft./sec. showed marked regularity of evaporation, and for given air conditions the rate of evaporation was determined only by water temperature and was not influenced by the depth of the cooling-water film. A working theory and design curves were evolved.

C. A. KING.

History of vacuum drying. E. PASSBURG (Chem. Fabr., 1930, 93—95).—The first vacuum dryer was an experimental one erected in 1881 in a sugar refinery in Petersburg for drying sugar containing 3—4% of moisture. After the success of this apparatus the author applied the principle to substances containing a greater amount of moisture and designed a shelf-dryer for logwood extract. The first vacuum paddle-dryer was used for brewers' grains. Later important applications were for the treatment of starch and white lead. Vacuum drying is also of importance in the manufacture of cables, armatures, and coils for dynamos and transformers, as it allows the last traces of moisture to be removed with safety from the insulating material. Experiments on the safe drying of mercury fulminate are described and the more important application to smokeless powder with vapour recovery is referred to. In the chemical industry the rotating-drum type of dryer is the one chiefly employed. Drying may be combined with calcination without breaking the vacuum, as is done with potash liquors.

C. IRWIN.

Statistical description of the size properties of non-uniform particulate substances. T. HATCH and S. P. CHOATE (J. Franklin Inst., 1929, 207, 369—387).—Adopting statistical methods, particle size is defined in

terms of the shape of the size-frequency curves of substances in the form of non-uniform particles. The size properties are related closely to the statistical parameters M_g and σ_g of the log. probability curves followed by the size-frequency distribution of such materials. This definition has advantages over the use of the average diameters Δ and D , in that the statistical parameters define one and only one size-frequency distribution curve and yield information concerning size-distribution and relative frequency of certain sizes. The average diameters Δ and D are functions of M_g and σ_g , which therefore yield all the information given by Δ and D , besides simplifying the calculation. The theoretical conclusions are justified by experiment.

J. W. SMITH.

Rapid determination of relative viscosities of opaque liquids. M. M. LOESERMAN (Ind. Eng. Chem. [Anal.], 1930, 2, 25).—A modification of the bubble method is described, for use when the liquids under examination are very opaque.

H. F. HARWOOD.

Construction of platinum wire chain for the Foulk chain hydrometer. W. W. KOCH and G. F. SMITH (Ind. Eng. Chem. [Anal.], 1930, 2, 41—42).—Platinum wire chains constructed from 40-gauge wire are well adapted for use in the Foulk precision chain hydrometer (J. Opt. Soc. Amer., 1923, 7, 327), and the method of preparing such a chain is described. If a thermostat be used to keep the temperature of the system constant to 0.01° it is possible with this device to detect in such a liquid as perchloric acid a difference in density corresponding to a change in acidity of only 0.001%, whereas 0.05% is the smallest change which can be detected by even the most refined analytical methods.

H. F. HARWOOD.

Decarburisation [and new type of Töpler pump]. HACKSPILL and SCHWARZ.—See X.

PATENTS.

Furnace and its operation. W. DE FRIES (U.S.P. 1,740,288, 17.12.29. Appl., 7.4.21).—In a regenerative, open-hearth, or similar type of furnace it is considered that the convective effect of a rapidly moving nearly theoretical mixture is, on balance, more efficient than the radiant effect of a slowly moving mixture with excess air, as hitherto used. In this furnace one combustible, preferably the air, is heated above the ignition temperature (by the regenerator), and is then mixed with the other cold combustible in proportion to give a mixture which is explosive but below the ignition temperature; this is then admitted to the furnace through a Venturi-shaped passage in which the velocity is high enough to prevent striking-back. The result is a

* The remainder of this set of Abstracts will appear in next week's issue.

short, high-temperature, turbulent and neutral flame, and in consequence the hearth may be made shorter and wider.

B. M. VENABLES.

Furnace [for solid fuel]. G. A. KONOUD (U.S.P. 1,739,907, 17.12.29. Appl., 22.8.25).—Within the furnace and parallel with the front wall is a hollow arch forming with the latter a hopper for the fuel. The fuel is drawn out below the arch by a mechanical grate. Air is supplied to the hollow of the arch, emerges over the fuel in the hopper, then passes over the arch, under another arch pendant from the roof rather further within the furnace, and into the combustion space over the burning fuel.

B. M. VENABLES.

Non-reversible [regenerative] furnace. L. MAMBOURG, ASSR. to LIBBEY-OWENS GLASS CO. (U.S.P. 1,739,973, 17.12.29. Appl., 10.10.25).—In a glass "tank" all the ports along both sides are continuously used for admission of combustibles, the exhaust ports are situated at the feed end, and the reversing through the regenerators is effected outside the furnace proper.

B. M. VENABLES.

Rotary kilns. J. S. FASTING (B.P. 324,521, 24.1.29).—In a kiln for the treatment of slurry, chains are suspended loosely in such a manner as to assist the progress of the material through the kiln; two arrangements of the chains are figured.

B. M. VENABLES.

Heat exchangers. J. HOWDEN & CO., LTD., and J. H. HUME (B.P. 325,697, 25.2.29).—A heat exchanger, *e.g.*, an air heater for boilers, of the type where the outer fluid makes a number (say 4) of passes across tubes carrying the inner fluid, is provided with diverting dampers so that the flow may be either full-series (4 passes) or series-parallel (2 passes).

B. M. VENABLES.

Heat exchanger. H. THOMAS, ASSR. to SUN OIL CO. (U.S.P. 1,742,833, 7.1.30. Appl., 13.1.26).—In an exchanger of the concentric tube type, a method of connecting the ends of the tubes in series is described. To allow for expansion the connectors, at one end, for the pairs of inner tubes lie entirely within those for the outer tubes. Removable blank flanges and plugs are provided to enable any tube to be cleaned without removal.

B. M. VENABLES.

Heat-exchange apparatus. A.-G. DER MASCHINEN-FABR. ESCHER WYSS & CO. (B.P. 309,570, 12.2.29. Switz., 13.4.28).—A safety device, *e.g.*, to protect feed-water heaters, works on the principle that if leakage arises so that more water goes in than comes out, the apparatus will be isolated and the water by-passed round it. The control is effected by a pair of "static-pressure plates" each connected to a double-acting cylinder the pistons of which are coupled together.

B. M. VENABLES.

Utilisation of waste heat. H. B. SMITH, ASSR. to BABCOCK & WILCOX CO. (U.S.P. 1,741,663, 31.12.29. Appl., 6.4.27).—Heat is transferred from hot, solid, non-combustible material to air, then from the air partly to a boiler. The partly cooled air without further contact with the hot solid is mixed with finely-divided fuel and burned for the production of additional hot solid material. *E.g.*, cement clinker from a rotary kiln may drop down a vertical passage the upper part of which is sealed

against air-flow by transverse, staggered, sloping shelves over which the clinker slides; after the vertical passage the clinker may pass through a rotary cooler. Air passes countercurrent through the rotary cooler and lower part of the passage, then through a boiler, a suction-and-pressure fan, to the burner of the kiln.

B. M. VENABLES.

Drying apparatus. B. DEWEY and T. L. TALIAFERRO (U.S.P. 1,741,981, 31.12.29. Appl., 22.1.26).—The apparatus is for supporting plate-like objects out of contact with each other as they pass through an enameling oven.

B. M. VENABLES.

Cooling tower. J. M. SEYMOUR (U.S.P. 1,739,867, 17.12.29. Appl., 12.8.27).—The water to be cooled is permitted to drop only a short distance at a time from an uppermost feed tank on to and through several storeys of perforated sheets, to a catch pit at the bottom. Horizontal air currents are maintained between the sheets by means of fans. The cooling zone is preferably annular with peripheral inlets and inner vertical outlets for the air. Owing to the low fall, splashing is slight and any spray is caught on the outer walls because of the rotation of the air.

B. M. VENABLES.

Removal of liquid with or without solid particles therein from the surface of rotary drums transporting it, particularly in cooling apparatus. APPAREILS ET EVAPORATEURS KESTNER, SOC. ANON. (B.P. 324,559, 7.3.29. Fr., 21.1.29).—The liquid is removed from the main drum by a roller making water-tight contact with it, and from the roller by a plate making rubbing contact. The materials of the roller and plate are chosen so that the coefficient of friction is low, *e.g.*, they may be rubber-covered and of glass, respectively.

B. M. VENABLES.

Anti-freeze compound. V. W. HAAG, H. E. BIGLER, and I. L. HAAG (U.S.P. 1,742,349, 7.1.30. Appl., 27.11.25).—The mixture comprises water, glycerin, and glucose in substantially equal proportions. The glucose may be the commercial quality containing dextrose, and is preferably mixed with part of the water when heated to 100°, then the rest of the water is added and finally the glycerin.

B. M. VENABLES.

Refrigerant. MARLO REFRIGERATING CO., LTD., and F. C. MALIN (B.P. 325,925, 27.12.28).—A refrigerant which functions at relatively low pressures and evaporates gradually comprises 80–94 pts. of methyl chloride, 6–3 pts. of sulphur dioxide, and 4–1 pts. of bromine or 10–2 pts. of carbon tetrachloride, the higher proportions of methyl chloride being used when the lower temperatures are desired.

L. A. COLES.

Crushing, grinding, and refining machine. S. BRAMLEY-MOORE, ASSR. to BRAMLEY MACHINERY CORP. (U.S.P. 1,740,194, 17.12.29. Appl., 18.6.29).—A corrugated surface is formed on the interior of the fixed casing by welding rods thereto, parallel to the axis. The moving grinding surface is also formed of rods which are supported on springs and forced outwards by a tapering inner shaft within the boss of the rotor, acting through push rods and sliding pieces.

B. M. VENABLES.

Comminuting mill. R. C. NEWHOUSE, ASSR. to

ALLIS-CHALMERS MANUF. CO. (U.S.P. 1,739,855, 17.12.29. Appl., 20.12.28).—The general construction of this multi-stage tube mill is similar to that described in U.S.P. 1,710,666 (B., 1929, 497), but the oversize from the surrounding screen is returned to the mill through a passage formed in a tyre. B. M. VENABLES.

Beater mill. [Disintegrator.] W. J. KUNTZ (U.S.P. 1,742,511, 7.1.30. Appl., 18.4.28).—A disintegrator is provided with a discharge chamber to which the communication from the mill is guarded by a series of hinged vanes, the adjustment of which determines the size of the product delivered. B. M. VENABLES.

Means for disintegrating solid materials. H. A. PERTWEE (B.P. 325,583, 22.11.28).—The machine is suitable for coke and materials of similar toughness and comprises a pair of rollers with very pronounced teeth. The teeth are formed on rings which are individually detachable from the rollers because they are divided into at least two segments and are held in place by longitudinal bolts. B. M. VENABLES.

Grinding or pulverisation of gummy, pasty, or viscous material. H. F. KLEINFELDT, ASSR. to ABBE ENG. CO. (U.S.P. 1,739,761, 17.12.29. Appl., 1.10.26).—The material is chilled and thereby rendered brittle by mixing with solid carbon dioxide, either in a separate mixer or in the pulveriser itself. B. M. VENABLES.

Washing of granular material. A. J. MASON (U.S.P. 1,741,063, 24.12.29. Appl., 21.1.29).—Material such as phosphate of lime which has already been washed with a large quantity of water, but still has indurated clay etc. adhering to the particles of sand, is mixed with water not exceeding 40%, preferably about 20%, of its own weight, treated to an abrasion process in some form of puddler, and then dried. B. M. VENABLES.

Mixing machine. C. NIELSEN (U.S.P. 1,737,609, 3.12.29. Appl., 28.5.28).—Solid material is fed into a mixing tank by means of an agitator operated through a clutch by the mixing shaft, which, in turn, is operated by an electric motor. The liquid is admitted to the tank by a float valve which also operates the clutch and switch for the motor in correct sequence. B. M. VENABLES.

Digestors. SILLER & RODENKIRCHEN GES.M.B.H., and W. SILLER (B.P. 325,760, 17.5.29).—The digester is rotated on trunnions to effect stirring. Through twin passages in one trunnion a heating fluid is passed to and from a jacket, and within the other hollow trunnion is a stuffing-box surrounding a fixed shaft which spreads out within the digester and forms a stirrer. This shaft is also provided with two internal passages and the stirrer with ports to admit acid or other liquid through its lower part and to discharge pulp through the upper part. The apparatus is suitable for the extraction of glucose from wood by means of acid. B. M. VENABLES.

Evaporators. O. ENGISCH (B.P. 308,622, 23.3.29. Ger., 24.3.28).—A heating element for sea-water evaporators is constructed in the form of a deeply corrugated cylinder which is rotated against scrapers

(or against which scrapers are rotated); the latter are flexibly supported to allow for expansion of the heater. B. M. VENABLES.

Evaporation of liquids [sulphite lye] and apparatus therefor. S. H. LEDIN and J. O. NAUCLER (B.P. 300,590, 15.11.28. Ger., 15.11.27).—Flue gases are forced through a perforated plate above which about 1–2 cm. of liquid are maintained, intense bubbling being thereby produced with consequent large contact surface. Various forms of foam traps are described. [Stat. ref.] B. M. VENABLES.

Apparatus for bringing liquids and gases into intimate contact. T. PIGGOTT & CO., LTD., J. W. MARTIN, and F. E. BANFORD (B.P. 325,724, 21.3.29).—The apparatus comprises one fixed drum and several rotating concentric drums. The annular spaces are filled with brush-like material. The gas flows in a longitudinal zig-zag manner through the upper parts of the drums, the general direction being from the circumference inwards, while liquid is supplied to the innermost compartment, forms a bath in each compartment in the lower part of the apparatus, and flows countercurrent to the gas. B. M. VENABLES.

Treatment of gases with solids and liquids. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & CO., LTD. (B.P. 324,747, 21.11.28).—This invention permits the renewal of a tower filling without stopping the flow of gases. The tower is divided into stories by means of flap valves upon which the filling (inert or reactive) rests, and the gases travel in a zig-zag course across the tower, passing from storey to storey through external passages. The lowest section of the filling is removed from the bottom, the remaining sections are dropped one stage, and a fresh charge is added at the top by operating the flap valves in succession upwards. B. M. VENABLES.

Separating apparatus, particularly for the separation of liquids from gases. G. E. T. EYSTON (B.P. 324,656, 30.10.28).—The gases pass through a fixed duct in the walls of which are apertures through which the liquid is deflected by moving baffles; e.g., the apertures may be annular and the baffles in the form of fans which are rotated by the gas current and of which the tips extend into an outer chamber where the atmosphere is substantially quiet, though a slight current of gas to carry off the separated matter may be permitted through it. B. M. VENABLES.

Treatment of gas or a mixture of gas and vapour in the presence of catalytic agents and reactivation of such agents. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 324,481, 18.12.28. Fr., 24.10.28).—The catalyst is contained in an annular chamber between two cylindrical shells and the gases to be treated pass through in a longitudinal direction, but the gases for regeneration are delivered into the mass at a number of different places. B. M. VENABLES.

Porous moulded body for use in diffusion, filtration, etc. and its manufacture. J. MÜLLER, ASSR. to SCHUMACHER'SCHE FABR. G.M.B.H. (U.S.P. 1,742,411, 7.1.30. Appl., 8.7.26. Austr., 10.7.25).—A mixture of hot, comminuted, refractory material,

such as sand, glass, or asbestos, with a bituminous binder (e.g., asphalt, hard pitch) is moulded under pressure at a temperature approaching the m.p. of the binder. After cooling, the moulded article is reheated at 300–400°, and then slowly cooled. S. K. TWEEDY.

Treatment of boiler water. C. T. MCGILL (U.S.P. 1,742,714, 7.1.30. Appl., 29.8.27).—Part of the raw water is passed through a softener of the base-exchange or lime-soda type and part is left unsoftened, the fractions are metered and mixed in a heater, and the mixture is fed to all the boilers. Each boiler is in circuit with a "deconcentrator" where mud is settled out. The water from each boiler is tested at intervals, and according to its condition either additional softening reagent or a coagulant is added to the individual boiler.

B. M. VENABLES.

Manufacture of a packing composition for stuffing-boxes etc. F. SCHAUB (B.P. 325,566, 14.11.28).—A lubricant such as oil or fat, binders, and graphite are mixed and kneaded with sufficient granular solid particles of graphite or carbon to form a support, so that the mixture is plastic, elastic, adherent, and sufficiently stiff to remain in the gland when used alone.

B. M. VENABLES.

[Radiation] pyrometer. F. C. WHALEN, and RADIOVISOR PARENT, LTD. (B.P. 324,500, 4.1.29).—The energy in any part of the spectrum from a "black" body being a function of the total energy radiated, the light radiated from the furnace or other article is measured. The light is reflected on to a selenium bridge or other sensitive device and a filter for the longer waves is interposed to prevent an undue rise of temperature.

B. M. VENABLES.

Colorimeters. A. HILGER, LTD., F. TWYMAN, and J. PERRY (B.P. 324,351, 18.8.28).—The light from a sample is analysed by three screens and the primary colours are allowed to fall separately on to a measuring and recording device such as a photographic film or thermo-electric cell with galvanometer, which gives a definite figure to the intensity of primary colour. Alternatively, a standard colour may be compared with the sample by means of a flicker device, thermo-electric cell, and telephone, the intensity of any primary colour being adjusted by calibrated neutral wedges until sample and standard give the same effect. Calculations are given for the preparation of the filters.

B. M. VENABLES.

Colorimeter. R. A. HOUSTOUN (B.P. 324,495, 1.1.29).—A mirror-lined tunnel is closed at the observer's end by a sheet of ground glass and at the other end by two filters sliding over each other, outside which is a source of light such as an opal gas-filled electric lamp. The filters are coloured magenta and yellow, peacock-blue and magenta, respectively in halves, the combination forming red, green, and blue rectangles of which the width and therefore the amount of each primary colour can be varied by sliding the filters to different measured positions. The tunnel combines the colours, and the uniform tint on the ground glass is observed simultaneously with the object by means of a partly silvered mirror.

B. M. VENABLES.

Calcining furnace and method of operating the

same. E. KLÖHN, Assr. to ZAHN & Co., G.M.B.H. (U.S.P. 1,751,161, 18.3.30. Appl., 3.8.27. Ger., 30.12.26).—See B.P. 290,837; B., 1928, 551.

Mixing apparatus. A. B. and C. R. SMITH (U.S.P. 1,751,221, 18.3.30. Appl., 24.9.27. U.K., 5.10.26).—See B.P. 278,177; B., 1927, 896.

Grinding mill. R. M. HARDGROVE, Assr. to FULLER-LEHIGH Co. (U.S.P. 1,751,918, 25.3.30. Appl., 14.5.28).—See B.P. 311,688; B., 1929, 1000.

Grinding mill. T. BRAGARD, Assr. to MACAOWALZENMÜHLEN-GES.M.B.H. (U.S.P. 1,750,088, 11.3.30. Appl., 3.7.28. Ger., 9.6.27).—See B.P. 291,803; B., 1928, 878.

[Centrifugal] separator drum. A. SCHMÜCKING, Assr. to F. KRUPP A.-G. (U.S.P. 1,750,364, 11.3.30. Appl., 19.10.28. Ger., 28.10.27).—See B.P. 299,454; B., 1929, 459.

Separately recovering soluble substances in a coarse granular condition. F. JEREMIASSEN, Assr. to A./S. KRYSTAL (U.S.P. 1,751,740, 25.3.30. Appl., 24.12.25. Norw., 12.1.25).—See F.P. 612,993; B., 1927, 463.

Filter. R. KNIESEL, Assr. to BAMAG-MEGUIN A.-G. (U.S.P. 1,751,819, 25.3.30. Appl., 22.6.28. Ger., 20.2.28).—See B.P. 304,967; B., 1929, 308.

[Filter] fabric and treatment of subdivided materials. S. C. SMITH (U.S.P. 1,752,210, 25.3.30. Appl., 31.3.28. U.K., 5.4.27).—See B.P. 298,172; B., 1929, 3.

[Discharge of liquids from] centrifugal separators. E. A. FORSBERG, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,749,764, 11.3.30. Appl., 6.2.28. Swed., 12.2.27).—See B.P. 285,088; B., 1928, 658.

Device for removing solid particles from flue gases. M. PRÉTOT and F. ULLMANN (U.S.P. 1,749,260, 4.3.30. Appl., 5.12.27. Fr., 4.12.26).—See B.P. 281,651; B., 1929, 269.

(A) Refrigerators, (B) method of refrigeration, using solid carbon dioxide as refrigerant. J. J. V. ARMSTRONG. From DRY ICE EQUIPMENT CORP. (B.P. 326,196—7, 1.10.28).

Tanks for transport of volatile liquids. G. V. TONTET (B.P. 325,766, 11.6.29).

Recovering fine material from settling tanks etc. (B.P. 325,320).—See II. Crystallisation (B.P. 325,650 and 325,757).—See VII. Porous objects (U.S.P. 1,742,515).—See VIII. Briquetting of flue dust (U.S.P. 1,741,544).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Choice of liquid fuels. E. W. STEINITZ (Papier-Fabr., 1930, 28, 154—158).—The calorific value and the sp. gr. are unsuitable as criteria for the evaluation of liquid fuels. The volatility as shown by the distillation curve is of greater value for this purpose. Various types of motor spirits and Diesel oils, their distillation curves, anti-knocking properties, and comparative prices, are briefly discussed.

A. B. MANNING.

Blast lamp for natural gas. H. H. STRAIN (Ind. Eng. Chem. [Anal.], 1930, 2, 128).—The burner consists

of a cylindrical tube mounted on a metal base which is pivoted on a metal stand. The burner tube is provided with a sliding top and a rotating air shutter. The gas enters the burner through a jet in the burner-tube base and the compressed air enters through a longer curved tube ending in a jet on a level with the sliding top. Both jets screw into place, and are replaceable by others of various sizes.

H. S. GARLICK.

Determination of sulphur in illuminating gas.

H. A. J. PIETERS (Z. anal. Chem., 1930, 80, 12—20).—The colorimetric determination of the total sulphur in gas as colloidal lead sulphide gives satisfactory results in the case of an unpurified gas or one rich in sulphur, but the combustion method is preferable for gas containing relatively little of this constituent; in the latter case the determination of the sulphuric acid formed must take place gravimetrically as barium sulphate and not by titration, as the volumetric process yields results which are much too high when but little sulphur is present. The sulphur present as hydrogen sulphide is best determined by passing the gas through a solution of cadmium acetate in dilute acetic acid, then adding standard iodine solution, and thoroughly shaking; excess iodine is back-titrated with 0.05*N*-thiosulphate.

H. F. HARWOOD.

Utilisation of industrial vegetable residues. I.

Dry distillation. P. LEONE and F. OCCHIPINTI (Annali Chim. Appl., 1929, 19, 563—568).—Lemon pastazzo, representing the whole of the lemon after removal of the juice and essential oil, contains about 80% of moisture, the percentage composition of the dry matter being: fats, waxes, and resins 24—35; cellulose 19—22; pectic substances 14—16; mucilage, gum, and hemicellulose 10—14; lignin 8—9; dextrose 1—2.5; and ash 4—4.2. When distilled at 400—425°, 100 g. of the air-dried material (11.7% of water) yield 15 litres of gas of calorific value 2680, 40.5 g. of distillate, 9.5 g. of tar, and 25 g. of carbon. The distillate contains (%): acetic acid 4.51, methyl alcohol 1.33, ketones 0.64, acetone 0.48, reducing substances 3.89, and tar 4.02; the percentage composition of the gas is: CO₂ 51.9, CO 18.0, heavy hydrocarbons 0.6, CH₄ 23.1, H₂ 2.0, and N₂ 4.5; the granular carbon contains: C 72.23, H 3.76, N 0.36, and S 0.27%, and has calorific value 4600; the tar has *d*₄¹⁵ 0.9925. Exhausted sansa contains (on dry matter): fats soluble in carbon disulphide 0.45; fats, resins, and waxes soluble in alcohol or benzene 12.2; cellulose 44.0; lignin 36.9; ash 4.1. On distillation, 100 g. of the dry sansa yield 14.3 litres of gas, 30.8 g. of distillate, 6.3 g. of tar, and 35 g. of carbon. The distillate contains (%): acetic acid 12.03, methyl alcohol 4.58, acetone 0.74, reducing substances 3.94, and tar 2.0. The gas consists of CO₂ 51.9, CO 22.5, H₂ 3.3, CH₄ 21.9, and N₂ 0.4%, the calorific value being 2420. The charcoal is granular, contains 11.05% of ash, and has apparent *d* 0.35 and calorific value 5400.

T. H. POPE.

Modernisation of a wood-distillation plant. J.

SCHWYZER (Chem.-Ztg., 1930, 54, 65—66, 86—87, 106—108, 122—124).—A modernised plant for the carbonisation of deciduous woods is described. The ten retorts, each of 175 cub. m. capacity, are of thick wrought

iron and supported by a lattice of angle irons. Each is in an independent setting fired by producer gas, and the exit gases are used for drying acetate of lime. The retorts are charged from above by a wagon on a runway, an operation requiring 1½ days. For the first 2½ days the heat is applied to the upper part of the retort, then by changing dampers to the lower part. Cooling is effected by opening airports. Pairs of retorts are connected to cylindrical condensers from which the undensified gases pass at the beginning of the distillation to the air, and afterwards to the washing plant. The condensate from the first cooler is stored separately. The gases pass by a copper pipe to a packed scrubber tower designed, for reasons of safety, to impart a minimum back-pressure. Here they are washed with an alkaline liquor and pass to the producer-gas flues. Such large retorts give a better charcoal than small ones, and an increase of 10—15% in the yields of acetic acid and methyl alcohol; they are also more economical in fuel and labour. The distillate settling tanks are large enough to take 15 days' output. The crude wood spirit is distilled in a 6000-litre copper still, and the distillate passes through two acetic acid absorption vessels, which are charged with liquor from the scrubber to which lime has been added. The residue from the still is run down to the crude acetic acid still. This is of 3600 litres capacity, and is also connected to two receivers containing a solution of 8% CaO content. As foaming does not occur after the methyl alcohol and acetone have been driven off, this still is worked much more quickly. The residual tar passes to a vacuum still. The separation of the distillation into stages as above tends to economy in first cost and repairs. The distillate from the tar, consisting of acetic acid and its homologues, is purified by passing over sodium dichromate and sulphuric acid and fractionating in a high Raschig column. The calcium acetate solution is filterpressed and evaporated *in vacuo*. The vapours from the crude spirit still are condensed, stirred with milk of lime, and allowed to settle. It must be definitely alkaline or the methyl alcohol produced will be discoloured. The product contains about 15% of methyl alcohol, 2% of methyl acetate, and 2% of acetone, together with allyl alcohol, tar oil, and resins. It is rectified in a still consisting of two boilers connected to one column. These two boilers will work either into the column or into each other, and by the system described will give a resin-free distillate in one operation. This is divided into two fractions, which are further redistilled with separation of forerunnings containing acetone and permanganate treatments. The alcohols are recovered practically quantitatively. The calcium acetate is placed in a still connected to a cooler collecting vessel and column, and sulphuric acid is slowly run in. The acetic acid is then distilled off by indirect steam under vacuum. The tar is distilled to give the following fractions: crude acetic acid, light oil, and creosote. The light oil is then washed, and further fractionated to "light benzine" and "turpentine substitute."

C. IRWIN.

Crude oil from bore hole No. 20 in Chusovaya (Perm). S. S. NAMETKIN (Nef. Choz., 1929, 17, 79—82).—Characteristics of the oil and its distillates

are recorded. The bore-hole water is noteworthy for its high content of ammonium ions and for the absence of carbonate and hydrogen carbonate ions.

CHEMICAL ABSTRACTS.

Viscosities of crude-oil products. I. D. AFANASEV (Neft. Choz., 1929, 17, 218—229).—Calculated and observed viscosities of various gasolines, kerosenes, and lubricating oils were in agreement when Ubbelohde's equation was used. Intermediate points on the viscosity-temperature curve were calculated. Changes of viscosity on treatment of turbine oil with fuming sulphuric acid and of spindle oil with concentrated sulphuric acid were determined. CHEMICAL ABSTRACTS.

Thermal conductivity of petroleum products. V. ZHUZE (Azerbeid. Neft. Choz., 1929, No. 8—9, 70—80).—Thermal conductivities of distillates from Baku crude oils, determined by Schleiermacher's and Winkelmann's methods, are recorded.

CHEMICAL ABSTRACTS.

Determination of aromatics in gasoline by the nitrobenzene method. A. DOBRYANSKI and I. KHESIN (Azerbeid. Neft. Choz., 1929, No. 8—9, 80—84).—The nitrobenzene coefficient diminishes rapidly with increase in concentration of aromatic compounds, but is independent of the nature of the gasoline. The following coefficients (coefficient, and aromatic compounds [vol.-%] for 1—13° depression, respectively) are recommended for oils pretreated with sulphuric acid as usual: 1.37, 0.8; 1.34, 1.5; 1.30, 2.3; 1.27, 3.0; 1.24, 3.9; 1.21, 4.8; 1.18, 5.8; 1.15, 6.8; 1.12, 7.9; 1.09, 9.1; 1.06, 10.6; 1.03, 12.1; 1.00, 14.0.

CHEMICAL ABSTRACTS.

Composition of cracked gasoline from Baku and Grozni. I. E. BESPOLOV (Azerbeid. Neft. Choz., 1929, No. 6—7, 79—91).—The product (of Vickers plant cracking mazout from Surakhani) has a low initial b.p. and good anti-detonating properties.

CHEMICAL ABSTRACTS.

Gasification of heavy oils. M. CHILOWSKY (Compt. rend., 1930, 190, 490—492).—The necessary and sufficient conditions for the gasification of heavy oils with production of no fixed carbon and not more than 1% of tar are that (i) the oil be blown in by air in an atomised state; (ii) the air used for this purpose be heated above the inflammation temperature of the oil (best at 500—700°); and (iii) the flow of oil and gases be so arranged that the walls of the combustion chamber are maintained at about 1000°. In this way a heavy oil distilling at 230—370° and ground-nut oil yielded gases of the following compositions, respectively: CO 13.8, 17.95; CH₄ 9.1, 7.3; C₂H₄ 4.4, 5.6; H 8.6, 4.8; N 59.3, 57.8; CO₂ 4.8, 5.9%. The theory of the process is discussed and its application to internal-combustion engines, in which its use would allow of a compression ratio of 8, is advocated, a reduction of 75% in expense being claimed.

C. A. SILBERRAD.

Modification of lamp method for determination of total sulphur in petroleum distillates. A. E. WOOD and W. MATTOX (Ind. Eng. Chem. [Anal.], 1930, 2, 24—25).—Natural-gas gasoline or other light distillate is placed in a test-tube graduated in tenths of a c.c., and a wick, consisting of two strands of cotton

fibre extending almost to the bottom of the test-tube, are inserted in a glass tube (12 cm. × 4 mm.) held in a cork of appropriate size. The graduated test-tube is placed in an ice-water bath provided with a levelling attachment. By regulating the height of the cooling liquid with reference to the gasoline in the test-tube a steady flame can be maintained indefinitely. After adjustments, the volume of gasoline in the test-tube is read and the flame immediately placed under the absorption apparatus of the standard lamp method for sulphur determination. Burning is continued for about 90 min. or until about 2 c.c. of the gasoline have been consumed, when the flame is extinguished and the volume of the gasoline again read. For ordinary gasoline or kerosene the ice-water bath is dispensed with, and for heavy distillates free from tarry or asphaltic matter a heated liquid bath is used. The method is not suitable for crude oil. H. S. GARLICK.

Determination of sulphur in gasoline. G. EDGAR and G. CALINGAERT (Ind. Eng. Chem. [Anal.], 1930, 2, 104—106).—Several modifications of the standard A.S.T.M. apparatus are described, viz., means for adjusting the flame of the standard lamp, lamps permitting adjustment of the flame during the determination and allowing the amount of fuel burned to be determined by volume, a new type of absorption tube, and a modified chimney permitting the elimination of the blank determination, all of which facilitate routine handling of large numbers of samples. The results obtained are essentially the same as those obtained by the standard method. H. S. GARLICK.

Determination of vapour pressures of naphthas. J. C. STAUFFER, J. K. ROBERTS, and W. G. WHITMAN (Ind. Eng. Chem. [Anal.], 1930, 2, 88—91).—Measured volumes of dry air are bubbled through the naphtha sample and the increase in volume is measured. Saturation of the air with hydrocarbon vapour is obtained by reference to the vapour pressure of pure organic liquids of known vapour pressures. The vapour pressure is calculated from measurements of the total gaseous volumes, the decrease in liquid volume, and the pressure existing in the system. The determined vapour pressures are plotted against the percentage evaporation and the initial vapour pressure is estimated by extrapolation of the curve to 0% loss. The vapour pressures determined by this method check with those computed by means of Raoult's law from the fractional analyses of the naphthas, and afford a convenient means for investigating evaporation losses during storage or transportation and the performance of stabilising and condensing equipment. H. S. GARLICK.

Behaviour of hydrocarbons in high-tension discharge. H. BECKER (Wiss. Veröff. Siemens-Konz., 1929, 8, [2], 199—217).—On passing a high-tension discharge through a saturated paraffin oil in the Siemens ozonising tube, hydrogen is evolved and a higher paraffin formed thus: $C_nH_{2n+2} + C_mH_{2m+2} = H_2 + C_{(n+m)}H_{(n+m)+2}$. Under the same conditions unsaturated hydrocarbons yield mixtures of saturated hydrocarbons of high mol. wt., the hydrogen evolved in the first reaction being absorbed again at the double linking. Cyclic hydrocarbons behave in a complex manner, mixtures of satur-

ated and unsaturated compounds being formed from degradation products of the original hydrocarbons. Thus, decahydronaphthalene (decalin) yields butane and small quantities of various unsaturated hydrocarbons together with up to 25% of resinous condensation products of high mol. wt. and b.p. above 200° (<1 mm.). Tetralin, when treated in air, yields hydrogen cyanide and other compounds containing nitrogen, but in hydrogen a yield of 23% of a colourless almost solid resin is obtained if the tetralin is treated in the vapour form. If the operation is conducted in a Geissler tube the resin produced yields, after extraction with acetone, colourless crystals of *ditetralyl*, $C_{20}H_{22}$, formed by the condensation of two mols. of tetralin with the elimination of 1 mol. of hydrogen. From the remaining resin two fractions were obtained by distillation *in vacuo*, viz., a yellow oil (n_D^{20} 1.5948) and colourless crystals. Methane yields small quantities of liquid hydrocarbons when subjected to the silent electric discharge in the Siemens ozonising tube.

A. R. POWELL.

Cracking of mineral oils in presence of the electric arc. G. FESTER and C. CHRISTEN (Z. Elektrochem., 1930, 36, 17—20).—Mineral oil, through which a current of hydrogen gas was passed, and above the surface of which an electric arc was made to operate between electrodes formed of rods of either nickel or iron, was subjected to distillation. Numerous data are given of the compositions of the cracked gases, and of the distillation ranges of the cracked distillates.

H. T. S. BRITTON.

Action of high-tension alternating currents on natural brine-petroleum emulsions. S. STARZEWSKI (Przemysl Chem., 1930, 14, 97—105).—The frequency with which sparking takes place between two electrodes immersed in brine-petroleum emulsions increases with increasing voltage, with decreasing distance between the electrodes, and with the temperature of the emulsion. Coagulation of the dispersed phase is more rapid and complete in the absence of sparking. The optimal temperature of demulsification has to be experimentally determined for each emulsion; for two Galician natural water-in-oil emulsions studied, this temperature was about 55°. It is concluded that the electrical method is not, in its present form, practically applicable to the separation of such emulsions.

R. TRUSZKOWSKI.

PATENTS.

Cleaning of carbonaceous materials. CLEAN COAL CO., LTD., R. LESSING, R. H. ALLEN, and E. S. GYNGELL (B.P. 325,031, 27.11.28. Cf. B.P. 276,723 and 282,874; B., 1927, 866; 1928, 179).—The materials are subjected to gravity separation in an aqueous solution of suitable density, the bath liquor being given an upward current of controlled magnitude greater than about 6 in./min. Automatic means are provided for maintaining a constant liquid level in the bath.

A. B. MANNING.

Apparatus for separating coal from dirt and like foreign substances. W. H. BERRISFORD (B.P. 325,284, 17.11.28).—The separation is based on the different rates of travel of the coal and the dirt along a plate of hard material, preferably glass, on to which the impure coal

is allowed to fall, the coal receiver being separated from the edge of the plate by a gap into which the dirt falls. The plate is curved and is so arranged that the direction of travel of the coal and dirt is substantially horizontal when they reach the lower edge.

A. B. MANNING.

Apparatus for recovering fine material, and more particularly fine coal, from settling tanks or towers. COPPÉE Co. (GREAT BRITAIN), LTD., and T. H. HUNTER (B.P. 325,320, 6.12.28).—A power-operated valve, which is either controlled by an operator from the point where the material is discharged or is automatically opened at predetermined intervals of time, is provided at the bottom of the settling tank. The particles of material that collect in the tank are thereby discharged as required, and are forced by the head of material in the tank to a higher level. The material is discharged into a second tank, which is fitted with an overflow whereby any excess material delivered thereto is returned into circulation. Revolving nozzles are provided in the sides of the tank in order to deal with any undesirable accumulation of solid material therein.

A. B. MANNING.

Preparation of pulverised solid fuels. G. S. LOY (B.P. 301,887, 27.7.28. Fr., 9.12.27).—The raw fuel is subjected to drying, pulverising, and sorting operations in a closed circuit, from which the required supply of finely-pulverised material is automatically withdrawn by the suction of the blowers which feed the fuel to the burners. The whole supply of dust in excess of the actual consumption is recirculated, and fresh raw fuel is automatically added to the circuit in amount equivalent to the pulverised fuel consumed. A form of apparatus for carrying out the process is described.

A. B. MANNING.

Pretreatment of moist fuels, particularly lignites, prior to carbonisation or gasification. H. FLEISSNER (Austr.P. 109,406, 7.8.25).—The fuel is treated with steam in a closed vessel under conditions that do not permit the escape of the moisture contained in it, and is then dried by passing through it the hot gases from the carbonisation or gasification process itself.

A. B. MANNING.

Treatment of peat for production of fuel therefrom. D. W. BERLIN and J. A. BRINCK (B.P. 325,444, 22.5.29).—The raw peat is disintegrated and formed into a liquid sludge with water, and is then subjected to wet carbonisation. The bulk of the residual water is removed from the product by centrifuging, followed, if necessary, by compression in a filter press. The material is further dried by heat treatment and may finally be briquetted.

A. B. MANNING.

Treatment of peat. G. E. A. CUYLITS (F.P. 634,826, 21.5.27).—Crude peat is treated with one or more diastases in order to separate part of the water in the hydro-cellulose in the material. The product, separated from the liquid by centrifuging or similar means, appears in powdered, granular, or other form.

A. B. MANNING.

Vertical continuous distillation retorts. J. PIETERS (B.P. 325,735, 5.4.29).—A rigid casing, slightly smaller in cross-section than the upper end of the distillation chamber, is provided between the latter and

the feed hopper. A piston, open in the centre and forming a frame the sides of which have a vertical cross-section of double-wedge form, can be moved vertically within the casing by means of bars which are attached to its two ends and can be operated from above the retort by a discharging device. Clogging of the material in the upper end of the distillation chamber is thereby prevented. A. B. MANNING.

Apparatus for heat treatment of solid or semi-solid carbonaceous material. W. E. TRENT (B.P. 325,324, 8.12.28).—The retorts and combustion chambers of the apparatus are arranged side by side, and the heat is transmitted from the latter to the former by means of metal rods extending horizontally through both. The material to be carbonised is introduced into the retorts on trays which are supported therein by the conducting rods. Means are provided for passing steam through the retorts and for withdrawing and condensing the vapours evolved. A. B. MANNING.

[Regenerative] coke ovens. E. LEYH, Assee. of H. KOPPERS A.-G. (B.P. 301,386, 14.11.28. Ger., 28.11.27).—The heating walls are connected in pairs by conduits passing above the oven chambers, and are so arranged that during any one draught alternation the gases pass unidirectionally (up or down) through each wall. A regenerator below each chamber is divided into two sections by a partition wall transverse to the length of the chamber. The sections are connected, respectively, to two adjacent heating walls, each section being connected directly to the portion of the heating wall immediately above it, and by means of an extension passage to the portion of the wall lying above the companion regenerator section. A. B. MANNING.

Regenerative coke ovens. F. TOTZEK, Assee. of H. KOPPERS A.-G. (B.P. 302,225 and Addn. B.P. 303,107, [A] 14.11.28, [B] 15.11.28. Ger., [A] 12.12.27, [B] 28.12.27).—(A) A coke oven has a regenerative heating system comprising cavity-heating walls alternating with the oven chambers, two regenerators or groups of regenerators extending beneath the oven transversely to the heating walls, and two base channels extending lengthwise beneath each oven chamber, each connected, respectively, with one of the regenerators and with the heating walls. The two base channels are situated one above the other, and are arranged to cross scissorwise midway in their lengths. (B) The heating walls, instead of consisting of a series of twin flues, may be coupled in pairs in such a manner that during any one cycle of draught alternation the gases pass unidirectionally through each wall. The base channels are so arranged that single channels each connected with two walls alternate with pairs of channels each connected with one wall only. A. B. MANNING.

Apparatus for cooling coke. HUMPHREYS & GLASGOW, LTD., and J. S. HAUG (B.P. 310,283, 22.4.29. U.S., 21.4.28).—An inclined coke container is provided with a sloping wharf which supports the hot coke. The wharf is composed of overlapping plates between which are gas passages. Above the container and connected therewith is a horizontal water-tube boiler so arranged that air or products of combustion may be circulated

by means of a fan through the hot coke and through the boiler tubes in succession. A. B. MANNING.

Dry-cooling of coke. J. RUDE (B.P. 325,120, 21.2.29).—The coke is cooled by the circulation through it of steam under pressure. Subsidiary charging and discharging chambers are provided so that the coke-cooling chamber itself never comes into direct communication with the atmosphere. The increased pressure of the circuit is maintained by the addition of water or fresh steam thereto, and the surplus steam is removed in a highly superheated state and utilised, e.g., for the production of water-gas. A. B. MANNING.

Burning of pulverised fuel. T. A. MCGEE (B.P. 314,979, 7.5.29. Can., 6.7.28).—The pulverised combustible mixture is injected into the furnace, wherein the volatile matter and part of the carbon are burnt while the particles are in suspension; the residual carbon falls on to the furnace grate and is burnt by means of air flowing through the grate. The latter is formed of grate castings which are supplied with air-inlet and ash-outlet passages, and are supported on tubes which are water-cooled to prevent clinker formation. Provision is made for the discharge of pieces of clinker which are too large to pass through the ash-outlet passages, and for the return to the furnace of the residual carbon deposited from the flue gases. A. B. MANNING.

Low-temperature carbonisation of granular or dusty fuels. C. ARNEMANN (G.P. 460,420, 6.5.22. Addn. to G.P. 458,879; B., 1930, 311).—The combustion gases from a powdered fuel-fired furnace are mixed with the cold, detarred distillation gases in such proportion that the temperature of the mixed gases is just high enough to carbonise the fuel and produce a gas which is still combustible. A. B. MANNING.

Low-temperature carbonisation process. CHEM.-TECHN. GES.M.B.H. (G.P. 459,824, 25.12.23).—The material is carbonised in vertical or horizontal cells which are combined in a rotatable ring in such a manner that each cell can be suitably heated. The material is fed into the cells at one position in the ring and discharged at another. Apparatus for carrying out the process is described. A. B. MANNING.

Manufacture of activated carbon. E. BERL (B.P. 324,729, 7.11.28. Addn. to B.P. 283,968; B., 1929, 465).—Carbonaceous substances which can be activated, such as wood, peat, charcoal, brown coal coke, etc., are added to the acid tars which have been neutralised with potassium compounds, and the mixture, moulded if desired, is heated to activating temperatures, preferably in the presence of oxidising gases or vapours. The organic substances in the tars may first be separated from the excess sulphuric acid by the addition of water before they are submitted to the above process. A. B. MANNING.

Production of highly active carbon. CHEM. WERKE CARBON GES.M.B.H. (G.P. 460,697, 10.5.27).—Organic chlorides, e.g., carbon tetrachloride, trichloroethylene, chlorinated naphthalene, in the gaseous, vapour, or finely-divided liquid or solid state, are allowed to react with powdered metals, e.g., zinc, aluminium, iron,

magnesium, in a heated reaction chamber, and the product is washed with water or acidified water. Substances which modify the rate of reaction, *e.g.*, kieselguhr, may be incorporated with the metals. Carbonaceous substances or charcoal may also be introduced into the chamber.

A. B. MANNING.

Manufacture of carbon black. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,959 and 325,207, [A, B] 3.8. and 19.12.29).—(A) Gaseous unsaturated hydrocarbons are treated with a dehydrogenating catalyst consisting of a metal of the iron group, or a compound thereof, at a moderate temperature, usually between 300° and 450°, and under elevated pressure. In (A) the treatment may be carried in the presence of other gases *e.g.*, hydrogen, water vapour, oxides of carbon, methane. In (B) the catalyst is activated by the addition of other metallic oxides or compounds, such as the oxides or hydroxides of zinc, copper, chromium, vanadium, uranium, the alkali or alkaline-earth metals, etc. The carbon blacks produced are particularly suitable for vulcanising or pigmenting purposes.

A. B. MANNING.

Determination of volatile matter in combustible material. G. H. HALVORSON (U.S.P. 1,743,035, 7.1.30. Appl., 15.1.26).—The crucible containing the sample of material is enclosed in a casing of refractory material, *e.g.*, magnesite, provided with a closely fitting cover, and is placed directly in a muffle at 950°. A uniform rate of heating is thereby attained and comparable results for the volatile matter content, given by the loss of weight of the sample, are obtained.

A. B. MANNING.

Gas producer. A. BÉCHEVOT (F.P. 632,574, 2.8.26).—The producer comprises a chamber of refractory material divided into an upper and a lower part, with a metal retort which serves as a fuel container in the former. Between the outer cover of the generator and the divided chamber is a free space wherein the air for combustion is preheated.

A. B. MANNING.

Apparatus for the continuous production of illuminating gas. É. FANCONNIER and A. SIMPÈRE (F.P. 637,179, 4.11.26).—The air carburettor consists of several inclined and parallel tubes arranged in a vertical shaft and surrounded by water. They are open at both ends, the lower ends opening into a vertical tube. The air charged with the atomised carburetting liquid in the apparatus is cooled in the tubes.

A. B. MANNING.

Water-gas generating apparatus. TULLY, SONS & CO., LTD., and C. B. TULLY (B.P. 325,437, 15.5.29).—The apparatus comprises two or more retorts arranged above a gas generator in such a manner that the blow gases can pass up through flues or checker-work between, or surrounding the retorts, while the water-gas is passed through the fuel in the retorts and thence to suitable gas-purifying apparatus. Uniform heating of the fuel is ensured by providing each retort with a separate valve-controlled outlet, the valves being automatically opened periodically and in sequence so that the water-gas passes through the retorts in turn.

A. B. MANNING.

Manufacture of water-gas. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 324,867, 23.4.29).—

Steam is superheated alternately or simultaneously inside and outside the tubes of a water-gas generator tubular superheater (*cf.* B.P. 288,463; B., 1928, 437). The superheater tubes are heated by the blow gases and the steam is then superheated by being passed through the tubes during the up-run and round them during the down-run. The steam superheated may be partly that employed in the water-gas process and partly steam for some other purpose.

A. B. MANNING.

Production of mixed gas from coal gas and other gases. STETTINER CHAMOTTE-FABR. A.-G. VORM. DIDIER (G.P. 459,653, 28.9.20).—A generator with a carbonising zone for bituminous coal is also provided with chambers wherein inferior fuels (wood, brown coal, peat) may be carbonised and gasified; the gases from the latter are then mixed with the carbonisation gases from the bituminous coal. The gasification chamber for the inferior fuel may be directly connected with the retorts which are charged with that fuel. Apparatus for carrying out the process is described.

A. B. MANNING.

Production of gas from wood, waste materials, etc. J. LERAT (F.P. 637,140, 29.10.26).—The material is charged into a container and the lower part of the charge is burnt with air which is supplied through a tube from above.

A. B. MANNING.

Dismountable apparatus for the carbonisation and distillation of wood. J. BONELLO and P. AUBÉ (F.P. 632,377, 23.7.26).—The charge is heated in a closed container by means of hot gases from an external source which pass up through the centre. The air required for the carbonisation of the wood is drawn through a series of perforations around the lower part of the container. The combustion chamber is closed with a cover having a damper to prevent too rapid removal of the products of combustion, which are eventually collected in an air-cooled condenser consisting of a series of vertical pipes.

A. R. POWELL.

Production of mechanical work in connexion with the liquefaction of coal. E. GIGER (Swiss P. 123,928 and 124,135, [A] 11.1.27, [B] 6.1.27).—(A) The waste gases from the hydrogenation process, together with the vapours and suspended substances contained therein, are burnt as near as possible to the place of production and while still under pressure, and are used as an engine fuel for the production of useful mechanical work. The waste heat of the gases leaving the engine is utilised in the hydrogenation process. (B) The high-pressure waste gases are utilised as they leave the apparatus in one or more machines of the turbine type. The hydrogen is then recovered from these gases by treating them with steam under pressure, and is returned to the hydrogenating vessel. The carbon dioxide thereby formed may likewise be allowed to expand in a turbine with the production of useful work.

A. B. MANNING.

Carbonisation of briquetted fuel. E. GOUTAL and H. HENNEBUTTE (F.P. 632,856, 14.4.27).—Porous charcoal obtained from wood, peat, or lignite, is briquetted, using tar as the binding agent, and the briquettes are carbonised surrounded by the powdered material.

A. B. MANNING.

Treatment of gases containing hydrogen and unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,309, 26.11.28).—The unsaturated hydrocarbons in gaseous mixtures obtained by the thermal treatment of gases containing hydrocarbons are recovered by adsorption under pressure on activated carbon or other solid adsorbent. The residual compressed gas, principally hydrogen, is utilised in some reaction requiring hydrogen under pressure, *e.g.*, in the destructive hydrogenation of coal, tar, etc.

A. B. MANNING.

Compositions for the purification of gases. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 309,585, 27.3.29. Fr., 13.4.28).—Gases from the distillation or gasification of carbonaceous material are purified by being passed over a composition comprising a metal or metallic oxide, *e.g.*, of nickel, copper, supported on an inert carrier, *e.g.*, pumice, kieselguhr, the proportions of metal and carrier being such that the composition will not retain more than 10% of its weight of sulphur. This ensures that on regeneration of the composition in a current of air the temperature will not rise sufficiently to reduce appreciably the activity of the metal.

A. B. MANNING.

Refining of low-temperature tar, tar oils of any origin, crude benzene, and products obtained by destructive hydrogenation of carbonaceous materials. F. HOFMANN and C. WULFF (B.P. 298,484, 8.10.28. Ger., 6.10.27).—The crude material is treated with 2–3% of a concentrated solution of hydrogen chloride in ethyl or methyl alcohol, or other organic solvent, after a pretreatment, if desired, with gaseous or aqueous hydrogen chloride. The heavier oils are preferably diluted with benzene before treatment. The phenols are refined simultaneously with the neutral oils and may be extracted in pure form from the product.

A. B. MANNING.

Simultaneous removal and emulsification of tars from tar-yielding media. T. W. BARBER (B.P. 325,484, 8.10.28).—The tar-yielding medium, *e.g.*, crude petroleum, previously neutralised if necessary, is treated with an alkali (caustic soda) in such a manner that the tars are removed and emulsified at the same time. The apparatus described in B.P. 302,745 and 323,758 (B., 1929, 307; 1930, 270) may be used for adjusting the exact proportion of medium and alkali and for effecting the subsequent emulsification, respectively.

A. B. MANNING.

Treatment of asphaltic bituminous and resinous products. P. GIRARD and F. PETIT (B.P. 315,381, 2.11.28. Fr., 13.7.28).—The asphaltic residual oil from any cracking process is treated with an alkaline earth, *e.g.*, about 4–6% of lime, the amount depending on the oxygen content of the asphalt, at a temperature above 300° but below that at which cracking would normally occur, and at atmospheric pressure or above. The asphalt may advantageously be submitted to a preliminary oxidation. It is converted largely into gasoline and non-asphaltic products suitable for re-treatment in the cracking process.

A. B. MANNING.

Retort [for distillation of shale etc.]. L. G. NILSON, Assr. to INTERNAT. BITUMEN OIL CORP. (U.S.P.

1,742,273, 7.1.30. Appl., 15.1.26).—The material is passed through a double-walled, cylindrical, rotary retort. The annular space between the walls is packed with a heat-equalising material, *e.g.*, metal bars, metal filings, or sand, and is divided into sections by rings of heat-insulating material, corresponding with zones of successively higher temperatures within the retort. The retort is surrounded by a combustion chamber which is also divided into sections by vertical walls partially surrounding the retort. It is heated from below by gas burners.

A. B. MANNING.

Decomposition of hydrocarbons to produce hydrocarbons of higher mol. wt. T. S. WHEELER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 324,939, 3.9.28).—Methane or ethane is decomposed by thermal treatment to form aromatic hydrocarbons, after the separation of which the hydrogen is removed from the residual gas prior to further thermal treatment, *e.g.*, by passing over copper oxide at 250°, by diffusion, or by interaction with added carbon monoxide in the presence of a nickel catalyst.

A. B. MANNING.

Splitting of hydrocarbons. N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 302,349, 21.11.28. Holl., 16.12.27).—Unsaturated hydrocarbons may be obtained at comparatively low temperatures from light hydrocarbons such as propane and butane when the heating is carried out in the presence of halogens or halogen-containing substances. Organic or hydrogen halides, or sulphur-, phosphorus-, and oxygen-containing halogen compounds may be used. Metals and metallic salts may be added as activators. The addition of a few hundredths vol.-% of iodine to propane at 575° causes the production of 15.5% of unsaturated substances, mainly ethylene, whilst at 650° 35.3% of unsaturated substances is obtained. Butane in the presence of carbon tetrachloride at 600° gives butylene 6%, propylene 14.3%, and ethylene 21.9%.

T. A. SMITH.

Manufacture of liquid hydrocarbons. J. L. FOHLEN (F.P. 631,927, 14.4.27).—Hydrogen or water-gas, steam, and methane, or ethane are injected into a blast-furnace and the exit gases are led over a nickel, cobalt, or platinum catalyst at 100–450°, preferably under pressure. With a zinc oxide catalyst alcohols are obtained; with aluminium chloride, polymerised products.

C. HOLLINS.

Manufacture of light-coloured [oxidation] products from paraffin wax, montan wax, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,903, 27.9.28).—Waxes etc. are oxidised to almost colourless products (substitutes for beeswax etc.) by air or oxygen at 120–180° in presence of small amounts of aqueous non-oxidising, strong acids or of an ansolvo-acid. Examples of such catalysts are 0.1% of 45% sulphuric acid, 0.1% of oxalic acid, 0.3% of the ansolvo-acid obtained from zinc chloride and acetic acid, *i.e.*, $[\text{ZnCl}_2, 2\text{AcO}]\text{H}_2$.

C. HOLLINS.

Coking of [petroleum] oils. D. R. WELLER and L. LINK, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,741,895, 31.12.29. Appl., 8.4.22).—Distillation down to the coke stage is carried out in a horizontal

cylindrical still surmounted by a dome of cross-section at least half that of the still. Vapour off-takes from the top of the dome are each at least one sixth of the cross-section of the dome. The period of distillation is expected to be 10—18 hrs., and these offtakes will deal with the vapours evolved during that period. The dome is fitted with a catch-tray and run-off so that wax-containing oil condensed in the dome may be separately run off. The vapours are carried to a series of fractionating towers the vapour inlets of which are at the bottom. Condensate from the towers may be led off to storage or returned to the still. Cuts may be taken from each tower. The uncondensed vapours leaving the last tower are liquefied in a condenser.

T. A. SMITH.

Purification of petroleum distillates. M. L. CHAPPELL and G. J. ZISER, ASSTS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,741,555, 31.12.29. Appl., 20.7.25).—Viscous petroleum oils or lubricating stocks are mixed with about an equal volume of aniline oil and heated to approx. 77° to effect homogeneous dissolution; the mixture is then cooled to below 27° until the saturated hydrocarbons separate from the aniline oil, the latter containing the unsaturated hydrocarbons, sulphur, and asphaltic and resinous compounds in solution. These are subsequently separated from the aniline by distillation in steam. H. S. GARLICK.

Apparatus for refining oil. C. W. STRATFORD and W. S. JAMES (U.S.P. 1,741,887, 31.12.29. Appl., 8.7.26).—Oil is injected through a control valve so that it strikes tangentially the inner surface of one of a pair of vertical cylinders each having openings in each end. Vapours and any entrained liquids are led from the upper opening in the first cylinder and are delivered to the interior of the second cylinder, substantially tangentially, thereby separating vapours from liquids in each of the cylinders. The separated liquids are removed from the lower ends of the cylinders through a liquid seal located exterior to each of the cylinders. H. S. GARLICK.

Oil-purifying apparatus. L. H. CLARK, ASST. to SHARPLES SPECIALTY CO. (U.S.P. 1,741,756, 31.12.29. Appl., 12.5.25).—Oil from electric installations, used crank-case oil, or other oils containing finely-divided carbon or similar impurities, are drawn by means of a pump through a pipe controlled by a valve to a closed heater and passed to a closed agitating tank. Aqueous treating reagent is drawn from a storage tank through a valve-controlled pipe having a by-pass, and passed to the agitating tank. Means are provided for introducing absorptive material into the agitating tank if desired. From the bottom of the agitating tank a valve-controlled pipe leads to a centrifugal separator. Oil separated from the mixture is discharged into a storage tank. H. S. GARLICK.

Treatment of hydrocarbon oils. C. J. GREEN-STREET, ASST. to GASOLINE CORP. (U.S.P. 1,740,691, 24.12.29. Appl., 4.8.27. Renewed 13.3.29).—Residual oil mixed with superheated steam is passed through cracking coils to well-lagged expansion vessels which also receive heated crude oil which has passed through a topping coil. Unvaporised oil settles out in these vessels and is not retreated. The uncondensed vapours

are passed through a series of six further expansion vessels, entering each vessel at the bottom and leaving at the top. A certain amount of condensation takes place in these vessels and the condensed oil is passed again through the cracking coil. The final expansion chamber is cooled by means of a coil so that only benzene vapours are uncondensed. The benzene vapours are passed to a separate condenser. T. A. SMITH.

(A) Fractionation of vapours from pressure stills and the like. (B) Operation of pressure stills. (C, D) Cracking of hydrocarbons. J. E. BELL, ASST. to SINCLAIR REFINING CO. (U.S.P. 1,741,275—7 and 1,741,357, 31.12.29. Appl., [A, B] 24.3.24, [C] 22.1.25, [D] 9.7.24).—The cracking unit consists of vertical tubes arranged in parallel in a furnace setting. A pressure still is arranged to receive hot oil from the upper ends of the heating tubes and a line is also taken from lower portion of the still to the bottom of the heating tubes so that oil may be circulated. Circulation is brought about by means of a pump the bearings of which are kept cool by pumping cool cracking stock into them. A drain for the removal of tar from the pressure still is provided. The cracking stock is preheated by using it as a cooling medium in a special dephlegmator tower in which it does not come in direct contact with the vapours from the still and also in a dephlegmator tower in which it does come in intimate contact with the hot vapours. Vapours from the dome of the pressure still are taken to the first dephlegmator tower up which they pursue a tortuous course through baffles over which cracking stock is flowing to the still. From this tower the vapours proceed to a second similar tower where the baffles are sprayed with condensate or the vapours are taken to a special tower in which the cooling is more definitely controlled. This tower consists of a number of superposed tube-boiler-like units, the vapours passing through the tubes, whilst the shells contain cracking stock. The reflux from the upper units can be distributed in desired amount over the lower units and the temperatures are controlled so that at any part the difference between the temperature of the gases and the cooling fluid is not more than 25°. From this tower cuts may be taken or undesired reflux returned to the pressure still. The vapours from the tower are passed to a condenser. The pumps provide efficient means of controlling each stage of the operation. T. A. SMITH.

Apparatus for evaporating and recovering sulphurous acid from mixtures of hydrocarbons and sulphur dioxide, possessing a large excess of sulphur dioxide. S. G. C. MARKS. From ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 325,315, 23.11.28).—The removal of sulphur dioxide from such mixtures in one stage is uneconomical owing to the large ratio of the gas to hydrocarbon. In continuous plant the sulphur dioxide has all to be raised to the temperature at which the hydrocarbon leaves this portion of the plant. In the present process its removal is carried out in stages, the mixture being passed through a series of vessels. The passage from one vessel to the next is downwards through a steam-jacketed pipe and then through an unjacketed, upward delivery pipe to the next vessel. The flow of steam and extract is counter-current. The sulphur dioxide exits from the evaporators

are connected to a common main. Instead of using separate vessels, a common vessel, which is divided by partitions, may be used. By this method it is possible to raise the temperature of the hydrocarbon extract to 85° at 4—6 atm. when it contains only about 6% SO₂. This reduces the load on the portion of the plant in which the final traces of gas are removed under vacuum.
T. A. SMITH.

Production of motor fuels. S. SCHANZER (DR. WERNICKE & BEYER) (B.P. 313,115, 17.11.28. Ger., 7.6.28. Addn. to B.P. 301,076; B., 1930, 314).—A mixture containing 2—5% of quinol, which was dissolved in 15 times its weight of benzene, is prepared in mineral oil boiling at 170—200°. Addition of 1% of this mixture to ordinary motor spirit confers improved qualities on the fuel and is 15—20% more economical.
T. A. SMITH.

Preparation of a lubricating oil containing rubber. SOC. MARITIME ET INDUSTRIELLE (F.P. 636,242, 20.6.27).—Lubricating oil (100 pts.) is mixed with 0.5—15% of rubber and 5—20% of graphite or molybdenite. The product is characterised by its high viscosity and adhesiveness.
A. B. MANNING.

Preparation of stabilised dispersions. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 324,663, 24.10.28).—Aqueous dispersions of mineral oils, tars, asphalts, rubber, etc. are stabilised by reversing the charge of the dispersed particles from negative to positive; before introducing the reversing agent, however, a protective colloid, e.g., 1—2% of gelatin, is added to the dispersion. Acids, basic dyes, solutions of multivalent metallic salts, positively-charged hydrosols such as freshly precipitated aluminium hydroxide, etc. may be used as reversing agents.
A. B. MANNING.

Rotary retort, kiln, or drum. G. CANTHENY, ASST. to INTERNAT. COAL CARBONIZATION CO. (U.S.P. 1,751,127, 18.3.30. Appl., 8.3.24. Ger., 24.3.23).—See B.P. 232,456; B., 1925, 491.

Regenerative heating structure [for retort oven batteries]. J. VAN ACKEREN, ASST. to KOPFERS CO. (U.S.P. 1,749,116, 4.3.30. Appl., 5.10.23).—See B.P. 222,818; B., 1925, 490.

Apparatus for carbonising or distilling materials. S. McEWEN, ASST. to COAL OIL EXTRACTION, LTD. (U.S.P. 1,749,641, 4.3.30. Appl., 11.7.29. U.K., 8.6.28).—See B.P. 316,734; B., 1929, 932.

Gas scrubber. W. A. S. CALDER and W. H. PALMER (U.S.P. 1,752,260, 25.3.30. Appl., 14.7.24).—See B.P. 240,224; B., 1925, 945.

Recovery of gas constituents from gas mixtures. M. SCHROEDER (U.S.P. 1,751,103, 18.3.30. Appl., 29.10.24. Ger., 12.8.24).—See G.P. 421,725; B., 1926, 321.

Manufacture of high-compression automotive distillate. C. J. PRATT (U.S.P. 1,752,264, 25.3.30. Appl., 13.7.27).—See B.P. 293,829; B., 1930, 180.

Regeneration or purification of lubricating oils. H. J. YOUNG, ASST. to SWAN, HUNTER & WIGHAM RICHARDSON, LTD. (U.S.P. 1,752,050, 25.3.30. Appl.,

22.3.28. U.K., 12.4.27).—See B.P. 292,300; B., 1928, 633.

[Multi-burner] coal-dust furnaces [for locomotives]. G. HAYN (B.P. 307,057, 13.2.29. Ger., 2.3.28).

Burners for liquid fuel. H. D. FITZPATRICK. From C. F. M. VAN BERKEL (B.P. 325,953, 19.1.29).

Apparatus for burning liquid fuel. FILMA OIL BURNERS, LTD., and A. MARSDEN (B.P. 326,388, 25.4. and 29.6.29).

Bringing liquids and gases into contact (B.P. 325,724). **Treatment of gases with solids and liquids** (B.P. 324,747). **Treatment of gases etc. catalytically** (B.P. 324,481).—See I. **Hydration of olefines** (B.P. 324,897). **Organic acids and their salts** (B.P. 324,538).—See III. **Thiocyanates** (B.P. 311,725).—See VII. **Production of carbon monoxide** (U.S.P. 1,742,750). **Floth-flotation of ores** (B.P. 301,832).—See X. **Purification of waste water** (B.P. 305,657).—See XXIII.

III.—ORGANIC INTERMEDIATES.

Direct synthetic urea process. H. J. KRASE, V. L. GADDY, and K. G. CLARK (Ind. Eng. Chem., 1930, 22, 289—293).—Liquid ammonia and liquid carbon dioxide are delivered at controlled rates by means of special pumps to a jacketed steel autoclave with lead lining, in which a reaction time of 1.83 hrs. at 150° is allowed for 40% conversion. Calculation of the heat balance on the basis of formation of ammonium carbamate, 40% of which is converted into urea and water, as far as data are available, shows a small balance available over that required to evaporate and heat the liquid reagents supplied, and to heat and melt the carbonate formed. The reaction mixture passes to a still, in which unchanged carbamate is decomposed, the carbon dioxide and ammonia being recovered, whilst the urea solution is discharged.
S. I. LEVY.

PATENTS.

Manufacture of ethylene from acetylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,152, 23.3.29).—Polymerisation is minimised by adding 5—20% by vol. of water vapour to the acetylene-hydrogen mixture. Preferably the gases are passed through an evaporator containing water at 95—96°. C. HOLLINS.

Hydration of [propylene and higher] olefines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,897, 1.11.28).—Propylene or a higher olefine is forced at a partial pressure above 10 atm. into dilute hydrochloric or sulphuric acid at 150—220° containing a compound of bismuth or copper. An apparatus for continuous working, in which the alcohol is distilled off under pressure as produced, is described. The process may be used to remove propylene etc. from gases containing ethylene.
C. HOLLINS.

Production of higher hydrocarbons [from methane]. F. FISCHER (B.P. 319,340, 15.12.28. Ger., 22.9.28).—A tower lined with silica is heated by furnace gases and when sufficiently hot a reverse (downward) current of methane is substituted. As soon as the

methane conversion ceases the furnace gases are again led in, and the alternation is repeated as often as desired, the conversion products being removed at the base of the tower. A heat exchange is arranged to preheat the methane and the combustion gases alternately.

C. HOLLINS.

Oxidation of organic compounds [hydrocarbons] of high mol. wt. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,492, 31.12.28).—As oxidising agents, nitrogen oxides, particularly nitrogen peroxide vaporised from the liquid tetroxide, are used at 48–80° and 2–6 atm. pressure.

C. HOLLINS.

Manufacture of trichloroethylene. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 302,321, 14.12.28. Ger., 14.12.27).—In the conversion of tetrachloroethane at about 500° into trichloroethylene the vapour of the former is passed at such velocity over the heated surface that a large proportion remains unchanged and is returned to the entering gas. An output of 50 tons per 24 hrs. per cub. m. of chamber space is reached when 1.2 kg. of tetrachloroethane are passed per hour at 460° over pumice impregnated with cupric chloride in a contact tube 3.8 cm. in diam. and 27 cm. long.

C. HOLLINS.

Manufacture of α -chloro- α -sulphoacetyl chloride. COMP. DE PROD. CHIM. ET ELECTROMÉTALLURG. ALAIS, FRÖGES, & CAMARGUE (F.P. 602,395, 25.11.24).—Trichloroethylene and 0.67 pt. of 100% sulphuric acid are refluxed for 20–25 hrs. at 80–88°.

C. HOLLINS.

Separation of formic acid from acetic acid. I. G. FARBENIND. A.-G. (B.P. 305,594, 7.2.29. Ger., 7.2.28).—An entraining liquid soluble in acetic acid, but not in formic acid, is added to the mixture of acids. On distillation the azeotropic mixture of formic acid, and added liquid (together with any water present) is first collected and separates into two layers; glacial acetic acid remains in the distilling vessel. Suitable entraining liquids are benzene, trichloroethylene, and carbon tetrachloride.

C. HOLLINS.

Recovery of anhydrous fatty acids from dilute aqueous solutions. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 636,825, 29.6.27. Belg., 1.7.26 and 23.5.27).—The solution is agitated with an ester of the acid to be extracted, with or without the addition of a hydrocarbon, and the ester layer is subjected to fractional distillation. Thus, 10% acetic acid solution is stirred with ethyl acetate; on distillation of the ester layer, which contains 3% of water, the constant-boiling mixture of ester and water distils at 70.4° and the pure ester at 77°, leaving a residue of pure acetic acid. A 20% solution of acetic acid is extracted with a mixture of 25% of benzene and 75% of ethyl acetate.

A. R. POWELL.

Production of acetaldehyde from ethyl alcohol. J. W. WOOLCOCK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 325,105, 15.2.29).—The vapour from 95% alcohol is passed with 35–75% of the oxygen necessary for oxidation, with or without steam, over a dehydrogenating catalyst (silver gauze) at 450–550°. A heat exchange is used to preheat the gases. With an alcohol-air ratio 1:1.1 by vol., 20% of the aldehyde formed results

from dehydrogenation and the exit gases contain 10% of hydrogen.

C. HOLLINS.

Manufacture of ethyl ether. K. E. STÄRBLÖM (Swed.P. 60,916, 11.6.24).—Alcohol vapour is led over kaolin at temperatures below that at which ethylene is formed.

C. HOLLINS.

Manufacture of dihalogenoethyl esters. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 325,115, 19.2.29).—Vinyl esters are treated with halogen below 0°, preferably in presence of diluent gas and/or liquid, e.g., carbon tetrachloride or benzene. The preparation of $\alpha\beta$ -dichloroethyl acetate, b.p. 55–56°/5 mm., $\alpha\beta$ -dibromoethyl acetate, b.p. 84°/5 mm., $\alpha\beta$ -dichloroethyl chloroacetate, and $\alpha\beta$ -dibromoethyl butyrate is described.

C. HOLLINS.

Manufacture of organic acid and their salts. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,538, 8.2.29).—Oxidation products of paraffin wax etc. are saponified with caustic alkali, and the resulting soap solution is diluted and emulsified with light petroleum at 70–100°; clear soap solution is separated by centrifuging, and the emulsion is split by shaking with methyl or ethyl alcohol. The soap solutions are acidified to precipitate pure fatty acids.

C. HOLLINS.

Preparation of organic peroxides. J. STRAUB (B.P. 309,118, 11.12.28. Holl., 5.4.28).—By preparing two or more acyl peroxides from mixtures of acid chlorides in the same vessel a low-melting and/or easily supercooled mixture of peroxides is obtained.

C. HOLLINS.

Manufacture of thiocarbamide from cyanamide. SOC. D'ÉTUDES CHIM. POUR L'IND. (F.P. 630,883, 9.2.27. Switz., 11.2.26).—Calcium sulphide is added to calcium cyanamide in water and carbon dioxide is led in at 75° and 5 atm. pressure. The nascent hydrogen sulphide reacts as formed to give thiocarbamide.

C. HOLLINS.

[Preparation of] diguanidines. M. PEYN, Assr. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,737,192, 26.11.29. Appl., 16.7.26. Ger., 4.8.25).— $\alpha\omega$ -Hexylenediamine or other polymethylenediamine is added to concentrated aqueous *S*-methylisothiocabamide sulphate, and the mixture warmed until no more methyl mercaptan escapes. The *NN'*-diguanyl derivative of the diamine is produced.

C. HOLLINS.

Manufacture of organic nitrogen compounds. KNOLL & Co., and K. F. SCHMIDT (G.P. 455,585, 15.4.25. Cf. also B.P. 257,418 and 250,897; B., 1926, 932; 1927, 172).—Further to U.S.P. 1,637,661 (B., 1927, 974), cyclopentanone gives with azoimide and zinc chloride *tetrahydrobenzotetrazole* [*tetrahydro-1:2-pyrido-1:5-tetrazole*]

$$\begin{array}{c} \text{CH}_2 \cdot \text{CH}_2 \cdot \text{N} \cdot \text{N} \\ | \qquad \qquad | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} : \text{N} \end{array} \text{N}, \text{ m.p. } 138^\circ, \text{ b.p. } 168^\circ$$

170°/6 mm.

C. HOLLINS.

Production of dye intermediates etc. [Conversion of aroylaromatic acids into aromatic acids.] H. A. E. DRESCHER, D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 324,935, 4.8.28).—Aroyl-aromatic acids or their alkaline-earth salts are heated for a few minutes at 300–350° in presence of the oxide or hydroxide of an alkaline-earth metal. *o*-Benzoylbenzoic acid with lime gives calcium benzoate; calcium

2- α -naphthoylbenzoate with lime gives calcium α -naphthoate and benzoate. C. HOLLINS.

Manufacture of water-soluble condensation products. [Synthetic tanning agents and resists for wool or silk.] A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 321,190, 28.6.28. Cf. B.P. 320,056; B., 1930, 315).—A mixture of an aromatic hydrocarbon or phenol and a natural resin is sulphonated and condensed with a benzyl chloride. The products may be salted out, and are used as tanning agents and as resists for wool and silk against direct dyes. Examples are: naphthalene and colophony are sulphonated with 100% sulphuric acid at 40–50°, and benzyl chloride is added; colophony, crude cresol, and benzyl chloride; colophony, phenol, and *o*-chlorobenzyl chloride. C. HOLLINS.

Hydrogenation catalysts. COMP. DE PROD. CHIM. ET ELECTROMÉTALLURG. ALAIS, FROGES, & CAMARGUE (F.P. 609,786, 28.4.25).—Mixtures of sodium and potassium, e.g., NaK or NaK₂, are more active catalysts than the single metals for the hydrogenation, e.g., of naphthalene. C. HOLLINS.

Manufacture of [wax-like] chlorinated compounds of naphthalene. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,774, 21.12.28).—Molten naphthalene is treated with chlorine below 130° in presence of antimony chloride, phosphorus chlorides, sulphur, or iodine, to give wax-like products. C. HOLLINS.

Manufacture of halogenoarylthioglycollic acids. I. G. FARBENIND. A.-G. (B.P. 299,327, 22.10.28. Ger., 22.10.27).—A suitable arylamine is thiocyanated in the *p*-position by the method of B.P. 257,619 (B., 1928, 516), hydrolysed to the *p*-aminothiol, which is condensed with chloroacetic acid, the amino-group being finally exchanged for halogen by Sandmeyer's reaction. The following thioglycollic acids are described: 5-amino-*m*-tolyl- (m.p. 172°), 5-chloro-*m*-tolyl- (m.p. 127–129°), 4-chloro-6-amino-*m*-tolyl- (m.p. 120°), 4:6-dichloro-*m*-tolyl- (m.p. 112°), 2-amino-*p*-5-xylyl- (m.p. 185°), and 2-chloro-*p*-5-xylyl- (m.p. 96°). C. HOLLINS.

Manufacture of bromine-substituted arylthioglycollic acids. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 324,484, 20.12.28).—*o*-Cyanoarylthioglycollic acids or the corresponding *o*-amides are treated with bromine in a solvent (concentrated sulphuric acid) at 20–25°. The preparation of 5-chloro-6-bromo-2-carbamyl-*m*-tolylthioglycollic acid, m.p. 195–196°, the corresponding 2-cyano-compound, m.p. 197–200°, and bromo-4-cyano-*m*-methoxyphenylthioglycollic acid, m.p. 186–187°, is described. C. HOLLINS.

Manufacture of *o*-arylcarboxyamidothioglycollic [o-amidocarbonylarylthioglycollic] acids. I. G. FARBENIND. A.-G. (B.P. 310,757, 29.4.29. Ger., 28.4.28).—*o*-Cyanoarylthioglycollic acids are hydrolysed to the carboxyamides by means of aromatic sulphonic acids, e.g., sulphonated phenol or naphthalene. C. HOLLINS.

Manufacture of condensation products from 2-methylene-1:3:3-trimethylindolines. Soc. CHEM. IND. IN BASLE (B.P. 312,174, 21.5.29. Switz., 19.5.28).—2 Mols. of 2-methylene-1:3:3-trimethylindoline are condensed with formaldehyde in aqueous alcohol at 30–35°. The white, crystalline product,

m.p. 124°, becomes coloured on exposure to air, forming a red basic dye. C. HOLLINS.

Production of chloro-derivatives of *N*-dihydro-1:2:2':1'-anthraquinoneazine. J. H. SACKS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,739,736, 17.12.29. Appl., 29.7.25).—Indanthrone is suspended in 52% sulphuric acid (12 pts.) containing sodium nitrate (1 pt.), and sodium chloride (2 pts.) is added at 45° during 3 hrs.; the product contains 5–6% Cl. With more concentrated sulphuric acid (78%) and more salt (3 pts.) 9–10% Cl may be introduced. C. HOLLINS.

Manufacture of *o*-anisidine and *o*-aminophenol ethers. J. TCHERNIAC (U.S.P. 1,752,121, 25.3.30. Appl., 29.9.27. U.K., 2.10.26).—See B.P. 282,907; B., 1928, 152.

Production of acylresorcinols. A. R. L. DOHME. Assr. to SHARP & DOHME, INC. (U.S.P. 1,750,299, 11.3.30. Appl., 16.4.25).—See B.P. 250,893; B., 1927, 59.

Purification of quinoline and quinaldine. M. WYLER, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,750,082, 11.3.30. Appl., 5.5.27. U.K., 6.9.26).—See B.P. 276,156; B., 1927, 809.

Catalytically reducing anthraquinone compounds and hydrogenated products derived therefrom. J. VON BRAUN and O. BAYER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,751,670, 25.3.30. Appl., 27.2.26. Ger., 3.3.25).—See B.P. 248,759; B., 1927, 597.

Preparation of [aromatic] diazosulphamic acids. K. and W. SCHIRMACHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,750,057, 11.3.30. Appl., 17.2.27. Ger., 22.2.26).—See B.P. 266,388; B., 1928, 327.

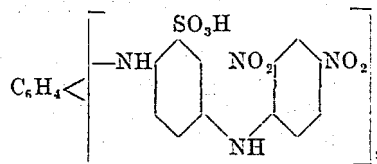
Manufacture of dinaphthylene dioxide quinone. L. ZEH, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,014, 4.3.30. Appl., 4.10.28. Ger., 13.10.27).—See B.P. 298,640; B., 1930, 362.

Liquid hydrocarbons (F.P. 631,927).—See II. **Azo intermediates** (B.P. 299,331). **Benzoylated amino-anthraquinones** (B.P. 324,311).—See IV.

IV.—DYESTUFFS.

PATENTS.

Manufacture of brown wool dyes. I. G. FARBENIND. A.-G., Assees. of H. POLIKIER and A. HASSELBACH (G.P. 455,033, 3.6.26).—A *m*- or *p*-phenylenediamine is condensed with 2 mols. of 1-chloro-4-nitrobenzene-2-sulphonic acid, the nitro-groups are reduced, and the product is finally condensed with 2 mols. of dinitrated chloro- or *p*-dichloro-benzene. From *p*-phenylenediamine, 1-chloro-4-nitrobenzene-2-sulphonic acid (2 mols.), and 1-chloro-2:4-dinitrobenzene (2 mols.) a yellow-brown wool dye (annexed formula) is obtained.



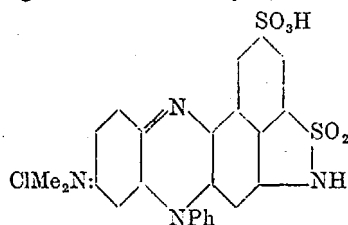
m-Phenylenediamine, with 2:6-dinitro-1:4-dichlorobenzene as end reactant, gives a red-brown.

C. HOLLINS.

Manufacture of azo dyes and intermediate products containing the thiazole ring, with the aid of cyanuric halides. SOC. CHEM. IND. IN BASLE (B.P. 299,331, 22.10.28. Switz., 22.10.27).—The cyanuric chloride method (cf. B.P. 221,843 and 209,723; B., 1924, 937; 1925, 437) is employed for the introduction of a thiazole residue into azo dyes or into intermediates capable of conversion into azo dyes. In the 124 examples the thiazole derivative used is dehydrothio-*p*-toluidine (D.H.T.), its sulphonic acid (D.H.T.S.), primulinesulphonic acid or dehydrothio-*m*-4-xyldine-sulphonic acid. The following are typical: Cyanuric chloride with 2 mols. of *o*-*p*-toluenesulphonyl-H-acid \rightarrow cresidine and 1 mol. of D.H.T.S. (yellowish-red on cotton); cyanuric chloride with 1 mol. of H-acid and 1 mol. of D.H.T.S., finally coupled with diazotised anthranilic acid (pink; reddish-violet on coppering or when pre-coppered); cyanuric chloride with 1 mol. of *p*-phenylenediaminesulphonic acid and 1 mol. of D.H.T.S., diazotised and coupled with acetoacetic anilide (greenish-yellow); *p*-nitroaniline-*o*-sulphonic acid \rightarrow cresidine \rightarrow compound from cyanuric chloride with 1 mol. of H-acid and 1 mol. of D.H.T. (greenish-blue); anthranilic acid \rightarrow compound from cyanuric chloride with J-acid, D.H.T.S., and aniline (coppered, orange-brown); cyanuric chloride with 1 mol. of 4-chloro-*m*-phenylenediamine and 1 mol. of D.H.T.S., diazotised and coupled with compound from cyanuric chloride with 1 mol. of J-acid and 1 mol. of *p*-phenylenediamine-*o*-sulphonic acid, developed on the fibre with β -naphthol (red).

C. HOLLINS.

Dyes of the phenonaphthazine series obtained from 3-arylamino-1:8-naphthasultam compounds. W. NEELMEIER and T. NOCKEN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,723,199, 6.8.29. Appl., 21.7.26. Ger., 28.7.25).—A 3-arylamino-1:8-naphthasultam, especially when carrying a 6-sulphonic group, is condensed with a *p*-nitrosodialkylaniline or oxidised with a *p*-diamine. 3-Anilino-1:8-naphthasultam-6-sulphonic acid with nitrosodimethylaniline hydrochloride gives a red-violet dye (annexed formula);



the *p*-tolyl compound is a violet dye. By the use of *p*-phenylenediaminesulphonic acid a red results; the oxidation of 3-anilino-*N*-methyl-1:8-naphthasultam with *p*-aminodimethylaniline-3-sulphonic acid for a violet is also described.

C. HOLLINS.

Production of dyes and dye intermediates [benzoylation of aminoanthraquinones]. J. F. DUNWORTH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 324,311, 16.7.28).—An aminoanthraquinone is treated with large excess of benzoyl chloride (or a substituted benzoyl chloride) in chloro- or nitro-benzene at about 100°; the product may be filtered off cold, and the excess benzoyl chloride recovered or the filtrate used

for further benzoylations. The benzoylation of 1-amino-, 5-chloro-1-amino-, and 1:5-diamino-anthraquinones is described.

C. HOLLINS.

Manufacture of substitution products of dibenzopyrenequinones. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,964, 8.10.28).—2:3:7:8-Dibenzpyrene-1:6-quinone (from 1:5-dibenzoylnaphthalene) is mononitrated, e.g., with 96% nitric acid in nitrobenzene at 80–85°, and the nitro-group in the product is replaced by chlorine or bromine by boiling with benzoyl chloride or bromide; or the mononitro-compound is treated with chlorine in trichlorobenzene at 175° to give dichlorodibenzpyrenequinone. The amino-group in the reduced mononitroquinone may be replaced by iodine, or by the cyano- or thiocyanogroup. All these products are yellow vat dyes. Methylation of the hydroxydibenzpyrenequinone obtained from the aminoquinone gives an orange vat dye.

C. HOLLINS.

Manufacture of acid dyes of the triphenylmethane series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,966, 10.10.28).—A non-sulphonated arylamine is condensed with a benzaldehyde carrying an *o*-sulphonic group (cf. B.P. 21,365 of 1900; B., 1901, 1205). Superior light-fastness is claimed. Examples are: benzaldehyde-2:4-disulphonic acid with *m*-2-xyldine (bluish-violet); benzaldehyde-*o*-sulphonic acid with *o*-toluidine (violet).

C. HOLLINS.

[Manufacture of] sulphur dye pastes. E. VOETTER, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,963, 11.3.30. Appl., 24.2.28. Ger., 16.6.25).—See B.P. 309,061; B., 1929, 551.

Dyes which are alanines of the anthraquinone series and their derivatives. W. DUISBERG, W. HENTRICH, and L. ZEH, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,242, 4.3.30. Appl., 16.1.26. Ger., 31.1.25).—See B.P. 246,840; B., 1927, 326.

Azo dyes derived from aminoacylamino-salicyldiphenylsulphones. W. NEELMEIER and W. HENTRICH, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,836, 11.3.30. Appl., 5.11.26. Ger., 13.1.25).—See F.P. 609,136; B., 1927, 646.

Products from 2-methylene-1:3:3-trimethylindoline (B.P. 312,174). *N*-Dihydroanthraquinone-azine derivatives (U.S.P. 1,739,736).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Esterified cotton. A. E. WOODHEAD (J. Soc. Dyers and Col., 1930, 46, 69–74).—The preparation and physical and chemical properties of immunised cotton, of which about half the cellulose is in the form of the toluene-*p*-sulphonyl ester, are described. The fibre is found to consist of an inner core of unaltered cellulose surrounded by a tube of the above ester, which, unlike most cellulosic esters, is relatively stable to alkali. Immunised cotton is now being largely replaced by "passive" cotton, which consists of the mono- and diacetates, prepared by the action of acetic acid, acetic anhydride, and an acid catalyst on cellulose. Passive cotton is softer and more flexible than immunised cotton

and possesses slightly greater resistance to direct dyes, but slightly less to alkaline agents. Esterified cotton is, broadly speaking, undyed by direct cotton colours. A good reserve is also obtained with suitable sulphur colours dyed below 40°, and with some vat colours dyed at 30–35°. The dyeing properties of esterified cotton agree with those of cellulose acetate silk, the most important colours for the purpose being the S.R.A. Duranol, Setacyl, Cellit, Cibacet, and other ranges. Immunised and passive cotton are not injuriously affected by boiling water. The esterification of already dyed cotton is also discussed. Selected vat and direct dyes are suitable, the latter becoming faster to light, washing, and bleaching. The application of esterified cotton in textile fabrics is discussed. The preparation and properties of amidated cotton, which, however, is not yet a commercial commodity, are described. Bright shades having good fastness to washing are obtained on amidated cotton, using acid colours in a neutral or acid bath.

N. CHAPPELL.

Isoelectric point of silk fibroin. T. G. HAWLEY, JUN., and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 297–299).—Degummed dried silk can be so finely ground by prolonged treatment in a ball mill that 80% is retained in clear aqueous solution after treating with water and passing through a Berkefeld candle. The isoelectric range determined by cataphoresis and by flocculation was found to be between p_H 1.4 and 2.8, with a probable isoelectric point at about p_H 2.1.

S. I. LEVY.

Swelling and dissolution processes of cellulose and its derivatives. K. HESS (Papier-Fabr., 1930, 28, 169–177).—Investigations have been made of the phenomena attendant on the swelling and dissolution of cellulose in the system cellulose-cuprammonium-sodium hydroxide, and of cellulose esters and ethers in organic liquids such as cyclohexanol, pyridine, chloroform, etc. under various conditions. Absorption of copper and sodium hydroxide by the cellulose, the degree of swelling, and amount of cellulose dissolved under different conditions of copper and sodium hydroxide concentrations of the liquid phase have been measured, and comparisons are made between the Röntgen diagrams obtained under the different experimental conditions. With increasing copper concentration of the liquid, the absorption both of copper and sodium hydroxide increases rapidly up to a point and then respectively decreases and increases very slowly. X-Ray analysis shows one new diagram at the turning point of the copper-absorption curve and a second for still higher concentrations. It is concluded that these diagrams indicate the presence of new copper-alkali-cellulose compounds, each of which predominates according to the copper concentration of the liquid. The degree of swelling and the amount of cellulose dissolved increase with the copper absorption, whilst the amount of dissolved cellulose first increases and then decreases rapidly with increasing sodium hydroxide concentration of the liquid. The reversibility of the absorption and Röntgen diagram effects and the characteristics of the diagrams obtained from cellulose derivatives swollen in organic solvents are discussed. Cellulose acetate gives different diagrams at different temperatures of treatment, and examination of the

swelling effects by means of these diagrams is very complicated, especially as mixed diagrams may be obtained.

B. P. RIDGE.

Viscose. XXXIII. Spinning experiments with viscose prepared from unaged alkali-cellulose. G. KITA, T. NAKASHIMA, S. OOHORA, and J. MURAKAMI (J. Cellulose Inst., Tokyo, 1930, 6, 36–38).—In order to observe the importance of ageing alkali-cellulose from the point of view of producing stronger threads, unaged alkali-cellulose was steeped for various times from 30 min. to 3 weeks at temperatures of 8–10° and 25–28°, and threads were spun from the viscoses subsequently prepared. In no case were specially strong threads obtained. It is concluded that a special reaction occurs in ageing, which is not occasioned by longer steeping, but which is of advantage in the manufacture of artificial silk. Probably oxidation of the alkali-cellulose is to some extent advantageous, but this should be uniform throughout the mass and not limited to one part only. The viscosity in cuprammonium of the threads obtained was determined and threads from the longer-steeped alkali-cellulose were found to give the most viscous solutions. This may depend on the fact that the dissolution of hemicellulose by the sodium hydroxide is of greater importance than the action of the alkali on the cellulose itself.

B. P. RIDGE.

Beating of [paper] pulp. VI. Wet and dried pulps. M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 31–35; cf. B., 1930, 235).—The rate of increase of the degree of beating, and the properties of finished papers obtained from them, have been investigated for wet, air-dried, and hot-dried (100–110°) sulphite pulps. For the same time of beating the degree of wetness is highest for wet pulp, whilst air-dried pulp is next in order. For both the same degree of wetness and time of beating, wet pulp gives papers of the highest bursting and tensile strengths, elongation, and relative transparency. Strength differences between the wet and the dried pulps are a maximum for sheets formed without beating. The high strength of the wet-pulp paper formed without beating is ascribed to adhesive forces due to the colloidal state of the surface of the wet-pulp fibres. Wet pulp suffers an irreversible colloidal change of fibre surface on drying.

B. P. RIDGE.

Chemistry and papermaking. A. S. KLEIN (Papier-Fabr., 1930, 28, 193–200, 213–219).—A summary and bibliography of current theories relating to the constitution, swelling, and “hydration” of cellulose, the strength of paper, sheet-formation, and sizing.

T. T. PORTS.

[Electrical] strength of paper for cables. A. GYEMANT (Wiss. Veröff. Siemens Konz., 1930, 8, [3], 191–202).—Below 75% relative humidity, unimpregnated cable-paper has a resistance to electrical rupture which is independent of the humidity of the air. Two glazed papers which showed a 16:1 difference in porosity with regard to air had electrical strengths in the ratio 1:1.7. The resistance to rupture is some 10–15% greater for a direct potential than for an alternating potential (maximum value). In the case of multiple layers of paper the resistance to rupture decreases on repeating the test, and remains at this value on

further repetitions. The theory of these results is discussed.

W. E. DOWNEY.

Consistency in the application of coatings to paper. R. M. COBB and D. V. LOWE (*J. Rheology*, 1930, 1, 158–166).—By plotting apparent viscosity of colour mixtures against percentage vehicle, paper-coating pulps may be divided into three classes, which correspond to divisions based on mill experience. Results obtained with the Gardner mobilometer and the de Noüy tensiometer do not indicate the superior ability of soft-sized mixtures to stay in place and give a perfect coating. Apparent yield value, as determined by the mobilometer intercept, is without significance in this case. The tensiometer indicates that the troublesome pulps affect the surface tension of the vehicle adversely. Considerable seepage of casein into the paper surface occurs, resulting in a separation of vehicle and pigment; this indicates that the mobility of the layer of coating mixture in immediate contact with the paper surface cannot be the same as that of the coating mixture as measured by ordinary consistency tests before it is applied to paper. The difference in behaviour of soft- and hard-sized coatings can only be explained by consideration of (a) the internal structure of the suspension as indicated by the apparent viscosity, and (b) the change in composition occurring at the liquid-solid boundary during the application of the coating to paper.

E. S. HEDGES.

Capillary siphons [for paper-ruling]. E. C. BINGHAM and D. FIGLIOLI (*J. Rheology*, 1930, 1, 167–172).—A description is given of capillary siphons, which are used for the continuous ruling of ink-lines on paper, the ink being carried from a saturated flannel by means of a woollen thread. Experiments have shown that a flannel will hold 5.5 times its own weight of ink at saturation, and the ink ceases to flow in drops from the thread when the flannel becomes 90% saturated. When the flannel becomes less than 55% saturated, it is no longer possible to withdraw ink from the thread by means of dry blotting paper. The functioning of the machine depends on a high percentage of saturation in the flannels. Experiments are also described on the effects of varying the height of the siphon, the number of threads, and the structure of the threads.

E. S. HEDGES.

PATENTS.

Manufacture of preparations for oiling fibrous materials. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 324,967, 27.10.28).—The preparation comprises a mixture of unsaturated acids obtained by eliminating water from the oxidation products of organic compounds of high mol. wt., e.g., paraffin hydrocarbons or waxes, or from the acids separated therefrom, or from mixtures of such acids with their esters, anhydrides, and lactones. These unsaturated acids have a lower viscosity than olein, show no tendency to resinify, remain fluid above 0°, and are not liable to spontaneous ignition when finely distributed on fibres. They readily emulsify with small quantities of alkalis and dissolve when completely neutralised. Thus washed raw wool may be treated with an emulsified mixture of unsaturated acids obtained by distilling at 100–300° the hydroxy-

fatty acids resulting from the oxidation of paraffin wax (cf. B.P. 322,427; B., 1930, 315). D. J. NORMAN.

Manufacture of substitutes for catgut. P. BOST (B.P. 310,884, 1.5.29. Ger., 2.5.28).—An unsterilised catgut substitute is produced by spinning animal fibres obtained from muscle flesh which has been leached, freed from grease, and hardened.

F. R. ENNOS.

Production of artificial threads by the stretch-spinning process. COURTAULDS, LTD., W. F. HOLLELY, and G. D. BOND (B.P. 325,714, 9.3.29).—The lower end of the spinning funnel used in the spinning of cuprammonium artificial silk is provided with a detachable tip having a longitudinal slot so that it can be removed, together with any deposit of copper hydroxide formed thereon, without interruption of the production of the thread.

F. R. ENNOS.

Production of artificial filaments, threads, bands, sheets, films, etc. from aqueous cellulose solutions. H. KINDERMANN (B.P. 324,963, 4.12.28).—Cellulose solutions with a low alkali content are spun into a coagulating bath containing 35–40% of zinc chloride at 50–60°, the product being subsequently hardened in water or dilute acid. [Stat. ref.]

F. R. ENNOS.

Dry-spinning of solutions of cellulose derivatives. BRIT. CELANESE, LTD., J. F. BRIGGS, and R. P. ROBERTS (B.P. 325,233, 13.8.28).—The uniformity and character of artificial filaments dry-spun in countercurrent to an evaporative medium are improved if additional evaporative medium is introduced into the current in the region of the spinning jet, preferably in the plane of the jet face or on the side remote from the filaments. Suitable devices for this purpose are described.

D. J. NORMAN.

Manufacture of artificial silk. M. LEON, and HARBENS (VISCOSE SILK MANUFACTURING CO., LTD. (B.P. 325,605 and 325,617, [A] 28.11.28, [B] 6.12.28).—A sizing or adhesive material is added (A) to the viscose prior to spinning, (B) to the coagulating bath, and the spun threads are washed with cold water without removing the size and dried in individual lengths (i.e., not in bobbins or packages), whereby the filaments are bound together while leaving the threads supple for subsequent treatment.

F. R. ENNOS.

Production of artificial silk and the like. B. BORZYKOWSKI (B.P. 311,391 and 325,246, 9.8.28. Ger., 11.5.28).—(A) After being washed and subjected to any desired after-treatment such as desulphurising, bleaching, dyeing, or softening while on the spinning bobbin or in the spun cake, the artificial silk is brought directly into the wound form necessary for textile manufacture without intermediate re-winding. (B) For carrying out the process described in (A) the spun cake is held in position, by an inner and an outer elastic, perforated sleeve or ring, inside a double-walled spinning pot having a space between its outer wall and its inner perforated wall for passage of the treating liquids.

F. R. ENNOS.

Cellulose ether film and composition for making the same. S. J. CARROLL, Assr. to EASTMAN KODAK CO. (U.S.P. 1,716,418–20, 11.6.29. Appl., 9.8.26).—Water-insoluble cellulose ethyl ether is compounded with a

volatile thinner (*e.g.*, methyl acetate and 10% of methyl alcohol) and (A) tribromophenol, (B) bromocamphor, or (C) bromobenzene or a homologue, with enough alkali to give $p_H > 7$. Heat-resistant films are obtained.

C. HOLLINS.

Manufacture of cellulose ethers. I. G. FARBEN-IND. A.-G. (B.P. 305,946, 11.2.29. Ger., 10.2.28).—Cellulose ethers containing $1\frac{1}{2}$ — $2\frac{1}{2}$ mols. of alkyl per mol. of cellulose ($C_6H_{10}O_5$) are produced by treating alkali-cellulose with a mixture of alkyl and aralkyl halides in the proportion of 10—30 mols. of the former and 2 mols. of the latter per mol. of cellulose, the temperature and pressure being such that the formation of free acid from the excess of alkylating agent is avoided.

F. R. ENNOS.

Manufacture and treatment of cellulose esters.

H. DREYFUS (B.P. 325,224, 13.8.28).—The ripening of cellulose acetate or other cellulose ester is carried out in an organic medium in which the primary cellulose acetate is insoluble and in which the ripening agent, *e.g.*, water, is soluble in sufficient quantity for ripening to take place satisfactorily. A suitable medium is ether, and may, if desired, be used as a diluent in the acetylation process itself. Thus cotton cellulose is soaked in glacial acetic acid and sulphuric acid for 3—12 hrs. at the ordinary temperature and is then introduced into an ether-acetic anhydride mixture. Acetylation is conducted under reflux at 30—35° until the desired degree of acetylation is attained. Water is then added and the ripening carried out at 30—35°.

D. J. NORMAN.

Manufacture of cellulose esters. H. DREYFUS (B.P. 325,231, 13.8.28).—After pretreatment with an organic acid (formic or acetic) the cellulosic material is esterified with an acid anhydride and a catalyst while in suspension in an organic medium containing a non-solvent for the cellulose ester in which water is soluble to an appreciable extent, *e.g.*, ether mixed, if desired, with benzene (*cf.* B.P. 325,224; preceding).

F. R. ENNOS.

Manufacture of highly viscous acetylcellulose soluble in acetone. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H., and W. GRUBER (B.P. 325,209, 11.10.28).—Triacetylcellulose may be rendered soluble in acetone by treating it below 30° with an acid salt (containing water of crystallisation) of a heavy metal belonging to group I or II or with a mixture of such salts with each other or with other acid or neutral salts. Thus triacetylcellulose is dissolved in glacial acetic acid and treated with $ZnCl_2 \cdot 4H_2O$. After about 3—8 hrs. at 20° the product is completely soluble in acetone. When zinc chloride is used as the acetylation catalyst the above compound may be formed in the reaction mixture itself. Other suitable compounds include $CaCl_2 \cdot 6H_2O$ and $3HgCl_2 \cdot 4HCl \cdot 14H_2O$.

D. J. NORMAN.

Apparatus for the rapid and homogeneous transformation of alkali-cellulose into viscose. L. C. P. JARDIN (B.P. 310,495, 12.10.28. Fr., 28.4.28).—The alkali-cellulose is crushed, rolled, and triturated with carbon disulphide in a rotary drum of polygonal cross-section by the violent impact of leaden balls on the sides of the drum.

F. R. ENNOS.

Maintaining the concentration of magnesium sulphate spinning baths for viscose. BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assees. of N.V. NEDERL. KUNSTZIJDEFABR. (B.P. 325,763, 28.5.29. Holl., 9.4.29).—Equimolecular proportions of concentrated sulphuric acid and magnesium carbonate are added to the spinning bath.

F. R. ENNOS.

Manufacture of chemical products from wood.

A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 325,512, 19.11.28).—After being previously washed or deresinified, the wood is heated with an alkaline reagent, together with a metallic oxide (*e.g.*, of zinc, copper, or iron) if desired, and an alkylating or aralkylating agent at ordinary or increased pressure or in the superheated vapour of the latter reagent, with or without the addition of a diluent (benzene), a solvent or swelling agent (benzene), and a catalyst (copper powder). [Stat. ref.]

F. R. ENNOS.

Manufacture of pressed or moulded articles.

H. P. BAYON. FROM MAGNASCO ROGGERO & Co. (B.P. 325,281, 16.11.28).—In the manufacture of moulded or laminated materials by impregnating a cellulose base with synthetic resins, a more complete impregnation is obtained if the cellulosic material be pretreated with a dilute (10%) solution of an oxidising acid, *e.g.*, nitric acid either alone or in admixture with sulphuric acid. The material is then washed and dried.

D. J. NORMAN.

Absorbent paper. E. I. DU PONT DE NEMOURS & Co. (B.P. 313,085, 1.6.29. U.S., 6.6.28).—A permanent curl may be imparted to the fibres of cellulose pulp, *e.g.*, mechanical wood pulp, sulphite pulp, pulps made from linen, jute, etc., by treating the pulp with reagents which exert a mercerising effect on cellulose, *e.g.*, 18% caustic soda solution at 20—60°. The treated and washed fibres are then converted into paper optionally after admixture with untreated stock.

D. J. NORMAN.

Manufacture of waterproof paper or pasteboard.

I. G. FARBENIND. A.-G. (B.P. 301,807, 5.12.28. Ger., 5.12.27. Addn. to B.P. 289,063; B., 1930, 185).—From 0.5 to 10% or even 50% of the higher fatty acid esters of cellulose described in the prior patent is incorporated with the pulp in the beater and the finished paper or board is calendered at 110—120°.

D. J. NORMAN.

Manufacture of cigarette paper. B. SEFERIADIS

(B.P. 325,125, 27.2.29).—Cigarette paper which on combustion is free from any disagreeable odour is obtained by incorporating with the pulp 3—5% of a gum resin, *e.g.*, gum mastic. The resin may be macerated for 24 hrs. in acetone-alcohol and then (a) introduced into the beater at the end of the beating operation, or (b) dissolved in acetone and mixed with part of the calcium carbonate to be used as filler.

D. J. NORMAN.

Sizing of paper. L. F. C. GIRARDET (F.P. 635,419, 2.6.27).—The size is prepared by treating colophony with cold sodium hydroxide solution and adding aluminium sulphate just prior to mixing the size with the paper pulp.

A. R. POWELL.

Treatment of crude fibres and textile fabrics. R. H. POTT, ASSR. to CHEM. FABR. POTT & Co. (U.S.P.

1,750,430, 11.3.30. Appl., 22.1.23. Ger., 15.6.21).—See B.P. 248,814; B., 1926, 403.

Isolation of benzyl cellulose. E. DÖRR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,751,685, 25.3.30. Appl., 13.8.27. Ger., 17.8.25).—See B.P. 265,491; B., 1927, 296.

Treating residual liquors accruing from the treatment of cellulose materials. P. A. BARBOU, Assr. to SOC. BARBOU & CIE. (U.S.P. 1,749,557, 4.3.30. Appl., 5.4.27. Fr., 21.4.26).—See B.P. 269,909; B., 1928, 814.

Production of stencil sheets for use in duplicating. A. DE WAELE, Assr. to D. GESTETNER, LTD. (U.S.P. 1,749,241, 4.3.30. Appl., 3.5.28. U.K., 22.7.27).—See B.P. 298,705; B., 1929, 14.

Feed apparatus for machines for depulping fibre-containing leaves. I. G. FARBENIND. A.-G. (B.P. 319,653, 11.6.29. Ger., 25.9.28).

Twisting of rayon threads. H. WADE. From DU PONT RAYON CO. (B.P. 326,130, 3.10.28).

Continuous machines for making paper and other sheet material from pulp. SOCIETA INVENZIONI BREVETTI ANONIMA (B.P. 315,690, 18.4.29. It., 16.7.28).

Fourdrinier papermaking machines. S. MILNE (B.P. 326,330, 27.2.29).

Evaporation of sulphite lye (B.P. 300,590).—See I. **Caustic liquor** (U.S.P. 1,742,220).—See VII. **Artificial timber** (U.S.P. 1,742,410).—See IX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fast dyeing of cotton and wool. J. V. CROSSLEY (J. Soc. Dyers and Col., 1930, 46, 74—76).—Reference is made to recent additions to the range of anthraquinone vat dyes for fast dyeings on cotton piece and yarn. The Indigosol and Soledon dyes, azoic colours of the Naphthol AS range, and the sulphur colours, Indocarbon CL and CLG, are also mentioned. The dyeing of wool with vat dyes and vat "grains" (ready reduced colours) is described. The superior handle, strength, elasticity, and felting properties compared with those of wool dyed with alizarin dyes and after-chromed at the boil are discussed. N. CHAPPELL.

Formation of crystalline metallic antimony in antimony fluoride bath used for fixing basic dyes on printed fabric. A. SCHERRER (Bull. Soc. Ind. Mulhouse, 1929, 95, 746—747).—In the production of an "imitation Jacquard" fabric obtained by padding fabric with tannic acid, printing with a resist paste containing caustic soda, steaming, passing through a fixing bath of antimony fluoride, and then dyeing with a basic dye, a deposit of about 175 g. of crystalline metallic antimony was observed in the fixing bath. The formation of this is attributed to the reducing action of caustic soda and tannic acid (introduced by the fabric into the fixing bath) on antimony oxide present in the fixing bath, as a result of insufficient acid being added to neutralise the alkalinity of the fabric. A. J. HALL.

Printing with basic dyes without after-treating with tartar emetic. N. LYHRA (Sealed Note No.

1949, 15.11.09. Bull. Soc. Ind. Mulhouse, 1929, 95, 754—755). Report by P. SEYDER (*Ibid.*, 755—756).—A printing paste is used containing the basic dye, tannic acid, and water-insoluble antimony borate; the dye-antimony tannate lake only forms during the subsequent steaming. *E.g.*, fabric is printed with a paste consisting of 20 g. of basic dye, 60 g. of acetic acid (*d* 1.045), 690 g. of a 50% solution of gum Senegal, 10 g. of ammonium chloride (or 20 g. of potassium thiocyanate), 120 g. of a 50% solution of tannic acid (acidified with acetic acid), and 100 g. of antimony paste A, then steamed for 45 min., soaped for 8—10 min. at 40—45°, rinsed, and dried. Paste A is prepared by mixing 200 g. of antimony fluoride, 20 g. of caustic soda (*d* 1.24), 120 g. of borax, and 500 g. of water, filtering, pressing the resulting precipitate of antimony borate until it contains 50% of water, and then mixing with its own weight of glycerin. The resulting printed colour effects have the usual fastness to light, soap, and chlorine. SEYDER confirms the efficiency of the process and finds that purer blue shades are obtained with Methylene Blue than when the usual printing process is used. The use of ammonium chloride appears to be unnecessary. A. J. HALL.

[Printing] reserves under Aniline Black on wool. C. and P. SCHWARTZ (Sealed Note No. 1700, 31.1.07. Bull. Soc. Ind. Mulhouse, 1929, 95, 747—749). Report by A. LAU (*Ibid.*, 750—752).—White and coloured reserves are obtained on previously scoured and chlored wool fabrics by first padding with a solution containing 0.25 g. of ammonium vanadate per litre, drying in the hot flue, printing with a reserve paste containing potassium sulphite (the reserve agent), with or without a dye not affected by the sulphite (*e.g.*, Eosine, Formyl Violet, Auramine, and Guinea Green), then overprinted with an Aniline Black liquor containing a mixture of aniline and *p*-aminodiphenylamine (either of these amines alone is unsuitable), steamed for 5—6 min. at 102°, then washed and dried. Coloured discharges on a coloured ground may be obtained simultaneously by adding sodium hyposulphite to the reserve paste. LAU reports favourably on the process, although he finds that the resulting black has a brownish tone and that it yields a good black on wool chlored with but 12% of active chlorine, whereas by means of the usual prussiate-Aniline Black process an equal shade could only be obtained after chloring with 25% of chlorine. A. J. HALL.

Printing with indigoid dyes. A. DONDAIN and A. STIEGLER (Sealed Note No. 2082, 3.4.11. Bull. Soc. Ind. Mulhouse, 1929, 95, 757—758). Report by L. DISERENS (*Ibid.*, 758—760).—A more complete and regular fixation of indigo and Hydron Blue R and G in the usual printing process is obtained by replacing hyposulphite-formaldehyde by hyposulphite-acetaldehyde. DISERENS reports that hyposulphite-acetaldehyde is less stable than the corresponding formaldehyde compound, and that under unfavourable conditions of steaming the first-named substance may thus give superior results; otherwise the use of hyposulphite-acetaldehyde has little practical interest. A. J. HALL.

Discharges on indigo, thioindigo, and sulphur colours [in printing]. E. LUSTIG and L. PAULUS (Sealed Note No. 1710, 21.2.07. Bull. Soc. Ind. Mulhouse, 1929, 95, 752—753). Report by H. WAGNER (*Ibid.*, 753—754).—In discharging by means of a hyposulphite a purer white is obtained by adding glucose to the discharge paste; the glucose retards re-oxidation of the reduced dye by exposure to air, so that the dye can be more completely removed. *E.g.*, fabric dyed with indigo or other similar dye is printed with a discharge paste consisting of 30 g. of Hydrosulphite C, 160 g. of a 33% solution of gum, and 10 g. of glucose, steamed for 3 min. at 100° in a small Mather-Platt, passed through a bath containing 50 g. of caustic soda (*d* 1.38) per litre, mangled, acidified, and washed. A somewhat inferior white discharge can also be obtained by omitting the Hydrosulphite C from the discharge paste and passing the printed fabric through a boiling bath of caustic soda. Glucose may also be replaced by other reducing substances such as tannic acid. WAGNER reports that the process gives good results with indigo but not with sulphur dyes. The introduction of Leucotrope W for the same purpose, as described in 1909, provides an alternative method. A. J. HALL.

Esterified cotton. WOODHEAD.—See V.

PATENTS.

Dyeing of mixed fabrics containing acetate silk and the manufacture of dye products suitable therefor. I. G. FARBENIND. A.-G. (B.P. 299,787, 30.10.28. Ger., 31.10.27).—Mixed acetate silk fabrics are dyed with a preparation containing a dye for the wool or cotton and a diazotisable amine having affinity for acetate silk, together with a wetting agent of the alkyl-naphthalene-sulphonic acid type. The mixture dissolves in hot water without residue. Examples are: Zambesi black D, α -naphthylamine, and sodium butyl-naphthalene-sulphonate on wool-acetate silk fabric, diazotised and developed with 2:3-hydroxynaphthoic acid for deep black; Diamine black BH, dianisidine, and the wetting agent on cotton-acetate silk, diazotised and developed as before for dark blue. C. HOLLINS.

Production of coloured patterns on textiles, paper, or plastic masses. I. G. FARBENIND. A.-G., Assees. of E. FRANZ (G.P. 455,808, 31.1.26).—The material is treated evenly or in parts with a colour-producing preparation, *e.g.*, one of the Rapid-fast colours and/or the sulphuric ester of a leuco-vat dye together with ammonium vanadate and sodium chlorate, and is then passed over a hot cylinder on which the pattern is marked. The colour is developed only where the damp material comes into contact with the hot roller, the remainder being white after washing.

C. HOLLINS.

Printing and dyeing. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,315, 16.8.28).—Alkali-metal compounds of hydroxylated alkyl- or cycloalkylamines, with or without an organic solvent, are incorporated with vat dyes or sulphide dyes in printing pastes not containing hyposulphite or other strong reducing agent; or are added to such dyes before or during the dyeing operation. Indanthrene blue GC with

1.5 pts. of di(hydroxypropyl)amine and thickener (6.5 pts.) gives clear, bright prints. Indanthrene Yellow G is dyed from a bath containing caustic alkali, dextrose, and "triethanolamine." *cyclo*Hexyl- β -hydroxyethylamine is also mentioned. C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND. A.-G. (B.P. 304,787, 25.1.29. Ger., 26.1.28. Addn. to B.P. 279,864; B., 1929, 15).—The fabric is printed with vat dye and a cellulose ether (*e.g.*, the methyl ether) and is passed through an alkaline hyposulphite bath. The cellulose ether is thus precipitated and the dye does not bleed even if the fabric is not dried before steaming. C. HOLLINS.

Treatment of textiles [Chloroamine-T as assistant]. CHEM. FABR. VON HEYDEN A.-G. (F.P. 610,985, 11.2.26).—Sodium *p*-toluenesulphonchloroamide is used with starch for sizing, with glycerin and tartaric or citric acid for silk finishing, alone for de-sizing, or with alkali for boiling-out. Wool treated with the chloroamide and hydrochloric acid and washed with bisulphite shows increased affinity for dyes. The chloroamide may also be added to wool-scouring baths.

C. HOLLINS.

Protection of wool, fur, hair, feathers, etc. against attack by moth. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 324,962, 5.10.28).—The materials are treated with arylsulphonylamides. Amongst the 50 examples are *p*-chlorophenol-2:6-disulphon-di-*p*-chloroanilide, *NN'*-bis-2:4:5-trichlorobenzenesulphonyl-*m*-phenylenediamine, and 2:4:5-trichlorobenzenesulphon-*p*-chloro-*m*-valeramidoanilide. The necessary sulphonyl chlorides are obtained by the chlorosulphonic acid method. C. HOLLINS.

Bleaching process. G. ADOLPH and A. PIETZSCH (U.S.P. 1,750,657, 18.3.30. Appl., 11.3.27. Ger., 29.3.26).—See B.P. 268,325; B., 1928, 155.

Propellers or screws employed in dyeing machines. E. and L. CONSTANT (Soc. E. & L. CONSTANT) (B.P. 302,186, 10.12.28. Fr., 10.12.27).

Condensation products (B.P. 321,190).—See III. **Artificial silk** (B.P. 311,391 and 325,246).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Improvements in the manufacture of chamber sulphuric acid with gases rich in nitrogen oxides. R. MORITZ (Oesterr. Chem.-Ztg., 1930, 33, 42).—The Gay-Lussac towers are fed with acid of *d* 1.753—1.797. To overcome the trouble incidental to the use of acid of this concentration, a cooling and drying apparatus, utilising the acid from the second Gay-Lussac tower, is inserted between the chambers and the first tower, thus cooling and drying the gases entering this tower. A marked economy is effected. W. J. WRIGHT.

Comparative economies of coke and electric furnaces [for electrothermal production of phosphoric acid]. A. KOCHS (Chem. Met. Eng., 1929, 36, 741—742). B. G. KLUGH (*Ibid.*, 1930, 37, 105—106).—Klugh's figure (*cf.* B., 1930, 55) of 3.085 lb. of carbon per lb. of phosphorus pentoxide as the energy requirement of the coke process is queried. Assuming that coke

contains 90% C, the heat produced by its oxidation to carbon monoxide is likely to be cheaper than the equivalent heat produced electrically. Further, a considerable proportion of the heat of combination of the carbon monoxide leaving the blast furnace is capable of recovery.

KLUGH states that the figure in question is based on the heat of combustion of carbon to carbon monoxide, a thermal efficiency of 62.8%, and a recovery of 80% of pentoxide. The efficiency figure is that of standard blast-furnace practice. The preheating of the blast to 500° by the combustion of carbon monoxide is allowed for, and no other form of heat exchange is likely to be practicable. Further, the power requirements of a blast furnace are considerable. The electric furnace offers greater possibilities of ultimate economies.

C. IRWIN.

Iodometric analysis of a mixture of hyposulphite, sulphite, and thiosulphate. R. WOLLAK (Z. anal. Chem., 1930, 80, 1—4).—The method used involves three iodine titrations. Hyposulphite and thiosulphate are first determined in a portion of the sample by Bacha's method (A., 1922, ii, 311); a second portion is dissolved in an excess of iodine solution containing sodium acetate. The excess of iodine is removed with 0.4*N*-sodium sulphite, and an additional 30 c.c. of this solution are added. The liquid after being neutralised with *N*-sodium hydroxide and kept for 5 min. behaves according to the following reaction: $\text{Na}_2\text{S}_4\text{O}_6 + \text{Na}_2\text{SO}_3 = \text{Na}_2\text{S}_2\text{O}_6 + \text{Na}_2\text{S}_2\text{O}_3$. On adding 5 c.c. of formalin to combine with the excess of sulphite, acidifying the solution with acetic acid, and titrating with iodine, the amount of this required corresponds to half the thiosulphate initially present in the sample. The total iodine required by a third portion of substance is then found, and the amount of sulphite present found by difference.

H. F. HARWOOD.

Manufacture of a new form of chloride of lime. A. CARUGHI and C. PAOLONI (Giorn. Chim. Ind. Appl., 1930, 12, 9—13).—By the action of chlorine on lime suspended in carbon tetrachloride a new form of bleaching powder is obtained with an available chlorine content of 38—40%. By varying the conditions of preparation it may be obtained in the crystalline or amorphous form. The crystalline anhydrous form is the more stable, and can be kept for a long period without decomposing even at 50—80°. It has no smell of chlorine, is easily soluble in water, and is denser and less hygroscopic than ordinary bleaching powder.

O. J. WALKER.

Chlorometric, bromometric, and iodometric determination of available chlorine in hypochlorite solutions and bleaching powder. II. H. DITZ and R. MAY (Z. anal. Chem., 1930, 79, 371—388; cf. A., 1930, 310).—Modifications of Penot's method (titration with arsenite) have been examined and compared with Rupp's iodometric method. The former all give lower results than the latter with the exception of Mohr's modification (addition of excess of arsenite followed by back-titration with iodine solution), the results from which agree satisfactorily with those obtained in the iodometric process. Experiments showed that the presence of calcium hydroxide has no influence

on the reaction between the arsenite and the iodine, as the sodium hydrogen carbonate present in the former solution reacts with the dissolved lime to form insoluble calcium carbonate. The Penot-Mohr method may also be employed to determine the available chlorine in alkali hypochlorite solutions, provided that 10 c.c. of a 10% solution of calcium chloride be added 5 min. before the back-titration with iodine. Kolthoff's method of titration, using the hypochlorite solution in the burette, gives results which are both too high and discordant.

H. F. HARWOOD.

Determination of chlorides in salt brines. S. L. NEAVE (Ind. Eng. Chem. [Anal.], 1930, 2, 28).—In the analysis of brines it is necessary that the chlorine content should be determined as accurately as possible, as owing to the great preponderance of sodium and chlorine ions in these liquids small errors in their determination seriously affect the calculation of the hypothetical combinations of the other ions present. The following method is recommended as affording the requisite accuracy without excessive expenditure of time. A suitable volume of the brine is precipitated with an excess of 0.1*N*-silver nitrate, the silver chloride is filtered off and weighed, and the filtrate titrated with 0.1*N*-potassium thiocyanate. The chlorine present is then taken from the mean of the gravimetric and volumetric results, which are subject to errors of opposite sign. Test analyses showed that the figure so obtained was very close to the true value for the chlorine actually present.

H. F. HARWOOD.

Preparation of potassium dichromate from Solikamsk sylvite. V. S. YATLOV (J. Appl. Chem., Russia, 1929, 2, 561—568).—The material is used for interaction with sodium dichromate. The purity of the product is 98.32%.

CHEMICAL ABSTRACTS.

Incomplete distillation of ammonia in the analysis of ammonium sulphate. A. W. CLARK and H. M. ELLIS (J. Assoc. Off. Agric. Chem., 1930, 13, 147—148).—Kjeldahl ammonia distillation is incomplete unless 350 c.c. out of a total of 400 c.c. are distilled over.

K. V. THIMANN.

Experimental production of ammonium molybdate. A. S. GARNAK (J. Chem. Ind., Russia, 1929, 6, 534—535).—Ferromolybdenum (65 kg.) is heated at 600° in a 10-cm. layer, being stirred every 20—30 min.; after 2.5—3 hrs. the reaction becomes slower and the temperature is finally raised to 750—790°. The mixture then contains 55—65% MoO₃. The roasted material (105 kg.) is mixed with water (120 litres) and 25% alcoholic ammonia solution (45 kg.) in an iron ball-mill; the product is filter-pressed, and the solution, *d* 1.22, is treated with ammonium sulphide (4 litres for 500 litres) to remove iron and heavy metals. The excess of ammonium sulphide is removed with lead nitrate solution. Before crystallisation, nitric acid is removed with sulphur dioxide.

CHEMICAL ABSTRACTS.

Perchloric acid as oxidising agent in the determination of chromium [in chrome alum liquors and crystals]. J. J. LICHTIN (Ind. Eng. Chem. [Anal.], 1930, 2, 126—127).—The chromium salt (0.2—1 g.) is heated in a flask with 5 c.c. each of water and 60%

perchloric acid until oxidation is complete. After cooling, 40–50 c.c. of water are added and the remaining chlorine is expelled by boiling. Ammonia is added and any hydroxides of iron and aluminium are removed by filtration, the chromate in the filtrate being determined iodometrically. The method is rapid and accurate and affords a good separation of chromium from iron and aluminium. H. F. HARWOOD.

Synthetic ammonia from soil. WINOGRADSKY.—See XVI.

PATENTS.

Manufacture of phosphoric acid and hydrogen. J. Y. JOHNSON. From I. G. FARBERIND A.-G. (B.P. 325,533, 16.11.28).—Water or steam is allowed to react with phosphorus at elevated temperatures (650–700°) and the product is oxidised almost wholly to phosphoric acid without addition of oxygen by slightly reducing the temperature (to 600°), *e.g.*, by addition of water or steam, preferably in the presence of catalysts (copper-nickel alloys, carborundum, carbon, etc.), and maintaining this reduced temperature for some time.

S. K. TWEEDY.

Decolorisation of caustic liquors. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,742,220, 7.1.30. Appl., 13.2.28).—The colloidal colour impurities are adsorbed by fibrous α -cellulose, *e.g.*, by mixing the liquor with 1% by wt. of fibre. The adsorptive surface of the fibre may be enlarged by beating. S. K. TWEEDY.

Process and apparatus for operating with ammonia. J. Y. JOHNSON. From I. G. FARBERIND A.-G. (B.P. 325,557, 15.9.28).—Those parts of apparatus coming in contact with ammonia at temperatures exceeding 600° are constructed of, or lined with, silicon or substances containing uncombined silicon. The silicon may be applied as a cement with a binding medium such as water-glass. Silicon alloys, *e.g.*, with copper, may be used provided the amount of the metal constituents capable of decomposing ammonia does not exceed the limits at which their decomposing action would become apparent to an undesirable extent.

S. K. TWEEDY.

Manufacture of alkali formates and ammonia from carbon-containing alkali cyanide. FABR. DE PROD. CHIM. DE THANN ET DE MULHOUSE (F.P. 630,686, 7.6.26).—Cyanide made according to B.P. 225,160 (B., 1928, 98) is hydrolysed at 230–270° and gives a dry powder containing sodium formate (22%), sodium hydroxide (3–4%), lime (55%), and carbon (15–20%). The aqueous extract is made feebly acid with sulphuric or formic acid, and evaporated to give alkali formate.

C. HOLLINS.

Manufacture of alkaline-earth cyanamides. J. GUILLISSEN, and UNION CHIM. BELGE, SOC. ANON. (B.P. 326,117, 5.7.29).—Calcium carbonate is treated with ammonia mixed with carbon dioxide in proportion such that the partial pressure of the latter gas is at least equal to the dissociation pressure of the carbonate at the temperature employed, *e.g.*, working at 900°, a mixture of equal vols. of the gases is used. L. A. COLES.

Production of basic aluminium sulphate. C. C. FEAGLEY, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,742,345, 7.1.30. Appl., 25.6.25).—Heavy hydrated alumina,

substantially free from insoluble matter, is added to sulphuric acid (*e.g.*, 35% solution) in a quantity which exceeds by about 10–20% that theoretically required to form the normal sulphate, and the solution is evaporated. Alumina originally containing iron when so treated will give a commercially iron-free product.

S. K. TWEEDY.

Crystallisation of salts. P. H. MÜLLER (B.P. 325,650, 2.1.29).—Solutions containing, *e.g.*, sodium carbonate or sulphate are concentrated in a series of vacuum vessels operated under successively decreasing pressures and temperatures and provided at the top with coolers and means for withdrawing condensed vapour, the solutions being preheated before entry into the first vessel by passing in the reverse direction through coils situated in the vapour spaces of the vessels. The liquor leaving the last vessel passes into a crystallising vessel working under a still higher vacuum and provided at the lower end with means for passing the magma into a centrifugal separator or filter, and at the upper end with a cooling coil serving as the vaporiser of a refrigerator.

L. A. COLES.

Manufacture of crystalline materials. H. O. DERING (B.P. 325,757, 13.5.29).—Crystals capable of rapid dissolution are obtained by agitating and/or seeding supersaturated solutions prepared by the slow cooling of quiescent, concentrated solutions in vessels having a smooth inner surface and under conditions such that superficial evaporation is prevented.

L. A. COLES.

Production of titanium [di]oxide. F. VON BICHOWSKY, Assr. to TITANIA CORP. (U.S.P. 1,742,674, 7.1.30. Appl., 15.6.27).—Titanium nitride prepared as previously described (*cf.* U.S.P. 1,408,661; B., 1922, 294 A) is treated with dilute nitric acid: $5\text{Ti}_2\text{N}_2 + 3\text{HNO}_3 = 10\text{TiO}_2 + 4\text{H}_2\text{O} + 9\text{N}_2$. Alternatively, the nitride may be heated with concentrated sulphuric acid containing a metal (sodium) nitrate or nitric acid; the cooled solution is then poured into cold water, when all the titanium enters solution as sulphate and is precipitated as pure oxide on boiling. Any iron present must be kept in solution by having hydrochloric acid, chlorine, or sulphuric acid present. The oxide obtained is finely divided and suitable for pigments. A method of directly obtaining a titania-barytes paint base is described.

S. K. TWEEDY.

Preparation of hydrogen and absorption of carbon dioxide from gas mixtures. F. GÜLKER (B.P. 301,499, 21.11.28. Ger., 1.12.27. Addn. to B.P. 275,273; B., 1929, 95).—The absorbent and catalytic material used in a modification of the prior process comprises approximately equimolecular proportions of calcium and magnesium oxides, obtained by calcining dolomite; other catalysts may also be added.

L. A. COLES.

Apparatus for solidifying [molten] sulphur. I. E. HANSON and J. B. GAFFNEY (U.S.P. 1,742,391, 7.1.30. Appl., 11.6.27. Renewed 30.10.29).—For this purpose a large aluminium tray of smooth interior surface is rotatably mounted and has an open side temporarily closed. On removing the closure, the tray automatically tips downwards and the cake, 6 in. thick, slides out.

S. K. TWEEDY.

Purification of impure titanium dioxide. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 309,598, 10.4.29. Ger., 14.4.28).—The dioxide is freed from contaminating chromium oxide by mixing with a small excess of alkali or alkaline earth (magnesia, alkali carbonate, etc.), drying, roasting at incandescent heat, and lixiviating out the soluble chromate with water or dilute acid. Oxidising agents may be added to accelerate the formation of chromate. S. K. TWEEDY.

Manufacture of nitrous oxide from ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,475, 12.11.28 and 25.7.29).—Ammonia mixed preferably with excess of oxygen, or gases containing oxygen, is passed over a catalyst (*e.g.*, platinum, iron oxide containing bismuth oxide or manganese oxide, etc.) at 200–500° at a suitable speed such that the product is substantially free from nitric and higher oxide, but the nitrous oxide formed is not decomposed (*e.g.*, 1000 litres/hr. per litre volume of the catalyst). S. K. TWEEDY.

Recovering nitric acid from weak nitric liquors. H. HIRTZ (U.S.P. 1,752,086, 25.3.30. Appl., 23.2.24. Ger., 15.6.23).—See B.P. 217,546; B., 1925, 98.

Production of hydrocyanic acid. F. LINDER and F. LINK, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,751,933, 25.3.30. Appl., 18.10.27. Ger., 30.10.26).—See B.P. 300,369; B., 1929, 52.

Manufacture of active silica. F. STOEWENER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,751,955, 25.3.30. Appl., 16.7.26. Ger., 23.7.25).—See B.P. 255,864; B., 1928, 230.

Synthetically producing ammonia from its elements. G. F. UHDE (U.S.P. 1,750,551, 11.3.30. Appl., 30.6.27. Ger., 2.7.26).—See B.P. 273,735; B., 1929, 52.

[Manufacture of] salts of alkaline-earth metals. F. LINDER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,751,932, 25.3.30. Appl., 25.4.27. Ger., 8.6.26).—See B.P. 289,196; B., 1928, 447.

Manufacture of anhydrous magnesium chloride. K. STAIB, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,749,854, 11.3.30. Appl., 6.5.26. Ger., 9.5.25).—See B.P. 259,498; B., 1926, 979.

Production of lead [mon]oxide in a state of high dispersion. L. SCHERTEL and W. LÜTY, Assrs. to T. GOLDSCHMIDT A.-G. (U.S.P. 1,749,781, 11.3.30. Appl., 4.2.25. Ger., 8.2.24).—See B.P. 228,900; B., 1925, 847.

Apparatus for production of hydrogen. F. E. LEFEBVRE, Assr. to OXYHYDRIQUE FRANÇ. (U.S.P. 1,752,187, 25.3.30. Appl., 16.7.28. Fr., 18.7.27).—See 294,150; B., 1929, 814.

Washing of granular material (U.S.P. 1,741,063). **Evaporation of sulphite lye** (B.P. 300,590).—See I. **Treatment of minerals** (U.S.P. 1,742,191).—See X. **Products by gaseous discharge** (B.P. 300,282).—See XI. **Fertilisers** (U.S.P. 1,742,448; B.P. 306,103 and 300,965).—See XVI.

VIII.—GLASS; CERAMICS.

Thermal expansion of Jena glass 16 III. W. H. KEESOM and A. BIJL (Proc. K. Akad. Wetensch. Amsterdam, 1929, 32, 1164–1166).—With a slightly modified form of the method used by van Agt and Onnes (*cf.* A., 1926, 231), measurements of the linear thermal expansion were made at 100°, –103°, –183°, and –253°. The value found for the coefficient of cubical expansion between 0° and 100° was 237.5×10^{-7} , in fair agreement with the results of the former workers. The coefficient of cubical expansion when remeasured by the weight-thermometer method gave the value 242.1×10^{-7} , in good agreement with Jansen's result. N. M. BLIGH.

PATENTS.

[Electric] tunnel or enamelling pottery kilns. ELECTRIC FURNACE CO., LTD., and W. J. MILLAR (B.P. 325,794, 21.11.28).—The heating coils, wound in longitudinal grooves in the brickwork lining of the kiln, can be connected independently to the source of current, so that the vertical temperature gradient can be regulated, and are of varying lengths, so that any required distribution of heat can be obtained along the kiln. The brickwork of the roof and side walls is recessed at intervals to prevent draughts when the end doors are opened, and air-inlet dampers and outlets are provided to allow of the escape of oily fumes etc. L. A. COLES.

Removal of impurities [iron compounds] from sand and like materials. CHANCE BROS. & CO., LTD., and J. ENGLISH (B.P. 325,386, 12.2.29).—Ferruginous sand and other silicious materials are rendered suitable for use in the manufacture of glass and of pottery etc., respectively, by successive or simultaneous treatment at 700–1000° in a rotary kiln with reducing gases (*e.g.*, carbon monoxide, sulphur dioxide, or, in the case of successive treatment, hydrogen) and with chlorine, after which the iron chloride formed is removed. L. A. COLES.

Manufacture of decorative glass. H. D. MURRAY and D. A. SPENCER (B.P. 325,924, 24.12.28).—The gelatin coatings on glass sheets for use in the manufacture of composite glass are given a coloured design by the use of diazo compounds. L. A. COLES.

Manufacture of porous [ceramic] objects. A. J. MANDELL (U.S.P. 1,742,515, 7.1.30. Appl., 8.10.24. Renewed 9.4.29).—The clay is mixed with particles of a solid substance that will volatilise below its m.p. and below the fritting point of the clay. Among substances that can be used, naphthalene and anthracene are more satisfactory than ammonium chloride because the former are non-miscible with the water that is also used. By using different mixes for different parts of the same object different degrees of porosity may be obtained; *e.g.*, bricks with a non-conducting porous back and a compact slag-resisting face may be made with only one firing. The volatile substance is removed during the ordinary drying process or a continuation thereof at a slightly increased temperature. B. M. VENABLES.

Preparation of refractory linings or crucibles for coreless electrical induction furnaces. N. R. DAVIS, and ASSOC. ELECTRICAL INDUSTRIES, LTD.

(B.P. 325,661, 8.1.29).—Crucibles made of refractory material capable of being sintered are provided at or within the mouth with a metal ring of thickness such that it is not melted by the eddy currents set up when the inductor is energised; when the initial charge is poured, it melts a portion of the ring near the spout, thus providing a gap for pouring out subsequent charges.

L. A. COLES.

Continuous kiln. C. B. WINZER (U.S.P. 1,749,866, 11.3.30. Appl., 28.3.29. U.K., 8.3.28).—See B.P. 318,650; B., 1929, 919.

Production of [tinted violet] glass. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE ST.-GOBAIN, CHAUNY, & CIREY (U.S.P. 1,749,823, 11.3.30. Appl., 14.6.28. Fr., 2.7.27).—See B.P. 293,310; B., 1928, 711.

Manufacture of ceramic ware. J. W. MELLOR (U.S.P. 1,749,642, 4.3.30. Appl., 2.10.28. U.K., 31.1.28).—See B.P. 302,519; B., 1929, 130.

Manufacture of ceramic materials, building elements, etc. from mud. A. LOESSIN (U.S.P. 1,751,163, 18.3.30. Appl., 20.9.26. Ger., 1.10.25).—See B.P. 259,236; B., 1927, 332.

Composition and manufacture of refractory basic brick and the like. C. S. GARNETT (U.S.P. 1,751,234, 18.3.30. Appl., 6.3.23. U.K., 18.3.22).—See B.P. 197,791; B., 1923, 721 A.

Manufacture of plate and sheet glass. C. HEUZE (B.P. 307,302, 4.3.29. Belg., 3.3.28).

[Glass] furnace (U.S.P. 1,739,973). Porous body for diffusion etc. (U.S.P. 1,742,411).—See I.

IX.—BUILDING MATERIALS.

Silication of limestone for road surfaces. S. MANTEL (Przemysl Chem., 1930, 14, 132—141).—The silication of limestone depends not on chemical combination to yield calcium silicate, but on the separation of silica in the pores of the limestone as a result of hydrolytic reactions. The mechanical resistance of calcium carbonate briquettes is greatly increased by treating with sodium silicate solutions, the best results being obtained with briquettes of a porosity of 12—17%. The chemical composition of the silicate solution used has no apparent influence on the results within the limits taken ($\text{SiO}_2 : \text{Na}_2\text{O} = 2.6—4.0$). The increase in mechanical resistance following on silication is directly proportional to the quantity of silica introduced into the given material; limestones of relatively lower porosity should therefore be treated with more concentrated solutions of sodium silicate.

R. TRUSZKOWSKI.

PATENTS.

Shaft kiln for calcining cement or lime. E. MEIER (G.P. 460,846, 11.5.26. Addn. to G.P. 445,554; B., 1928, 194. Cf. G.P. 449,789; B., 1928, 896).—A chamber with completely free cross-section is interposed before the combustion zone to serve as a preheating and de-acidifying chamber. It is heated only by radiation from the combustion zone. Baffles project from the side walls and from a central wall below the combustion zone in order to retard the free fall of the material.

A. B. MANNING.

Manufacture of waterproof composition for Portland cement. H. HAMAKADO (B.P. 325,991, 14.2.29).—Aluminium hydroxide, precipitated by treating aluminium sulphate etc. with sodium carbonate or bicarbonate, is washed free from sulphate, carbonate, etc. by decantation and heated with a soluble soap to yield a homogeneous emulsion which is boiled and, after the addition of boric acid, diluted with water. The product, after further dilution with water, is used for the mixing of Portland cement.

L. A. COLES.

Water-resistant composition of matter [for use in building]. O. W. STOREY and M. KLIEFOTH, Assrs. to C. F. BURGESS LABS., INC. (U.S.P. 1,742,794, 7.1.30. Appl., 1.4.27).—An intimate mixture of limestone of average particle size $8.4—11.0\ \mu$ with sodium silicate solution of viscosity not less than 2000 centipoises at 25° and containing $0.38—0.17$ pt. of sodium silicate per 1 pt. of filler, is heated at about 210° .

L. A. COLES.

Colouring [and glazing] of granular slate and the like. H. C. FISHER, Assr. to PHILIP CAREY MANUF. CO. (U.S.P. 1,742,440, 7.1.30. Appl., 13.4.25).—The material is successively treated with a solution containing lead acetate and borax, mixed with sodium silicate (and then with, e.g., iron sulphate, chromic oxide, copper sulphate if a coloured product is required), heated at about 1000° until it begins to coalesce, and cooled in the air.

L. A. COLES.

Cementitious compositions for manufacture of articles impervious to fluids. J. A. GREENE, and BINDPLAST PRODUCTS, LTD. (B.P. 325,255, 18.10.28).—The use is claimed of mixtures of sawdust, powdered leather, marble, sand, coal, etc. (100 pts.) with a binder consisting of 12.5 pts. of magnesium chloride and 35 pts. of calcined dolomite rendered plastic by the addition of dilute hydrochloric acid or chlorine solution; hardening agents, e.g., sodium silicate, zinc oxide, alum, or solutions of dextrin or celluloid in amyl acetate may also be added.

L. A. COLES.

Manufacture of flexible floor, wall, and like coverings. P. C. VAN DER WILLIGEN, and N.V. NEDERLANDSCHE LINOLEUMFABR. (B.P. 301,901, 22.11.28. Holl., 8.12.27).—The constituents of the material, e.g., oils or their oxidation or polymerisation products, or rubber latex, or asphaltic material, together with fillers, colouring material, vulcanisers, accelerators, anti-agers, etc. are mixed in the form of aqueous colloidal solutions or suspensions, and the colloidal material is coagulated, separated from the water, and worked up in the usual manner.

L. A. COLES.

Manufacture of coverings for floors, walls, etc. H. BÖRNSTEIN, and N.V. NEDERLANDSCHE LINOLEUMFABR. (B.P. 306,140, 12.2.29. Holl., 17.2.28).—Mixtures of cellulose benzoate plasticised, e.g., with tolyl phosphate, fillers, colouring material, and, if desired, oxidation products of drying oils are pressed hot into flat layers; a jute foundation may also be used.

L. A. COLES.

Manufacture of artificial timber and the like. A. E. MILLINGTON, Assr. to FIR-TEX INSULATING BOARD CO. (U.S.P. 1,742,410, 7.1.30. Appl., 26.5.27).—Fibrous wood stock etc., prepared as described in U.S.P. 1,708,586

(B., 1929, 554), is successively compressed to extract the water, loosened to a fluffy condition, impregnated with hot coal-tar creosote oil, and re-compressed in an unheated press to expel excess oil; the product may be strengthened by further compression with the application of heat. L. A. COLES.

Preparation of oxychloride cement. J. U. A. ORHSEN (U.S.P. 1,752,194, 25.3.30. Appl., 12.11.24. Denm., 13.11.23).—See B.P. 236,827; B., 1925, 720.

Manufacture of bituminous concrete. L. S. VAN WESTRUM, ASSR. to BITUCRETE, LTD. (U.S.P. 1,752,214, 25.3.30. Appl., 16.5.27. U.K., 31.5.26).—See B.P. 275,364; B., 1927, 780.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Decarburisation of carbon-iron alloys. L. HACKSPILL and E. SCHWARZ (Ann. Chim., 1930, [x], 13, 1—39).—At 1000° the chlorides and carbonates of the alkali and alkaline-earth metals effect superficial decarbonisation of cast iron and steel; in 2—3 hrs. the carbon is almost completely removed from hypoeutectoidal steels to a depth of about 1 mm. It is suggested that the active agent in this reaction is the alkali or alkaline-earth metal liberated from its salt by the iron. Calcium rapidly removes carbon and phosphorus almost completely from cast iron 1—2 mm. thick at 1000°, forming calcium carbide and phosphide. Lithium behaves similarly above 450°, but sodium and potassium above 800° act much more slowly as the carbides formed are relatively unstable at the ordinary pressure. Sodium carbide has been made by direct union of the elements under pressure at 1100°. A new and efficient form of Töpler vacuum pump is described and illustrated.

A. R. POWELL.

Jackhammer drill steel. G. R. HEYWOOD (Third Empire Min. Met. Congr., Apr., 1930, 23 pp.).—The paper contains a record of numerous life tests carried out on various drill steels of different origin and of different shapes and dimensions. It is concluded that the life of the steel is prolonged by smoothness of bore and by the use of an unforged shank; welding shortens the life of the jumper considerably. A. R. POWELL.

Cause of quenching deformation in tool steels. D. HATTORI (Sci. Rep. Tōhoku, 1929, 18, 665—698).—Experiments on the deformation produced by quenching prisms and cylinders of varying dimensions made of different kinds of steel are described. The causes of the deformation are the structural change and especially the thermal stress produced by unequal cooling of different parts of the specimen. The expansion due to structural change is produced in the steel specimen after it has been permanently deformed by thermal stress, and the change in shape is similar to that of a specimen which has been subjected to thermal stress only, except that the dimensions are increased in every direction by structural change. If the rate of cooling is slow this expansion and the thermal contraction occur at nearly the same rate, and the deformation becomes less. The warping of a tool steel is also due to structural change and thermal stress. To minimise the quenching deformation

and warping of a tool it should be made of a steel which hardens greatly even if it is cooled slowly, and it should be quenched at the lowest possible rate at which it becomes hard.

O. J. WALKER.

Changes in the properties of iron-nitrogen alloys by quenching and annealing below the A1 point. W. KÖSTER (Arch. Eisenhüttenw., 1929—1930, 3, 553—558; Stahl u. Eisen, 1930, 50, 254—255).—The solubility of nitrogen in α -iron decreases from about 0.5% N at the eutectoid temperature, 580°, to 0.015% at 20°, the iron nitride separating during cooling through this range in the form of thin lamellae. By quenching from just below 580° this separation can be prevented, the resulting brittle iron then undergoing age-hardening at the ordinary temperature; thus iron containing 0.12% N and having a hardness of 105 has a hardness of 190 after quenching from 550°, which increases to 270 in 28 days at the ordinary temperature and decreases to 105 in 1 hr. at 150°. Similar results are obtained with mild steel after nitriding and at the same time the temperature of the A1 transformation is depressed. The coercivity of steel containing nitrogen rises rapidly during the precipitation of the highly dispersed nitride on ageing. Magnetic and electrical resistance measurements indicate that the solubility of nitrogen in iron begins to increase at 100—150°, so that the tendency of the dispersed nitride to coagulate is very small.

A. R. POWELL.

Magnetic alloys of iron, nickel, and cobalt. G. W. ELMEN (J. Franklin Inst., 1929, 207, 583—617).—The results of 15 years' research on the magnetic properties of alloys of iron, nickel, and cobalt are reviewed, and the effect of heat-treatment is discussed. The intrinsic inductions for magnetising forces of 50 and 1500 gauss, the initial and maximum permeabilities, and the hysteresis loss for a maximum flux density of 5000 gauss are given in three-dimensional diagrams. Curves of magnetisation and permeability are also given for certain alloys. Two groups of alloys are of special interest. The iron-nickel alloys (permalloys) containing more than 30% Ni show after slow cooling a high permeability for low flux densities. The alloy containing 78.5% Ni has a maximum permeability of 120,000. A group of ternary alloys which have constant permeability and extremely low hysteresis loss at low flux densities are known as "perminvars." This group includes alloys containing 10—40% Fe, 10—80% Ni, and 10—80% Co, the most constant permeability for different magnetising forces being given by the 45% Ni, 25% Co, 30% Fe alloy.

C. J. SMITHELLS.

Determination of the specific volume of molten iron, nickel, and iron alloys. C. BENEDICKS, N. ERICSSON, and G. ERICSON (Arch. Eisenhüttenw., 1929—1930, 3, 473—486; Stahl u. Eisen, 1930, 50, 332—333).—The \bar{v} of molten iron at 1600° decreases in a smooth curve with increasing carbon content, the curve resembling the liquidus of the iron-carbon system. The sp. vol. of pure iron at 1600° is 0.1397, of the 1% C alloy 0.1461, and of the 4% C alloy 0.1566. The change of sp. vol. per 100° increases from 0.0020 with pure iron to 0.0038 with the 4% C alloy.

Iron-nickel alloys with 26, 36, and 40% Ni undergo a contraction when heated above 1600°, but pure nickel and alloys with 43.3, 60, and 80% Ni behave normally, although all the alloys have a sp. vol. greater than the weighted mean of the constituents, the deviation being greatest with alloys containing 30–40% Ni. These results tend to confirm the assumption that a definite compound Fe_2Ni exists. The sp. volumes of molten alloys of iron with aluminium, chromium, manganese, phosphorus, silicon, and tungsten are approximately directly proportional to the composition.

A. R. POWELL.

Corrosion of metals. F. MÜLLER (Z. angew. Chem., 1930, 43, 225–229).—A review of recent theories and experiments.

J. A. V. BUTLER.

Particle size as a factor in the corrosion of lead by soils. R. M. BURNS and D. J. SALLEY (Ind. Eng. Chem., 1930, 22, 293–297).—Corrosion of samples of lead buried in ground quartz of increasing particle size, and exposed to moist air at 40° for periods of 195–3601 hrs., was found to increase exponentially with particle size for the first 300 hrs., after which the rates of corrosion for all sizes became constant. Moisture influences the corrosion, which is caused by variations of oxygen concentration at the surface caused by contact with the particles. Similar results were obtained with natural soils.

S. I. LEVY.

"White rust" on galvanised ironware. E. H. SCHULZ (Stahl u. Eisen, 1930, 50, 360–362).—The white deposit, having a similar appearance to rust, which forms on galvanised articles during storage in a damp atmosphere, contains zinc oxide, water, and carbon dioxide, and, on addition of water, becomes pasty and gelatinises. Its formation is caused by condensation of water on the surface of the articles and the absorption by this water of carbon dioxide from the air. The composition of the zinc coating has no influence on the production of the deposit. Efficient ventilation of the storage room and the maintenance of an equable temperature prevent the "rusting."

A. R. POWELL.

Effects of impurities on copper. VI. Effect of phosphorus on copper. D. HANSON, S. L. ARCHBUTT, and G. W. FORD (Inst. Metals, Mar., 1930. Advance copy. 22 pp.; cf. B., 1927, 280).—In small quantities phosphorus removes oxides from copper (though both phosphorus and oxygen can be present together in copper), and improves both the cold-working properties and the fatigue range without impairing toughness. The presence of phosphorus increases the contraction on cooling and also the hardness of the cast ingots. Ingots containing 0.04% or more of phosphorus have a density approaching that of the corresponding rolled rod, but with more than 0.49% P the cast metal is liable to be unsound, though alloys up to 0.95% P may be hot-worked and further cold-worked after annealing. The limiting value for hot-working lies between 0.95 and 1.2% P. The tenacity of rolled copper is raised by the presence of phosphorus to a greater extent, at 250°, than by oxygen, iron, or arsenic, without any marked decrease in ductility. Ageing treatment did not improve the tensile properties of the alloys studied.

C. A. KING.

Gases in copper and their removal. W. E. PRYTHERCH (Inst. Metals, Mar., 1930. Advance copy. 8 pp.).—The marked reduction in the solubility of hydrogen and sulphur dioxide at the temperature of solidification of copper gives rise to unsound ingots (blowholes). The degree of unsoundness is dependent on the temperature of casting, the rate of solidification, and possibly on the size and shape of the ingot. Slow cooling facilitates escape of the occluded gases, which is aided also by passing an inert gas, e.g., nitrogen, into the metal, followed by remelting of the cooled copper. Under gas treatment, ingots had d 8.84 as compared with d 8.50 when cooled under ordinary conditions. Melting *in vacuo* was also an effective means of reducing dissolved gases, but the indirect oxidation of hydrogen by means of copper oxide with subsequent reduction of the oxidised copper was too slow to be of appreciable service.

C. A. KING.

Influence of gases on the soundness of copper ingots. N. P. ALLEN (Inst. Metals, Mar., 1930. Advance copy. 44 pp.).—The presence of hydrogen in molten copper can cause blowholes on cooling unless cooled sufficiently slowly, cooled under reduced pressure (about 6 cm. Hg), or cooled under the influence of a neutral gas, e.g., nitrogen, carbon dioxide, or carbon monoxide; the presence of arsenic or phosphorus tends to reduce or may even prevent the formation of blowholes, the nature of the reaction remaining unexplained. When oxygen is present in addition to hydrogen the blowholes are smaller, more numerous, and tend to segregate in the parts solidifying last, showing similarity to the unsoundness observed in commercial ingots. Oxygen to the extent of 0.002% is sufficient to give this effect, and unsoundness is considered due to the combined presence of cuprous oxide and hydrogen which may co-exist in equilibrium in molten copper. The removal of the last traces (0.0001%) of hydrogen is difficult, and the required action of deoxidisers consists in combining the oxygen present as an oxide incapable of being reduced by hydrogen, thereby preventing the production of steam during the freezing process.

C. A. KING.

Unsoundness in bronze castings. E. J. DANIELS (Inst. Metals, Mar., 1930. Advance copy. 18 pp.).—The work of Karr and Rawdon (cf. B., 1914, 1012; 1915, 1057, 1255), and of Carpenter and Elam (B., 1918, 245 A) on the effect of casting temperature and heat treatment on bronzes has been confirmed. Variations in temperature of casting and the composition of moulds influence the density of the cast alloy. The presence of hydrogen is a cause of unsoundness if the ingot is cooled too rapidly, but the bad influence may be annulled by a considerably longer cooling period. In this respect nitrogen, carbon dioxide, and carbon monoxide were found neutral towards bronze, and, generally, degasification of the molten alloy by means of nitrogen, deoxidisers, or presolidification gave negative results as far as improvement in density was concerned, the last-named, however, increasing the tensile strength. It is considered that the normally occurring unsoundness is due to the combined presence of hydrogen and oxygen, as it is different from the effect of hydrogen alone. Improved density when cast in sand is obtained by

melting in a pot furnace with a thin fuel bed and adequate draught.

C. A. KING.

Zinc-base die-casting alloys. R. LANCASTER and J. G. BERRY (Inst. Metals, Mar., 1930. Advance copy. 2 pp.).—The addition of 0.1% Mg to a die-cast alloy containing 4% Al, 3% Cu, and 93% Zn decreases appreciably the hot-shortness and increases the tensile strength from 13.5 to 15.7 tons/in.² and the angle of torque from 198° to 270°. With 0.2% Mg the corresponding values fall to 11.88 tons/in.² and 90°, and with 0.5% Mg to 10.76 tons/in.² and 95°. Apparently the magnesium prevents decomposition of the β -phase.

A. R. POWELL.

Macrostructure of cast alloys: effect of turbulence due to gases. R. GENDERS (Inst. Metals, Mar., 1930. Advance copy. 6 pp.).—The effect of volatile mould dressings on the macrostructure of brass ingots has been examined. During the casting process the mould dressing volatilises rapidly as the metal rises in the mould and the gases so formed escape through the body of the metal, the turbulence thus produced considerably modifying the macrostructure of the alloy. In long ingots the lowest portion consists of large crystals, but in the uppermost parts a finely-equiaxed structure containing numerous small blow-holes (sponginess) is produced. Photographs of the macrostructure of 70:30 brass cast in treated, partially treated, and in untreated moulds are included.

A. R. POWELL.

Aluminium-brasses. R. GENDERS (Inst. Metals, Mar., 1930. Advance copy. 19 pp.).—Addition of aluminium to brass reduces the range of copper content over which the alloy is ductile, but the alloy with 2% Al and 74–76% Cu has an elongation of 83% with a tensile strength of more than 17 tons/in.², and, owing to its great resistance to corrosion, it would appear to be a suitable alloy for use in the manufacture of condenser tubes. The alloy with 70% Cu and 2% Al is just as readily worked cold as the ordinary 70:30 brass, and is no more prone to season-cracking. Aluminium slightly reduces the workability of brasses at 500°, but at higher temperatures all the brasses become rapidly more plastic. As little as 0.1% Al increases the resistance of brass to oxidation at high temperatures and 2% Al confers a high immunity from oxidation during the annealing process usually performed in the manufacture of brass strip. The production of "red stain" during pickling is completely prevented by 1% Al, and with more aluminium pickling can probably be dispensed with. Careful casting is necessary with aluminium-brasses to prevent inclusion of oxide films, but with good casting conditions continual remelting produces little effect on the mechanical properties and results in only a small change in the chemical composition, the presence of aluminium reducing considerably the loss of zinc by volatilisation.

A. R. POWELL.

Concentration of ores by flotation. H. L. SULMAN (Third Empire Min. Met. Congr., Apr., 1930, 54 pp.).—The theory and practice of modern flotation processes are briefly discussed in all their aspects.

A. R. POWELL.

Water-concentration tests [for ores]. B. W. HOLMAN (Third Empire Min. Met. Congr., Apr., 1930,

62 pp.).—The value of various small-scale tests, panning, screening, heavy liquid separations, elutriation, and table tests, as a guide to the determination of the most satisfactory large-scale concentration procedure, is discussed with especial reference to certain Cornish tin ores.

A. R. POWELL.

Copper concentration as applied to Canadian ores. W. B. MAXWELL (Third Empire Min. Met. Congr., Apr., 1930, 16 pp.).—Details are given of the operation of flotation plants at five Canadian copper mines producing sulphide copper ores containing pyrite and pyrrhotite in a hard rock gangue. Except in minor details the methods used follow standard practice.

A. R. POWELL.

Metallurgical operations at Bwana M'Kubwa. C. S. VAN DER POEL (Third Empire Min. Met. Congr., Apr., 1930, 20 pp.).—Copper is extracted from the oxidised ore of this Rhodesian mine by leaching with a solution of cupric ammonium carbonate. The ore is first crushed to pass a $\frac{3}{4}$ -in. mesh and the product is screened on a $\frac{3}{4}$ -in. mesh. The oversize from the latter is heated to 350° by passing it through a rotating tube furnace internally fired with producer gas, and the hot ore then enters the reducing chambers, where it meets a current of producer gas which reduces the copper minerals to cuprous oxide and metallic copper, partial reoxidation taking place at the discharge end. The reduced ore and the fines from the previous screening are classified into sands and slimes, the sands being leached in layers 10 ft. deep by downward percolation of the ammonia liquor and the slimes in 4-in. layers in a Merrill filterpress by forcing the same liquor through the press. After washing both residues with a weak leach liquor, the sands are treated with steam and the slimes with hot water and compressed air to remove the remainder of the ammonia and copper. The rich leach liquor is boiled to effect precipitation of the copper and recovery of the ammonia, and the copper oxide is smelted to obtain a marketable product containing 99.88% Cu. Full details of the various operations with flow-sheets and some account of the geology of the deposits are given.

A. R. POWELL.

Metallurgy of Transvaal platinum ores. F. WARTENWEILER and A. KING (Third Empire Min. Met. Congr., Apr., 1930, 26 pp.).—A description with analyses is given of the various types of platinum ore which have been treated in the Transvaal for the recovery of their precious metal content. This is followed by a brief account with flow-sheets of the metallurgical methods which are used by the various mines in the production of concentrates by gravity and flotation (cf. Cooper and Watson; also Prentice, B., 1929, 602, 820).

A. R. POWELL.

Computation of the probable value of ore-reserves from assay results. S. J. TRUSCOTT (Third Empire Min. Met. Congr., Apr., 1930, 16 pp.).—An example is given, based on the results of sampling the ore reserves of a gold mine, of the method of calculating the probable value of these reserves. The assays are arranged in regular intervals and from the number of results within every interval the average frequency of these results is calculated. Each assay is then

weighted by the product of its frequency and itself, and the sum of these numbers is divided by the sum of the products of the frequency and assay. The result is shown to be a close approximation to the amount of metal subsequently recovered. A. R. POWELL.

Gold milling in Canada. J. J. DENNY (Third Empire Min. Met. Congr., Apr., 1930, 18 pp.).—A brief account, with the flow-sheets of the mills of seven Canadian gold mines, is given of modern methods of treating gold ores in Canada by flotation, amalgamation, and cyaniding. A. R. POWELL.

Gold metallurgy of Witwatersrand banket ores. H. A. WHITE (Third Empire Min. Met. Congr., Apr., 1930, 8 pp.).—Recent progress in the metallurgical treatment of gold ores on the Rand is reviewed. Modern tendencies are to dispense with stamp mills and separate treatment of the sand and slimes and to resort to "all sliming" in tube mills, so that of the ore going to the cyanide vats at least 90% passes 200-mesh. A. R. POWELL.

Metallic magnesium. W. R. D. JONES (Inst. Metals, Mar., 1930. Advance copy. 4 pp.).—Redistilled magnesium (99.99% Mg) after extrusion and annealing at 462° has a tensile strength of 7.6 tons/in.², an elongation of 3%, and a reduction of area of 2.1%; the corresponding values for commercial magnesium (0.051% Si, 0.059% Fe, 0.001% Cu, rest Mg) are: 12.1 tons/in.², 6%, and 5%. Quenching from 462° has no effect on these values for redistilled magnesium, but changes these for the commercial metal to 13.7 tons/in.², 3%, and 2.7%, respectively. The Brinell hardness of redistilled magnesium is 37–40, according to the amount of work and heat treatment it has undergone, and that of the commercial metal is 41–42. A. R. POWELL.

Discovery, preparation, properties, and applications of beryllium. G. MARCHAL (Chim. et Ind., 1929, 22, 1084–1092; 1930, 23, 30–33).—A history of the metal is given from the time of its discovery to the publication of the Stock-Goldschmidt process, which first produced the metal in a coherent form in 1921. The principal difficulties in the commercial development of this process are the high losses of metal by volatilisation and the use of sodium barium fluoride, which necessitates batch-working. The latter trouble has been removed by the use of beryllium oxyfluoride and barium fluoride, whilst the volatilisation products are recovered in a lead washer. The same washer may be used for absorbing the carbon fluoride produced. The crucible used is protected from the air by a covering of barium fluoride. The present mode of procedure, in which the current efficiency is 80% and the beryllium yield 95%, is described. A recent American process electrolyses beryllium and sodium chlorides at 700°, but the beryllium must be remelted to render it coherent. No method of electrolysis in solution has yet been successful. As pure beryllium has only recently been prepared the earlier descriptions of its properties are inaccurate. It is a very hard, brittle, steel-grey metal of d 1.84 and m.p. 1278°. In its physical properties it approaches boron in spite of its chemical similarity to aluminium. It is very difficult to work, but has been employed for windows in surgical X-ray tubes. In

alloys it raises the modulus of elasticity, being the only light metal with a high value for this property. It alloys with aluminium, but the products are not superior to alloys of the latter with the much cheaper element silicon. Copper and nickel with small proportions of beryllium form bronzes, the properties of which are greatly improved by heat treatment. They are resistant and sonorous. The pure metal is unaffected by air, but burns in oxygen. It is unaffected by water or by steam at a red heat, but is corroded by ammonium chloride solution and by hydrochloric and sulphuric acids or by hot nitric acid. It may be used as a reducing agent in the same way as aluminium. C. IRWIN.

Metallurgy of duralumin. J. MATHER (Rev. Aluminium, 1929, 6, 915–929).—Curves are given showing the effect of various heat treatments on the hardness and tensile properties of duralumin. The results show the bad effect of heating the metal, at any stage in its fabrication, above 510°; such metal should be immediately returned for remelting. For hot-rolling of duralumin the metal should be heated at 450° and work should cease when the temperature falls to 350°. Soft annealing is effected at 400° followed by slow cooling, but for age-hardening the metal must be quenched from 500°. Heat-treated and aged duralumin may be reheated at 225° for 10 min., at 200° for 1 hr., and at 150° for 100 hrs. without sensibly affecting its characteristic properties. A. R. POWELL.

Rational use of duralumin. M. PUBELLIER (Rev. Aluminium, 1929, 6, 931–949).—Practical hints are given for the heat treatment, mechanical working, joining, and polishing of duralumin, and various methods employed to protect it from corrosion are briefly reviewed. A table showing the mechanical properties of annealed, normal, and age-hardened duralumin is included. A. R. POWELL.

Almasilium. J. SUHR (Rev. Aluminium, 1929, 6, 955–972).—Almasilium is essentially an alloy of aluminium and magnesium silicide containing a slight excess of silicon and sometimes small amounts of chromium and manganese. The most satisfactory alloy is that with 1% Mg and 2% Si, which in the annealed state has an elastic limit of 5.6 kg./mm.², tensile strength of 10.6 kg./mm.², and elongation 29.5%; after quenching from 550° these values become 17.3, 29.2, and 27.5, respectively, but subsequent ageing at 175° for 6 hrs. produces a tensile strength of 40 kg./mm.² with an elongation of 6% and elastic limit of 36 kg./mm.² Addition of 0.5% Cr or of 0.5% Mn increases slightly the tensile strength, elastic limit, and hardness, but reduces the elongation. The metal takes a high polish and may be employed in place of mild steel for many purposes where good working properties combined with a high strength are required. A. R. POWELL.

Cold-working of lead, tin, and cadmium at different temperatures. A. MOLNAR (Compt. rend., 1930, 190, 587–589).—Cold-working of lead to the extent of 25–50% at –40° to –75° produces a permanent increase in hardness, whilst at –40° to +50° the hardness returns to its original value at a rate which is greatest at the higher temperatures and is

almost instantaneous above 50° . For tin and cadmium the effect is permanent below -20° and -40° , and recovery is instantaneous at $+50^{\circ}$ and $+25^{\circ}$, respectively. J. GRANT.

Spectrographic determination of cadmium, lead, and iron in zinc. D. M. SMITH (Trans. Faraday Soc., 1930, 26, 101—117).—A review is given of the methods of spectrographic examination of alloys for traces of impurities and the relative merits of the methods are compared. The arc method is to be preferred on account of its greater sensitivity in the case of lead. By the technique described impurities may be determined with certainty between definite limits and can be estimated approximately within these limits. Values are given of the wave-lengths and intensities of the most suitable lines for the identification and estimation of cadmium, lead, and iron in zinc of various qualities. F. G. TRYHORN.

Rapid volumetric method for determination of lead. R. C. WILEY (Ind. Eng. Chem. [Anal.], 1930, 2, 124—126).—The lead in the material is separated as sulphate and boiled with sodium carbonate to convert it into carbonate, which is dissolved in nitric acid. Ammonia is added until a permanent precipitate appears, an excess being avoided, and the solution is titrated at a temperature near its b.p. with sodium molybdate solution, a 1 : 1 mixture of stannous chloride and potassium thiocyanate dissolved in a little water being employed as external indicator; a red colour is produced when the end-point has been reached. The method can be used for the determination of lead in alloys containing antimony and tin; small amounts of iron and copper do not interfere. H. F. HARWOOD.

Atmospheric action in relation to fatigue in lead. B. P. HAIGH and B. JONES (Inst. Metals, Mar., 1930. Advance copy. 11 pp.).—Observation on the fatigue cracking of lead shows that the mode of fracture in the exterior metal is quite different from that of the interior, which is probably a characteristic of all fatigue fractures. The fracture in the marginal zone is usually formed by shearing along the surface of a "cup and cone," and is intercrystalline only in exceptional cases as in lead and certain other metals. The inner zone varies very little in character in relation to grain size, and it is thought that the inner zone reveals the fundamental action of fatigue and that the character of the marginal ring is relatively unimportant. In lead and lead alloys the inner zone is discoloured, which suggests oxidation, and it is inferred that oxygen diffuses through the metal during a fatigue test in air, reaching a depth at which the fatigue crack shows the characteristic form and discoloration. Fatigue was delayed when test pieces were immersed in oil or water or when protected by a layer of grease, and was eliminated when immersed in acetic acid, though unaffected if the surface of the test piece was only moistened with acetic acid during the test. Reduction in fatigue strength under the surface action of reagents is attributed only in small degree to superficial action and chiefly to diffusion into the metal of foreign substances that promote chemical or physical change under cyclic stress. C. A. KING.

Electrochemistry of chromium. A. V. PAMFILOV and G. F. FILLIPITSHEV (J. Russ. Phys. Chem. Soc., 1929, 61, 2221—2244).—The generally accepted opinion that chromium plating proceeds satisfactorily only with high concentrations of chromic acid, and in the absence of other than negligible quantities of tervalent chromium, is not correct, as electrodeposition of chromium takes place satisfactorily from chromium sulphate solutions containing 4—160 g. Cr per litre, the yield of metal being 0—25%, according to the voltage used, the density of current, etc. The yield and the quality of the deposit are better at 0.5 than at 0.05 amp. Carveth and Mott's view (B., 1905, 625) that the presence of bivalent chromium in the solution is essential to ensure proper deposition of metal is not confirmed. R. TRUSZKOWSKI.

Protective value of some electrodeposited coatings. L. DAVIES and L. WRIGHT (Inst. Metals, Mar., 1930. Advance copy. 12 pp.).—The protective value of coatings of zinc, cadmium, nickel, and chromium, 0.0001, 0.0005, 0.001, and 0.002 in. thick on steel, brass, phosphor-bronze, and copper has been determined by subjecting the plated metals to sprays of sodium chloride solution (24 g./litre) and 0.05*N*-sulphuric acid. Cadmium afforded better protection than zinc to all the metals in the acid spray, but the thin zinc deposits were better than the corresponding cadmium deposits in the salt spray. Chromium afforded no protection whatever to steel, but gave excellent protection to the non-ferrous metals tested. Only the thickest nickel deposit gave any degree of permanent protection to steel, but the non-ferrous metals were well protected by 0.001 in. of nickel deposit. A. R. POWELL.

PATENTS.

Cupola furnaces. F. A. STEVENSON, ASSR. to ECONOMY METAL PRODUCTS CORP. (U.S.P. 1,733,138—1,733,141, 29.10.29. Appl., [A] 7.2.28, [B] 29.6.28, [C, D] 26.2.29).—(A) A portion of the lining of the cupola consists of a number of loosely fitting rings composed of numerous segments connected together in such a way that every ring and segment can expand individually. (B) The upper part of the shaft has a metal lining surrounded by an outer sheathing, forming an annular chamber through which cooling air is passed. (C) A portion of the shaft is lined with a series of superimposed rings, and is surrounded by a sheathing so as to form a preheating chamber for the air; means independent of the sheathing are provided for preventing distortion of the rings. (D) The rings are maintained in vertical alinement by means of retainers supported close to the junction of the rings by fixtures at the top and bottom of the annular heating chamber. A. R. POWELL.

[Annealing] furnace and its operation. E. H. SWINDELL and F. W. BROOKE, ASSIS. to W. SWINDELL & BROS. (U.S.P. 1,741,209, 31.12.29. Appl., 29.1.23).—The furnace comprises a high-temperature, electrically-heated chamber with low-temperature, fuel-fired, pre-heating chambers on each side of it, so arranged as to minimise heat losses from the central chamber. The articles to be annealed pass through an outside chamber into the central chamber continuously on a series of

trucks running on rails, those leaving the central chamber passing close to those entering it to effect a heat interchange.

A. R. POWELL.

Heat treatment of metals. A. A. SOMERVILLE, Assr. to R. T. VANDERBILT Co., INC. (U.S.P. 1,742,791, 7.1.30. Appl., 15.7.26).—Oils used for tempering and quenching metals may be stabilised by the addition of a small amount of a condensation product of an aldehyde with ammonia or an amine, *e.g.*, of formaldehyde, acetaldehyde, etc. with aniline, naphthylamine, etc.

A. R. POWELL.

Production and utilisation of carbon monoxide [for treatment of iron ores]. L. BRADLEY (U.S.P. 1,742,750, 7.1.30. Appl., 7.4.26).—In the smelting of iron ores in the blast furnace, the blast used comprises a mixture of carbon dioxide and oxygen with subordinate amounts of nitrogen, with or without a certain proportion of steam, and the molten iron produced is tapped into an open-hearth furnace which is fired with the gas mixture obtained from the blast-furnace flues; this gas has a high content of carbon monoxide and hydrogen. Part of the waste gases from the open-hearth furnace and part of those from the blast furnace are mixed with oxygen and the mixture is returned to the tuyères of the blast furnace after being preheated by further quantities of the flue gases.

A. R. POWELL.

Carburising material [for iron or steel]. G. W. PRESSELL, Assr. to E. F. HOUGHTON & Co. (U.S.P. 1,741,336, 31.12.29. Appl., 14.7.28).—Granular carbonaceous material is coated with a mixture of barium carbonate and lime mixed with molasses, and the grains are then coated with kieselguhr and finally with an energising substance, *e.g.*, manganese dioxide. The product is a highly efficient case-hardening agent.

A. R. POWELL.

Production of soft iron. B. WEISHAN (U.S.P. 1,742,111, 31.12.29. Appl., 11.3.27. Ger., 29.9.26).—Puddled iron is formed into bars of equilateral triangular cross-section with a flattened apex, six of these bars are bound together to form a hexagonal bundle with a central, longitudinal passage-way, and the bundle is then welded into a coherent whole by rolling, whereby slag is extruded from the central hole and a bar having a long-grained fibrous structure is obtained.

A. R. POWELL.

Inhibitor [for steel pickling]. H. P. CORSON, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,742,986, 7.1.30. Appl., 9.8.28).—A small quantity of cinchona bark alkaloid, especially quinidine, is added to the usual acid pickling bath.

A. R. POWELL.

Manufacture of metal [iron] powders suitable for magnet cores. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,526, 21.9.28).—A mixture of 40% of iron powder, prepared by the thermal decomposition of iron carbonyl, and 60% of a similar powder which has been purified by heating in hydrogen at 500° is formed into a core in the usual way.

A. R. POWELL.

Manufacture of magnetic [iron-nickel] alloys. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. E. RUDER (B.P. 314,971, 1.10.28. U.S., 6.7.28).—An alloy of 40–50% of electrolytic nickel and 60–50% of iron

free from impurities is melted in an induction furnace in hydrogen or in a vacuum and the ingots are rolled into sheet which is annealed at 1200–1300° for 2–10 hrs. until a large twin-crystal structure is obtained. The alloy has a high initial permeability, and is thus suitable for transformer cores.

A. R. POWELL.

Applying zinc coatings to iron articles. T. LIBAN (B.P. 325,540, 21.11.28).—The zinc bath is covered with a layer of molten salts comprising 0.5–6% of sodium aluminium chloride, 10–20% of ammonium chloride, and 74–89.5% of zinc chloride. When the combined lead-zinc galvanising bath is used the salt mixture is placed above the lead portion of the bath.

A. R. POWELL.

Welding of silicon-containing steel alloys. F. KRUPP A.-G. (B.P. 313,434, 4.3.29. Appl., 11.6.28).—A welding rod containing about 1.5–3% Mn is used for arc-welding steel sheets containing more than 0.3% Si, so that the joint contains at least 0.3% Mn.

A. R. POWELL.

Froth-flotation concentration of ores. MINERALS SEPARATION, LTD., Assees. of C. H. KELLER (B.P. 301,832, 5.12.28. U.S., 6.12.27).—The flotation agent used is the heavy oil obtained by refluxing an aromatic hydrocarbon, *e.g.*, benzene, with sulphur and aluminium chloride or with sulphur monochloride and zinc; the reagent consists chiefly of phenyl sulphide.

A. R. POWELL.

Treatment of minerals containing potassium, aluminium, and iron. C. E. ARNOLD, Assr. to ELECTRO Co. (U.S.P. 1,742,191, 7.1.30. Appl., 19.2.25).—The mineral, *e.g.*, greensand, is heated with sulphuric acid and the filtered solution is evaporated until the anhydrous sulphates separate. These are collected and heated at 500° to decompose ferric sulphate, the product is ground with water in a ball mill, and the ferric oxide collected for use as a pigment. The filtrate is evaporated and crystallised to obtain potassium aluminium sulphate.

A. R. POWELL.

Refining of asbestos ore. F. A. METT, Assr. to POWHATAN MINING CORP. (U.S.P. 1,741,869, 31.12.29. Appl., 23.8.23).—The material passes through a jaw breaker into rolls immersed in a vessel through which an upward current of water is rising. The discharge from the rolls is thus graded into coarse sand, which sinks, and partially purified fibres, which float over to another vessel containing a submerged, inclined, reciprocating screen by the action of which the remainder of the sandy material is separated from the asbestos fibre. The latter floats away as a dense mat into a settling chamber.

A. R. POWELL.

Briquetting of flue dust. E. A. SLAGLE and B. M. O'HARRA, Assrs. to AMER. SMELTING & REFINING Co. (U.S.P. 1,741,544, 31.12.29. Appl., 9.9.26).—Dust from furnace flues, bag-houses, or the Cottrell plant is briquetted by mixing it with up to 2% of an acid deflocculating substance, *e.g.*, sulphuric acid, and the necessary water to give a paste suitable for the briquetting machine.

A. R. POWELL.

Manufacture of multiple metals [iron impregnated with copper]. INTERNAT. GEN. ELECTRIC Co.

INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 308,819, 27.3.29. Ger., 31.3.28).—Powdered iron, with or without a binding material, is pressed into the desired shape, covered with powdered copper, and heated until the copper melts and runs into the pores of the sintered iron, thus displacing the binder and making a tough, coherent mass. A. R. POWELL.

Provision of insulating coatings on aluminium or aluminium alloys. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of L. McCULLOCH (B.P. 300,912, 19.11.28. U.S., 19.11.27).—The metal is suspended in boiling water, to which are added 5 g. of slaked lime and 5 g. of gypsum per litre, whereby a coherent white film containing about 76.8% Al_2O_3 , 8.1% CaO , and 15.1% SO_4 is obtained after washing and drying at 200°. The film may be rendered more insulating by impregnating it with collodion or varnish. A. R. POWELL.

Production of beryllium alloys, in particular those with a high beryllium content, by means of fused electrolysis. SIEMENS & HALSKE A.-G. (B.P. 325,762, 23.5.29. Ger., 13.11.29).—The metal to be alloyed with the beryllium forms part or all of the anode of the cell, and the electrolyte is such that this metal is dissolved anodically and redeposited together with beryllium at the cathode, the temperature of the electrolyte being below that of the m.p. of the anode metal. The process is suitable for the production of copper or nickel alloys with 15–35% Be, which are useful as a means of introducing small quantities of beryllium into copper or nickel. A. R. POWELL.

Production of metals in electric furnaces. E. G. T. GUSTAFSSON (U.S.P. 1,751,083, 18.3.30. Appl., 11.5.27. Swed., 27.9.26).—See B.P. 278,005; B., 1928, 790.

Smelting process [for low-carbon cast iron]. F. WÜST (U.S.P. 1,751,185, 18.3.30. Appl., 18.10.27. Ger., 6.8.26).—See B.P. 275,646; B., 1928, 372.

Apparatus for reducing oxides of metals [copper, lead, or zinc]. J. W. HORNSEY, Assr. to GRANULAR IRON Co. (U.S.P. 1,751,736, 25.3.30. Appl., 8.9.26).—See B.P. 277,325; B., 1928, 931.

Manufacture of zinc from oxygen compounds of zinc or substances containing them. C. FREIHERR VON GIRSEWALD and H. NEUMARK (U.S.P. 1,751,778, 25.3.30. Appl., 12.10.28. Ger., 13.10.27).—See B.P. 298,636; B., 1929, 725.

Apparatus for continuous treatment of metal bodies. M. FOURMENT (U.S.P. 1,749,700, 4.3.30. Appl., 22.12.26. Fr., 29.12.25).—See B.P. 263,774; B., 1927, 943.

[Aluminium] alloy. P. BARTHÉLEMY and H. DE MONTBY (U.S.P. 1,750,700, 18.3.30. Appl., 9.8.27. Fr., 20.5.27).—See F.P. 634,751; B., 1930, 289.

Aluminium alloy. A. GEYER (U.S.P. 1,750,751, 18.3.30. Appl., 25.6.27. Fr., 4.2.27).—See B.P. 284,722; B., 1929, 100.

Autogenous welding of magnesium and its alloys. G. MICHEL, Assr. to H. O. BERG (U.S.P. 1,749,712, 4.3.30. Appl., 5.2.25. Fr., 23.12.24).—See B.P. 261,528; B., 1927, 80.

Manufacture of hard-metal alloys. K. SCHRÖTER, Assr. to GEN. ELECTRIC Co. (Re-issue 17,624, 18.3.30, of U.S.P. 1,549,615, 11.8.25).—See B., 1925, 766.

Protection of metals [from amalgamation with mercury]. S. Z. DE FERRANTI, Assr. to FERRANTI, LTD. (U.S.P. 1,752,239, 25.3.30. Appl., 10.2.25. U.K., 4.3.24).—See B.P. 234,210; B., 1925, 597.

Protection of readily oxidisable metals [magnesium and its alloys]. G. MICHEL, Assr. to H. O. BERG (U.S.P. 1,749,713, 4.3.30. Appl., 13.8.25. Fr., 20.3.25).—See B.P. 249,484; B., 1927, 785.

Decorating [tinplate] metal [with bronzing preparation]. J. DE FRENE (B.P. 326,257, 13.12.28).

Furnaces for de-tinning metal plates and other articles or surfaces coated with block tin or other like material. LEEDS DE-TINNING, LTD., and A. W. CALVERT (B.P. 326,237, 8.12.28).

Apparatus for electromagnetic separation of minerals. A. DAVIES (B.P. 326,223, 9.11.28).

Heat-treatment furnaces (B.P. 325,552).—See XI. Transferring subjects to metal (U.S.P. 1,742,710).—See XXI.

XI.—ELECTROTECHNICS.

Gas formation and dissociation due to electric arcs in oil. A. VON ENGEL (Wiss. Veröff. Siemens-Konz., 1930, 8, [iii], 97–108).—The theory of the formation of gas in electric oil-switches has been investigated. The energies of vaporisation and of dissociation of the paraffins have been calculated from the heat of formation and from the combustion equations. The formation of gas and soot by the dissociation of paraffins depends, in amount, on the ratios of the hydrogen, acetylene, and methane formed. Calculations show that the amount of gas formed is much smaller than that theoretically possible from the energy liberated in the arc when the switch is opened. It is concluded that the rest of the energy is utilised in raising the temperature, in general, so that the highest temperature in the switch is such that dissociation of the paraffins can take place. W. E. DOWNEY.

Gas-filled photoelectric cells: their properties and calibration. J. KUNZ and V. E. SHELFORD (Rev. Sci. Instr., 1930, 2, 106–117).—Spherical cells show a direct proportionality between light intensity and current up to a limit depending on the size of the cell. For ecological work a cell with a convex sensitive surface is the most satisfactory. With concave surfaces the response depends very largely on the angle of incidence. C. W. GIBBY.

Hydrocarbons and a high-tension discharge. BECKER. High-tension currents and brine-petroleum emulsions. STARZEWSKI.—See II. Paper for cables. GYEMANT.—See V. Phosphoric acid production. KOCHS; KLUGH.—See VII. Chromium. PAMFILOV and FILLIPITSHEV. Protective coatings. DAVIES and WRIGHT. Magnetic alloys. ELMEN.—See X.

PATENTS.

Electric heating apparatus for liquids. H. H. HENNING (B.P. 325,258, 12.11.28).—Electric current

passes between electrodes immersed in liquid to be heated contained in an electrically-insulated vessel; the lower portions of the electrodes are parallel and overlapping, but the upper portions diverge widely, so that current in the liquid is concentrated in the lower part. J. S. G. THOMAS.

Electrodes for electric heat-treatment of metallic articles. F. W. THOMPSON (B.P. 325,552, 18.10.28).—Graded resistance elements provide paths of different electrical resistance between the source of current and the article to be treated, so that the portions of the article and electrode in contact may be given any desired temperature gradient. J. S. G. THOMAS.

[Carbonaceous] electrical resistor, conductor, etc. J. KELLEHER, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,742,259, 7.1.30. Appl., 17.7.25).—Carbonaceous material, coated with silicon carbide and borax, is heated in air until the borax melts, and a semi-plastic coating composed internally principally of silicon carbide and externally of oxidised boron is produced. J. S. G. THOMAS.

Luminous electric discharge tubes. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 315,704, 1.7.29. Ger., 16.7.28).—In luminous tubes filled with common gases and containing a substance which is heated during operation of the tube to replenish its gas content, an absorbent substance, *e.g.*, tin, bismuth, lead, or gold, which does not react with the gas is mixed with the gas-emitting substance to form a compound or alloy of very low vapour pressure with the residue resulting from the decomposition, *e.g.*, with the alkali or alkaline-earth metal that has given up nitrogen or hydrogen to the gas filling. J. S. G. THOMAS.

Electric discharge tubes. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 325,885, 3.12.28).—The insulation resistance of discharge tubes containing the vapours of an alkali metal (or metals) is increased by filling the tubes with hydrogen at a pressure of a few mm. of mercury. If desired, a discharge may be passed through the gas. [Stat. ref.] J. S. G. THOMAS.

Electron-discharge device. A. J. KLONECK (U.S.P. 1,742,262, 7.1.30. Appl., 23.10.22).—One of a number of electrodes arranged in a bulb is coated with silver upon which a coating of an alkali oxide is deposited, whilst the others, one is covered with mercury and another with an active alkali amalgam. J. S. G. THOMAS.

Thermionic cathodes for electric-discharge devices. STANDARD TELEPHONES & CABLES, LTD., and L. H. BEDFORD (B.P. 325,492, 16.11.28).—Pure alkaline-earth metal(s) is deposited from the vapour state upon a coating of oxide(s) of the same metal(s) formed upon a core, *e.g.*, of platinum-nickel, so that the thermionically-active material consists of a complex of alkaline-earth metal and oxide. If, *e.g.*, barium be used, the metal may be deposited from the product resulting from firing a mixture of barium oxide, barium peroxide, and aluminium with a fuse powder. J. S. G. THOMAS.

[Cathode for] gas-filled electric-discharge devices. SIEMENS & HALSKE A.-G. (B.P. 302,583,

14.12.28. Ger., 17.12.27).—A refractory-metal core *e.g.*, of tungsten or molybdenum, is coated with one or more rare-earth metals, *e.g.*, lanthanum, cerium, ytterbium, or elements of similar emissive characteristics, *e.g.*, yttrium, zirconium, together with thorium, if desired. J. S. G. THOMAS.

[Manufacture of] cathodes for thermionic devices. P. FREEDMAN (B.P. 325,317, 4.12.28 and 29.3.29).—A coating consisting principally or wholly of one or more alkaline-earth carbonates is electrolytically deposited upon a metallic core, *e.g.*, of platinum, tungsten, nickel, ferronickel (64% Fe, 36% Ni), from an electrolyte containing alkaline-earth compounds and carbon dioxide. If desired, the electrolyte may contain methyl or ethyl alcohol together with acetone and/or amyl acetate, ether, pyridine. An electrolytically deposited catalyst, *e.g.*, an oxide of chromium, magnesium, calcium, cadmium, may be incorporated in the coating. J. S. G. THOMAS.

Wehnelt cathodes. N.V. PHILIPS' GLÖEILAMPEN-FABR. (B.P. 309,578, 18.3.29. Holl., 13.4.28).—A core coated with a suspension of barium carbonate, beryllium carbonate, zirconium oxide, and, if desired, an agglutinant, *e.g.*, gum arabic, is heated at about 1000°, preferably *in vacuo*. J. S. G. THOMAS.

[Contact] current-rectifying device. W. O. SNELLING (U.S.P. 1,742,324, 7.1.30. Appl., 26.1.23).—A compound of a metal with an element of the sulphur group is employed. Thus, *e.g.*, a consolidated mixture of red lead and sulphur is heated at above the m.p. of sulphur. J. S. G. THOMAS.

Introducing alkaline-earth or rare-earth metal vapours into evacuated or partially evacuated vessels [e.g., electric vacuum tubes]. STANDARD TELEPHONES & CABLES, LTD., and L. H. BEDFORD (B.P. 325,534, 16.11.28).—The oxide of the metal is reduced aluminothermically and the mass powdered, mixed with paraffin wax, and applied as a paste to a valve plate. In this way barium may be conveniently introduced into a radio valve and, after evacuating the valve, the barium is readily volatilised. A. R. POWELL.

[Solid depolarising electrolyte for] primary electric cells and batteries. A. SCHMID, and METROPOLE DEVELOPMENTS, LTD. (B.P. 325,483, 17.9.28).—An oxidising agent, *e.g.*, sodium dichromate and/or chromic acid, is mixed to a paste with concentrated sulphuric acid and solidified by addition of silicic acid or water-glass. J. S. G. THOMAS.

[Positive plates for] electric accumulators [having an alkaline electrolyte]. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 325,581, 22.11.28).—The active mass consisting of oxygen compounds of nickel or cobalt, moistened, if desired, with a highly viscous water-soluble liquid, *e.g.*, glycerin, is mixed with foliated graphite of dimensions 0.06–0.75 mm., subjected to high pressure, *e.g.*, 3500 kg./cm.², and charged into pockets in the plates. J. S. G. THOMAS.

Manufacture of porous bodies, more particularly for electro-osmotic purposes. SIEMENS & HALSKE A.-G. (B.P. 305,020, 2.1.29. Ger., 28.1.28).—Diaphragms

for electric endosmosis are made by stirring one or more acid- and alkali-proof inorganic compounds with an acid- and alkali-proof hardening solvent, which is subsequently removed by heating the pressed mass at a low temperature (300°). If desired, the pore volume is increased by adding to the initial materials substances which can subsequently be removed either by dissolution or heat-treatment. Thus, *e.g.*, heavy spar, chromium oxide, or aluminium oxide, together with, if desired, sawdust, naphthalene, sodium chloride, potassium chloride, or copper sulphate, are added to a solution of celluloid in amyl acetate or to a solution of natural asphalt, the resulting mass being pressed and heated.

J. S. G. THOMAS.

Production of insulating masses of high disruptive strength. SIEMENS & HALSKE A.-G. (B.P. 306,900, 19.2.29. Ger., 27.2.28).—A gas or vapour is passed into liquefied material at such temperature that the gas or vapour is decomposed and finely-divided metal is deposited and remains suspended in the liquid. Thus jets of vaporised iron carbonyl or nickel carbonyl may be injected into melted paraffin, or silicon hydride or boron hydride into molten glass or enamel.

J. S. G. THOMAS.

Treatment of liquids and organic substances by irradiation. V. C. FROM, C. D. ROWLEY, and A. W. LARSKY (B.P. 325,824, 23.11.28, 21.12.28, and 2.2.29).—Substances are subjected to the simultaneous action of ultra-violet and infra-red radiation at about 0–15° in the presence of ozone. Suitable apparatus is described.

J. S. G. THOMAS.

Manufacture of liquid or solid products by gaseous reaction under the influence of silent electrical discharge. I. G. FARBENIND. A.-G. (B.P. 300,282, 10.11.28. Ger., 11.11.27. Addn. to B.P. 304,623; B., 1929, 290).—Deposition of reaction products is prevented on one electrode only. Thus, in the manufacture of hydrogen peroxide, the outer tube is cooled to 20° and the inner to 60° only, so that no condensation of hydrogen peroxide occurs on the inner tube.

J. S. G. THOMAS.

[Nickel] anode and its manufacture. W. J. HARSHAW, P. M. SAVAGE, and F. K. BEZZENBERGER, Assrs. to INTERNAT. NICKEL CO., INC. (U.S.P. 1,751,630, 25.3.30. Appl., 28.11.27).—See B.P. 314,667; B., 1929, 688.

[Plastic mass for] electric accumulator [plates]. H. LEITNER (U.S.P. 1,750,230, 11.3.30. Appl., 28.3.29. U.K., 27.3.28).—See B.P. 316,328; B., 1929, 824.

Securing good electrical contact with crystalline cuprous oxide. W. T. ANDERSON, JUN., and L. F. BIRD, Assrs. to HANOVIA CHEM. & MANUF. CO. (U.S.P. 1,749,995, 11.3.30. Appl., 28.10.27).—See B.P. 307,962; B., 1929, 401.

[Method of supporting bricks of] electric furnaces. WILD-BARFIELD ELECTRIC FURNACES, LTD., and L. W. WILD (B.P. 325,548, 21.11.28).

Electric discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 325,870, 1.11.28).

Electric discharge devices. [Lightning arresters.] BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. E. POTTER (B.P. 302,168, 10.12.28. U.S., 10.12.27).

X-Ray tubes. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 310,001, 15.4.29. Holl., 19.4.28).

Apparatus for treating liquids with ultra-violet rays. Apparatus for producing ultra-violet rays. E. O. SCHEIDT, and FOODSTUFFS IRRADIATION CO., LTD. (B.P. 326,249 and 326,250, 10.12.28).

[Spiral filaments for] electric incandescence lamps. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H., (B.P. 316,612, 5.7.28. Ger., 1.8.28).

Electric batteries and electrodes therefor. C. H. EVERETT and G. R. CARR (B.P. 303,472, 10.8.28. U.S., 4.1.28).

Pyrometer (B.P. 324,500). **Colorimeters** (B.P. 324,351 and 324,495).—See I. **Pottery kilns** (B.P. 325,794). **Furnace linings or crucibles** (B.P. 325,661).—See VIII. **Magnet cores** (B.P. 325,526). **Magnetic alloys** (B.P. 314,971). **Insulating coatings** (B.P. 300,912). **Beryllium alloys** (B.P. 325,762).—See X.

XII.—FATS; OILS; WAXES.

Thiocyanates of unsaturated fatty acids. W. KIMURA (J. Soc. Chem. Ind. Japan, 1930, 33, 58–59 B, and Chem. Umschau, 1930, 37, 72–74).—The thiocyanogen additive products of oleic, elaidic, and brassidic acid, prepared by the action of free thiocyanogen on the acids in carbon tetrachloride solution, have been isolated in the pure condition. The oleic acid derivative was a pale yellow oil, but *elaidic* and *brassidic thiocyanates*, on recrystallisation from absolute alcohol, formed fine needle-shaped crystals having m.p. 79–79.5° and 57.5–58°, respectively. These compounds are insoluble in water, but soluble in absolute alcohol, ether, carbon tetrachloride, and acetic acid. E. LEWKOWITTSCH.

Fatty oil from tobacco seeds. M. P. PIATNITZKI (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 61, 20 pp.).—Tobacco seeds on extraction with light petroleum yielded an oil (unsaponifiable matter 1.2%) containing about 60% of linoleic, 21.7% of oleic, 9.6% of palmitic acid, and about 10% of glycerol. Linolenic acid appears to be absent. No alkaloids have been found in the oil. W. O. KERMACK.

Tung oil. VI. A. EIBNER and E. ROSSMANN (Chem. Umschau, 1930, 37, 65–71).—The hypothesis of the formation of preliminary cracks in a drying wood oil film (cf. B., 1928, 934) is rejected and the observations and conclusions of Blom (cf. B., 1929, 783) that the primary wrinkling is due to the development of introverted folds are confirmed and extended, and photomicrographs of the phenomenon are shown. Similar folds can be produced artificially by pressure along the surface of the skin in linseed oil films. The occurrence of strain double-refraction in old and fresh wrinkled wood oil films is confirmed; with poppyseed oil films this property is lost after a few weeks. The isomerisation of wood oil on exposure to light and the rôle of β -elæostearin in the drying process are discussed. E. LEWKOWITTSCH.

Cacao butter. III. Concentration of the unsaturated components of cacao butter in order to ascertain the purity. H. P. KAUFMANN (Chem. Umschau, 1930, 37, 17—21. Cf. B., 1929, 441; 1930, 247). **IV. Detection of hardened fats by spectroscopic identification of nickel.** H. P. KAUFMANN and M. KELLER (*Ibid.*, 49—60).—III. In a few cases the adulteration of cacao butter (*e.g.*, by Makron butter) may be indicated by an abnormal thiocyanogen value. By removal of the more saturated component glycerides of cacao butter by crystallisation from acetone (20 g. dissolved in 200 c.c. is kept for 6—20 hrs. at 15°, and then for 1 hr. at 5°, when the precipitate is removed by filtration) a more unsaturated soluble fraction is obtained, of which the iodine value (54—58, usually 55—56) is characteristic for expressed cacao butter. All other [unhardened] fats are more soluble in acetone than is cacao butter and pass into the soluble fraction and the iodine value ("enriched iodine value") of the latter is either increased above (*e.g.*, with cottonseed and soya-bean oils) or depressed below (with coconut oil as adulterant) the normal value. Additions to cacao butter of 2% of coconut and/or cottonseed oil etc. are clearly detected by this method.

IV. The iodine value of typical hardened fats (coconut, arachis, whale, etc.) was approximately the same as the thiocyanogen value: the "partial iodine value" (determined by bromine), however, which in the original oils is about equal to the thiocyanogen value, falls considerably below it (sometimes down to 50%) in hardened oils. Similar relations between these constants is found with the "solid acids" from the lead salt-alcohol separation of the hardened oils. The characteristic low partial iodine value may on occasion reveal the presence of hardened fats in cacao butter; the test becomes more sensitive if applied to the acetone-soluble fraction as described above. With only small admixtures of hardened fats the results are inconclusive, and the most reliable indication of such adulteration is given by proof of the presence of nickel. The chemical processes extant for this purpose are reviewed at some length, and details of a new spectroscopic analysis have been worked out. The nickel solution obtained from cautiously ashed fat (1—50 g.), or by acid extraction, is electrolysed, the nickel being deposited on the tip of copper-plated platinum wire electrodes, and subsequently identified by its spark spectrum. Careful manipulation is essential, but quantities down to 10⁻⁶ mg. of nickel (0.0001 mg. of nickel/kg. of fat mixture; 0.001 mg./kg. was the lowest found in any pure hardened fat) can be identified with certainty: nickel could not be found in any pure cacao butter examined, nor even in a fat that had been stirred for $\frac{1}{2}$ hr. at 65° with a nickel spatula.

E. LEWKOWITSCH.

Nutritive value of hardened oils. III. Influence of ultra-violet irradiation. S. UENO, M. YAMASHITA, and Y. OTA (J. Soc. Chem. Ind. Japan, 1930, 33, 61 B).—The physical and chemical properties of hydrogenated sardine oil (m.p. 34.4—36°), herring oil (m.p. 39.8—41.3°), and cod-liver oil (m.p. 39.1—41.1°) are not appreciably affected by ultra-violet irradiation. Feeding

tests with rats indicate that the antirachitic value of the fats is considerably increased by irradiation.

C. W. SHOPPEE.

Deodorisation of fish oils. K. HASHI (J. Soc. Chem. Ind. Japan, 1930, 33, 58 B).—By polymerisation of fish oil in an atmosphere of hydrogen at 280—290° for 14 hrs. the characteristic odour of the oil disappears, and is not regenerated by hydrolysis, heating, or keeping of the product. It is found that oils deodorised by various processes can be preserved without alteration at room temperature in diffused light, but that irradiation with strong sunlight rapidly causes the products to become odorous.

C. W. SHOPPEE.

Relation between the refractive index and the iodine and saponification values of cod-liver oil. F. ENDER and A. JERMSTAD (Pharm. Zentr., 1930, 71, 193—195).—The refractive indices, iodine and saponification values of 50 samples of medicinal cod-liver oil have been determined. A low iodine value, in general, corresponds with a low refractive index, but there is no well-defined relationship between the saponif. value and refractive index. The constants determined fall within the following limits: n_D^{20} 1.4787—1.4795, iodine value 163.6—169.3, saponif. value 184.6—187.4, unsaponifiable matter 0.80—1.16. E. H. SHARPLES.

Thiocyanogen value of marine-animal oils and some of their constituents. Y. TOYAMA and T. TSUCHIYA (J. Soc. Chem. Ind. Japan, 1930, 33, 31—35 B).—The thiocyanogen values of sardine, cod-liver, herring, and sperm oils and of squalene were determined, using the reagent of Kaufmann (A., 1925, i, 1252), and indicate the presence of unsaturated acids having more than one double linking. Fairly constant values are obtained if the conditions of the experiment are constant, the mean molecular unsaturation agreeing closely with the theoretical in the case of pure unsaturated acids and alcohols.

K. V. THIMANN.

Bentonite and allied clays. WOODMAN and TAYLOR.—See XVI.

PATENTS.

Manufacture of condensation products [from oils and waxes]. A. CHWALA (G.P. 456,351, 7.4.23; cf. Austr. P. 104,727).—Thick viscous liquids or pasty solids having a high capacity for taking up water are produced by condensing rape-seed oil and carnauba wax with acetone at 90—100° under pressure, especially in presence of a little iron oxide. Other examples are: castor oil and paraformaldehyde (or acetone in presence of zinc dust); beeswax, vaseline, and formaldehyde (or acetone) in presence of finely-divided iron.

C. HOLLINS.

Production of a wax-like body. J. BAER (B.P. 300,200, 26.10.28. Switz., 8.11.27).—Alkylene halides, *e.g.*, ethylene chloride, alone or mixed with formaldehyde, acetaldehyde, etc., are treated at the ordinary or at a raised temperature with dilute ammonium sulphide solution and the product is purified by heating in the presence of alkalis. [Stat. ref.]

L. A. COLES.

Extraction of [fatty] oils. J. W. BECKMAN (B.P. 326,195, 7.9.28).—See U.S.P. 1,698,294; B., 1929, 608.

Production of sulphonation products from polymerised fats or oils or the acids thereof. H. BERTSCH, Assr. to H. T. BÖHME A.-G. (U.S.P. 1,749,463, 4.3.30. Appl., 12.9.27. Ger., 8.7.26).—See B.P. 274,104; B., 1928, 613.

Organic acids and their salts (B.P. 324,538).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Rational and automatic fine-sieving of lake, oil, and protective paint colours. ASSER (Chem. Fabr., 1930, 71—77).—The construction and operation of enclosed, self-operating, fine-sieving devices for paints are described. The apparatus consists essentially of a closed chamber divided into two compartments. In the first are coarse sieves of diminishing aperture, and in the second three or more fine sieves of the same dimensions. The latter are arranged like plates in a filter press, the paint flowing from both outer sides into the middle space, from which it leaves the apparatus. Means are provided for automatically removing skin and coarse particles arrested by the coarsest sieve in the first compartment.

S. I. LEVY.

Evaluation of the contrast-obliterating and brightening power of white pigments. G. S. HASLAM (Ind. Eng. Chem. [Anal.], 1930, 2, 69—72).—The terms "brightening power," "contrast ratio," and "contrast-obliterating power" are defined, the last-named being considered as the true definition of hiding power, avoiding confusion with tinting strength. It is shown that these two are not universally synonymous. The determination of the brightness over black glass of pigment-in-oil films of varying thickness, obtained by progressively spinning the films, is described and brightness-film thickness curves are drawn for the range "ultimate brightness" to "brightness of the black glass," from which it is shown to be possible to study the relationship between film thickness and brightness over any stated background. The results obtained with six common white pigments are given and the increase in hiding power with decrease in brightness is shown. It is considered that the value at which the eye is capable of detecting contrast is less than the generally accepted 98%.

S. S. WOOLF.

PATENTS.

Zinc white. J. M. H. CORNILLAT (F.P. 632,235, 15.7.26).—Zinc is rapidly melted in a mazout flame and oxidised in an air-blast, the zinc oxide being collected in a chamber lined with linen. C. HOLLINS.

Coating of [smooth] surfaces [with lacquers]. BRIT. CELANESE, LTD. (B.P. 298,608, 2.10.28. U.S., 12.10.27).—Coating compositions containing cellulose acetate or other organic esters or ethers of cellulose and suitable plasticisers are caused to adhere to metallic, glass, wood, or other smooth surfaces by means of an intermediate nitrocellulose lacquer film, preferably thicker than the cellulose acetate film.

S. S. WOOLF.

Refining of rosin. (A, D) I. W. HUMPHREY, (B, C, F) H. E. KAISER and R. S. HANCOCK, (E) G. M. NORMAN, Assrs. to HERCULES POWDER Co. (U.S.P. 1,715,083—8,

28.5.29. Appl., [A] 28.7.26, [B] 18.8.26, [C] 18.8.26, [D] 23.8.26, [E] 25.9.26, [F] 8.6.28. Renewed [A, D, E] 7.12.28, [B] 8.8.28).—Colouring matters are removed from rosin by dissolving the rosin in mineral oil and extracting impurities with (A) aniline, (B) furfuryl alcohol, (C) furfuraldehyde, (D) ethylene chlorohydrin, (E) liquid sulphur dioxide, (F) any suitable organic solvent, such as (A) to (D) or phenol. C. HOLLINS.

Manufacture of artificial resins by condensation of phenols with aldehydes. G. A. LACROUX (F.P. 623,535, 17.8.26).—Phenol-aldehyde resins are treated with calcium carbonate and the mixture is neutralised with lactic acid. The product may also be treated with sodium thiosulphate; it is a white substance highly resistant to alkalis. Addition of lithopone gives the resin an ivory, citric acid a mottled ivory, and calcium chloride a mother-of-pearl appearance. Admixture with lead salts renders it impenetrable to X-rays.

A. R. POWELL.

Manufacture of condensation products of phenols and formaldehyde. SOC. DES USINES CHIM. RHÔNE-POULENC, H. W. and P. O. HERREWART, and E. F. EHRHARDT (B.P. 325,861, 3.9.28. Addn. to B.P. 321,697; B., 1930, 69).—The polyhydric phenols, e.g., resorcinol, mentioned in the earlier patent are replaced by a mixture of poly- and mono-hydric phenols, with or without the addition of a catalyst.

S. S. WOOLF.

[Production of urea-formaldehyde] synthetic resin. A. V. KELLER (B.P. 326,070, 19.4.29).—Urea or a derivative is condensed with formaldehyde or paraformaldehyde in the presence of a small proportion of copper sulphate, the greenish-blue colour of the intermediate condensation product being removed or reduced by chlorination or like bleaching means. Fillers, pigments, or dyes may be added if desired.

S. S. WOOLF.

Apparatus for manufacture of zinc white. J. M. H. CORNILLAT (U.S.P. 1,751,396, 18.3.30. Appl., 9.7.27. Fr., 15.7.26).—See F.P. 632,235; preceding.

Production of permanent [pigments for] paints. N. SPECHT, Assr. to DEUTS. GASGLÜHLICHT-AUER-GES. M.B.H. (U.S.P. 1,750,287, 11.3.30. Appl., 21.10.25. Ger., 31.10.24).—See B.P. 242,282; B., 1926, 955.

Manufacture of a solid resin from the semi-fluid resinous matter extracted from crude gutta-percha and/or balata. A. B. CRAVEN (U.S.P. 1,751,724, 25.3.30. Appl., 23.7.27. U.K., 1.1.27).—See B.P. 278,922; B., 1927, 948.

Production of alkyl esters of abietic acid. A. C. JOHNSTON, Assr. to HERCULES POWDER Co. (U.S.P. 1,749,482—3, 4.3.30. Appl., [A] 18.10.27, [B] 12.6.28).—See B.P. 298,972; B., 1930, 249.

Carbon black (B.P. 324,959 and 325,207).—See II. **Titanium dioxide** (U.S.P. 1,742,674).—See VII. **Treatment of minerals** (U.S.P. 1,742,191).—See X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Factory economies in the preparation of raw rubber. B. J. EATON (Rubber Res. Inst. Malaya, 1930, 1, 232—240).—The advantages of correlating the dimensions and capacity of the coagulating tanks,

sheeting rolls, and packing cases for smoked sheet rubber are indicated. Standardisation is also desirable in the smoking process for which, in Malaya, the wood required ranges from 1 to 10 lb. per lb. of rubber. Observations are also made on possible advantageous process modifications in the production of crêpe rubber.

D. F. TWISS.

Some properties of sponge rubber. ANON. (U.S. Bur. Stand., 1929, Circ. No. 377, 8 pp.)—Thirteen samples of soft sponge rubber were examined as to weight per unit volume, hardness (load necessary for 40% compression), porosity (from shrinkage between plates with load of 4000 lb./in.²), tensile strength and elongation, permanent set after compression, buoyancy in water, hysteresis under slow compression and under impact, and thermal conductivity. A structure of closed pores was favourable to buoyancy in water and to small hysteresis loss under slow compression, but damping properties as revealed by hysteresis under impact appear to be independent of softness or hardness; for heat insulation a low percentage of solid material and the absence of the ordinary rubber fillers are desirable. Photographs are given showing the texture of the samples.

D. F. TWISS.

Swelling of [rubber] latex. F. EVERS (Kautschuk, 1930, 6, 46—50).—Whereas the addition of benzene or similar rubber solvent to richly ammoniacal latex causes coagulation to a jelly-like mass, it is possible easily to extract the rubber from ammonia-free latex by such a solvent, particularly if the latex be first diluted. This difference in behaviour is attributed to the presence of the ammonia and the consequent formation of a distensible surface film between the two liquids. This view is confirmed by ordinary and microscopical examination of the latex during the action of the solvent.

D. F. TWISS.

Sipalin as a softening agent in the rubber industry. E. WÜRM (Kautschuk, 1930, 6, 51—52; cf. Ditmar and Preusze, B., 1929, 949).—Sipalin AOM (methyl cyclohexyl adipate) is an advantageous softener for rubber; it enables the avoidance of excessive heating during mixing operations and the reduction of mastication periods.

D. F. TWISS.

Ageing of vulcanised rubber. X. Relation between degree of oxidation of vulcanised rubber and occurrence of maximum acetone extract. T. YAMAZAKI and K. OKUYAMA (J. Soc. Chem. Ind. Japan, 1930, 33, 68—72 B).—With progressive ageing of vulcanised rubber at 70° or 90° the percentage extractable by acetone attains a maximum and then decreases. This is probably caused by gradual increase in the degree of oxidation with formation of acetone-insoluble products. Samples which had been previously extracted with acetone were used for the experiments in order to expedite the ageing process, but evidence justifying this action is adduced.

D. F. TWISS.

Mathematical representation of the deformation curve of rubber. M. KRÖGER and E. MÖBIUS (Gummi-Ztg., 1930, 44, 1153—1154, 1209—1212).—Various possible methods for mathematical formulation of the stress-strain curve of well-vulcanised rubber are com-

pared, the small initial portion of the curve being disregarded.

D. F. TWISS.

Measurement of the p_H value of rubber latex. J. E. MACKAY (India-rubber J., 1930, 79, 353).—Trial of the Wulff colorimetric p_H tester with solutions of known hydrogen-ion content reveals certain ranges of p_H where the tester is not capable of giving exact results. Between p_H 6.0 and 12.0, however, which would include the values for alkaline latex, the usefulness of the tester is impaired only by a discontinuity at p_H 7.2—8.0.

D. F. TWISS.

PATENTS.

Rubber-coated fabrics and the like. IMPERIAL CHEM. INDUSTRIES, LTD., Assecs. of A. N. PARRETT (B.P. 299,321, 22.10.28. U.S., 22.10.27).—Rubberised fabrics or varnished rubberised fabrics are coated with a pyroxylin composition which has a good adhesion thereto, and has such extensibility and pliability that it will not substantially crack or peel when the material is flexed. For this purpose the pyroxylin composition contains a proportion of drying oil (blown or otherwise) at least equal to that of the pyroxylin.

D. F. TWISS.

Manufacture of rubber articles. DUNLOP RUBBER CO., LTD., and (A) G. W. TROBRIDGE, (B) D. F. TWISS, F. T. PURKIS and E. A. MURPHY (B.P. [A] 324,006, 11.10.28, and [B] 324,104, 18.10.28).—(A) Ornamental articles, comprising at least two pieces of rubber of different colours integrally united with one another, are produced by providing a mould or former with a coloured coating of an aqueous dispersion of rubber and then applying to this a coating of an aqueous rubber dispersion of another colour. Alternatively, grooves, indentations, or embossed parts on the mould may be filled or covered with at least one coloured, aqueous, rubber dispersion, and the mould is then coated with a dispersion of another colour, e.g., by dipping, spreading, or electrodeposition. In either case the whole is dried and vulcanised. (B) Concentrated rubber latex, compounded or otherwise, is gelled by heating after the addition of one or more non-coagulating substances, which on rise of temperature yield acidic coagulants for latex; such additions are ammonium persulphate, mixtures of this with trioxymethylene or ammonium thiosulphate and mixtures of ammonium or barium thiosulphate with hydrogen peroxide, salts of per-acids, or lead acetate.

D. F. TWISS.

Vulcanisation of natural or artificial rubber. I. G. FARBENIND. A.-G. (B.P. 308,275, 20.3.29. Ger., 20.3.28).—Mercaptobenzthiazoles carrying a 4-methoxyl group and preferably also a halogen substituent (e.g., 6-chloro-) are used as accelerators. The rubber shows improved properties compared with that vulcanised with the aid of mercaptobenzthiazole itself.

C. HOLLINS.

Manufacture of rubber [anti-ageing compounds]. GOODYEAR TIRE & RUBBER CO., Assecs. of A. M. CLIFFORD (B.P. 307,013, 25.1.29. U.S., 1.3.28).—Arylamino-benzyl alcohols, e.g., *p*-anilino- and *p*- α - or β -naphthyl-amino-benzyl alcohols, have anti-ageing properties.

C. HOLLINS.

Regeneration of rubber. M. GATTEFOSSÉ, and Soc. FRANÇ. DE PROD. AROMATIQUES (ANC. ETABL. GATTE-

FOSSÉ) (F.P. 636,641, 20.10.26).—The old rubber is heated with an ester of cyclohexanol, e.g., its acetate or butyrate, or with benzyl amyl ether. After removal of impurities, fillers, fibrous material, etc. by filtration or centrifuging, the rubber is precipitated by the addition of a non-solvent such as alcohol and, if necessary, is purified by dissolving in a rubber solvent.

D. F. TWISS.

Carbon black (B.P. 324,959 and 325,207). Lubricating oil (F.P. 636,242). Stabilised dispersions (B.P. 324,663).—See II.

XV.—LEATHER; GLUE.

Red discoloration of salted hides and salt stains.

M. BERGMANN (J. Soc. Leather Trades' Chem., 1929, 13, 599—611).—Salt-cured hides and skins frequently show large surfaces discoloured pink to brick-red on the flesh side. The hair is looser on the grain side of such skins than on skins which are free from such discoloration. The stains spread to other skins by contact. It is seen by microscopical examination that the layer of connective tissue containing thick deposits of fat beneath the corium in healthy skins is largely destroyed in discoloured skins and filled with bacteria, bacilli, and globulets. The damage is found on the flesh side only in the early stages, but later the epidermis may be destroyed and the hair loosened. *Sarcina lutea* and *auriantica*, *Micrococcus roseus* and *tetragenus*, varieties of *Proteus* and *Actinomyces*, and *B. subtilis* have been identified in the stained portions. *M. tetragenus* and the *Actinomyces* decompose fat and the others liquefy gelatin. A number of them show pronounced coloration. The growth of *S. auriantica* and of the *Actinomyces* is hindered by the presence of sodium chloride, but is not prevented by 2—4% of the salt, so that these two species can only develop in poorly-salted parts of the hide. Increased growth of *S. lutea* and *M. roseus* is observed with increase in salt content up to 8%, so that these develop on well-salted hides if the latter become warm and moist. The development of these bacteria is only partially checked by increasing the alkalinity to pH 9. More skin substance is lost by red, discoloured, salted calf-skins during soaking than by healthy skins. Patchy dyeing is shown by red discoloured skins owing to the altered absorptive power of the discoloured parts; the fat absorption and finishing are also affected. Small light yellow, orange, or dark blue spots have recently been observed on the grain of salted skins; usually several stains together are accompanied by hard rough contractions containing calcium phosphate and sulphate. A number of gelatin-liquefying organisms have been isolated which are present in air, soil, or water, the growth of which is not checked by the presence of 4% of salt. To prevent their growth, the hide should be washed immediately after flaying to remove blood, dung, lymph, etc., brined with salt free from iron and containing a minimum of gypsum or other sulphates, and stored in a cool room which is not too moist.

D. WOODROFFE.

Preparation of hide powder. R. TATARSKAJA (Collegium, 1929, 644—651).—Heavy hides are soaked for 24—48 hrs., limed first for 11 days in a saturated

lime liquor containing 0.1% of sodium sulphide and then for 3 days in a lime liquor alone, unhaired, split so as to remove the layers on flesh and grain sides (which contain the elastin), and the middle layer of collagen is treated with a solution of sodium chloride to remove soluble proteins. The product is then delimed with hydrochloric acid and ammonium chloride, reduced to pH 5.0—5.5 with acetic acid solution, washed, dehydrated with alcohol, and ground. To minimise the increased swelling properties, such pelt may be chromed or treated with 0.01% solutions of formaldehyde or with lead acetate before washing, dehydration, and grinding. The adsorptive properties of the powder in weak tannin solutions of analytical strength are not affected by this treatment, but they are diminished in stronger solutions. The hide powder takes up about 50% of the acid non-tans present in solutions of analytical strength. This is increased by suitable pretreatment with alkali. Vigorous shaking of the powder with alkali reduces its nitrogen content and causes partial gelatinisation. The powder must not be shaken vigorously with either acids or alkali and the temperature must not be raised.

D. WOODROFFE.

Fractional peptisation of vegetable tan liquors.

N. KOTELNIKOV and J. BASS (Collegium, 1929, 637—643).—Solutions of different tanning materials (mimosa, myrobalans, willow bark, and badan) are precipitated with 1:1, 1:2, and 1:8 solutions of sodium chloride, respectively, filtered through a Schott glass filter, and washed with the respective precipitating solution until the filtrate is free from colour and gives no reaction with ferric chloride. The precipitate is washed with hot water into a flask, and the sodium chloride, insoluble matter, and total solids are determined in it, from which figures the tannin content is deduced. Stiasny's method (B., 1924, 105) of determining the amount of tannin salted out is criticised. The smallest percentages of total tannin salted out by the 1:1 and 1:8 salt solutions were obtained with valonia, which indicates that it is the most stable tannin. By similar tests on actual tannery liquors composed of mimosa, mangrove, and quebracho extracts it is shown that the stability increases the longer the liquors have been in use. Freshly-prepared willow-bark liquors, extracted with hot water, are found to contain a large amount of very unstable tannins and consequently should not be used in the early stages of tannin without previous artificial stabilisation. Mimosa bark was extracted with three lots of liquor, and each liquor was tested by the above precipitation method. The most stable tannin was the first extracted, and was the first to be absorbed by the pelt, whilst the stability of the extracted fractions diminished in the order of their extraction. The amount precipitated by salt solutions from dry residues was greater than that from coagulates.

D. WOODROFFE.

Examples of fluorescence applied to the qualitative analysis of tannins. G. DESMURS (J. Soc. Leather Trades' Chem., 1929, 13, 616—624).—Mulhouse and calico strips each weighing 1 g. are immersed in 350 c.c. of a solution of the tanning material of analytical strength and the liquid is heated to boiling for 15 min. The Mulhouse strip is then removed, washed,

and dried, but the calico strip is left in the solution for 12 hrs., treated with a mixture of 350 c.c. of distilled water, 25 c.c. of a 0.2% solution of tartar emetic, and 25 c.c. of a 0.1% solution of sodium acetate for 15 min., washed in distilled water, and dried. The calico strip can be treated with a pure product which it is desired to identify, *e.g.*, with fustic when seeking to detect fisetin, or with a good pine-bark extract when examining the violet fluorescence. A yellow fluorescence is observed when strips treated with quebracho, tizerah, urunday, algarobbo wood, berberry, Japanese coptis, and Colombo root are examined by a Wood light, and a faint yellow with hemlock, mimosa, and true cutch. All these materials can be identified by the Mulhouse strips except quebracho and tizerah, the colours with which are confused. Violet fluorescence is shown by pine bark, malet, quassia wood, guaiacum, and quillaja bark. A bluish fluorescence was obtained with French turpentine and an alcoholic solution of colophony, garouille, canaigre, and badan. Algarobbo extract, mimosa, and cutch can be differentiated from algarobilla, malet, and gambier extracts, respectively, by the violet fluorescence manifested by the last three. Synthetic tannins derived from naphthalene or anthracene nuclei show a deep violet fluorescence, quinol a pale violet, and phloroglucinol a sky-blue. A white fluorescence is given by orchil, alkanet, a mixture of quebracho or tizerah and pine-bark extracts, mixtures of mimosa with a pyrogallol tannin or urunday.

D. WOODROFFE.

PATENTS.

Treatment of hides. D. L. LEVY (U.S.P. 1,742,514, 7.1.30. Appl., 20.5.27).—Hides are tanned, stretched on frames, tanned in the stretched condition with a liquor containing common salt, and afterwards fat-liquored in a drum with a fat-liquor containing glycerin.

D. WOODROFFE.

Tanning of animal hides. I. G. FARBEIND. A.-G., Assess. of K. DACHLAUER and C. THOMSEN (G.P. 453,477, 11.3.25).—The pelts are treated with the condensation products of one or more halogenated ketones, ethers, or aldehydes with an aromatic hydroxy-compound which have been rendered water-soluble by sulphonation or other treatment. *E.g.*, the sulphonated compounds prepared according to G.P. 453,430 may be neutralised with 10–20% sodium hydroxide or with acetic acid (in the case of those condensation products which have been made soluble by treatment with formaldehyde and alkali sulphite) and the resulting solution, if necessary, after addition of synthetic or natural tanning materials, is used for steeping the pelts.

A. R. POWELL.

Emulsion [for tanning purposes]. O. RÖHM (U.S.P. 1,751,217, 18.3.30. Appl., 5.10.27. Ger., 13.11.26).—See B.P. 280,509; B., 1928, 651.

Tanning of hides and skins. E. G. STIASNY and B. J. JALOWZER (U.S.P. 1,749,724, 4.3.30. Appl., 3.1.27. Ger., 30.11.25).—See B.P. 287,221; B., 1928, 420.

Condensation products (B.P. 321,190).—See III.

XVI.—AGRICULTURE.

Determination of the phosphoric acid requirement of soils. A. DEMOLON and G. BARBIER (Compt. rend., 1930, 190, 765–767; cf. B., 1930, 254).—The

critical equilibrium constant (number of mg. of P_2O_5 per litre which for the acid used for extraction, and a given soil, does not cause any exchange of P_2O_5 between the two phases) has been determined for 22 soils, using 1% acetic acid (by vol.) containing varying amounts of ammonium dihydrogen phosphate (*cf. loc. cit.*). The constant varies from 0.36 to 156, and those soils with a constant greater than 30 have a phosphoric acid content requisite to the needs of crops grown in them.

H. BURTON.

Emulsifying powers of bentonite and allied clays, and of clays derived from these by base exchange and by hydrolysis. R. M. WOODMAN and E. McK. TAYLOR (J. Physical Chem., 1930, 34, 299–325; cf. B., 1929, 730).—Clay-containing substances of different kinds and sources, and derivatives prepared from them by base exchange or by hydrolysis, give both water-in-oil and oil-in-water types of emulsions with fatty acids, oils containing these acids, and with phenols and hydrogenated phenols. This is considered to be due to interaction of the fatty acids etc. with sodium and calcium hydroxides resulting from hydrolysis of the clay or with decomposition products of the clay; the emulsifier is thus changed and soaps are introduced. Clays, as such, are not responsible for dual emulsification. Clays of all kinds are unsuitable as emulsifiers in the preparation of spraying emulsions containing free fatty acids, fixed oils, or phenols owing to the possible formation of the undesirable water-in-oil type of emulsion. A lignitic clay gave water-in-oil emulsions with fatty acids and fixed oils, and the suspended material was completely withdrawn from the aqueous phase.

L. S. THEOBALD.

Plant nutrition. II. Effect of manurial deficiency on the mechanical strength of barley straw. F. R. TUBBS (Ann. Bot., 1930, 44, 147–160).—Nitrogen and phosphorus deficiency results in a large increase in the strength of the lower internodes of barley straw, whilst potassium starvation decreases the strength of the lower and increases that of the middle internodes. Various morphological features of the straws have been measured and correlated with the mechanical strength.

W. O. KERMACK.

Mutual physiological replacement of calcium and potassium. H. LAGATU and L. MAUME (Compt. rend., 1930, 190, 389–391).—“Royal Kidney” potatoes grown in calcareous soil produce leaves which are richer in calcium than in potassium, even when supplied with a complete artificial manure. The predominance of calcium, not particularly marked in early May, increases until at the end of July it is nearly 500%. In potatoes grown in non-calcareous soil the potassium of the leaves predominates at the end of June, and by the middle of August is approximately equal in amount to that of the calcium. With complete artificial manuring the early predominance of potassium is increased, but falls off more rapidly until calcium again predominates.

P. G. MARSHALL.

Influence of humus substances on the growth of green plants in water cultures. C. OLSEN (Compt. rend. Trav. Lab. Carlsberg, 1930, 18, No. 1, 16 pp.).—The addition of humus extracts to water cultures of plants increased growth by the introduction of iron

compounds in a condition not precipitable at the p_H values obtaining in the experiment. Ferric citrate had a similar action. When iron was supplied to the cultures as ferric chloride precipitation of ferric hydroxide occurred in slightly acid, neutral, and alkaline conditions and plant growth was limited by the iron deficiency. The activity of "bacterised" peat (Bottomley) is ascribed to such an effect and not to the presence in it of "auximones."

A. G. POLLARD.

Magnesium and calcium requirements of the tobacco crop. W. W. GARNER, J. E. McMURTREY, JUN., J. D. BOWLING, JUN., and E. G. MOSS (J. Agric. Res., 1930, 40, 145—168).—The application of the newer highly concentrated fertilisers in place of the old bulky types containing fillers to tobacco grown in light sandy and sandy loam soils is leading to symptoms of magnesium and calcium deficiency. Addition of small amounts of magnesium salts alone to fertilisers may lead to toxic effects, preventable by adding lime. Magnesium hunger, or "sand drown," is characterised by a breakdown of both green and yellow pigments in the leaves, accompanied by a decrease in leaf size, and of dry matter and carbohydrates per unit of leaf area. A minimum of 0.25% of magnesium is required in the leaf to prevent deficiency symptoms, which on the basis of a crop of 1000 lb. of leaf and 500 lb. of stalk represents a removal of 3.75 lb. of magnesium per acre. Practical experience shows, however, that 12—18 lb. of water-soluble magnesium per acre is a useful application, and any toxic effect of magnesium salts by themselves may be prevented by the use of dolomitic limestone at the rate of 500—1000 lb. per acre.

E. HOLMES.

Influence of osmotic pressure on the growth, transpiration, and storage of ash in tobacco. E. M. KALITAEVA (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 62, 26 pp.).—Oriental tobacco plants ("Varatik") grown in water culture exhibit the maximal growth of tops when the osmotic pressure of the nutrient solution is 1.75 atm. and of the roots is 0.1 atm. The ash content of tobacco leaves increases with increase in the concentration of the nutrient solution. The optimum concentration of calcium to magnesium for the growth of the plant is 1:16. With increasing concentration of the nutrient solution the calcium content of the leaves tends to decrease, whilst the magnesium content remains approximately constant. Roots, stems, leaves, and flowers have an acid reaction. W. O. KERMACK.

Synthesis of ammonia by soil *Azotobacter*. S. WINOGRADSKY (Compt. rend., 1930, 190, 661—665).—Ammonia is evolved in appreciable quantities from cultures of soil *Azotobacter* grown on silica gel or gelose impregnated with sodium lactate or succinate (cf. Kostytshev and Ryskaltchouk, A., 1925, i, 1014). The evolution is a function of the alkalinity produced in the medium by the organism, since it is apparent only above p_H 9 and does not occur in the presence of calcium salts. Its bearing on the hypothesis of the bacterial hydrogenation of nitrogen is discussed.

J. GRANT.

Evaluation of calcareous mixtures used in agriculture. LENGLEN and DURIER (Compt. rend.,

1930, 190, 391—393).—The method is based on the solubility of pure calcium carbonate in carbonic acid. A quantity of calcareous matter equivalent to 0.20 g. CaCO_3 is agitated with 500 c.c. of water containing 0.8 g. CO_2 for 2 hrs. at 15°. After filtration the filtrate is titrated with 0.1N-sulphuric acid, using Orange III as indicator. The solubility varies within wide limits according to physical structure for particles of equal size, and enables the products to be classed as hard, semi-hard, or soft. Solubility can therefore be used as a criterion of the value of different samples.

P. G. MARSHALL.

Corrosion of lead by soils. BURNS and SALLEY.—See X.

PATENTS.

Fertiliser. H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,742,448, 7.1.30. Appl., 29.4.26).—A mixture of oxides of nitrogen (produced, e.g., by the catalytic oxidation of ammonia) with an excess of air is brought into contact, e.g., in towers, with phosphoric acid, and the mixture of nitric and phosphoric acids obtained is neutralised with ammonia; the product on evaporation yields a fertiliser of low hygroscopicity and containing, e.g., N 19.5—21.7% and P_2O_5 38.5—36.0%.

L. A. COLES.

Production of fertilisers. STOCKHOLMS SUPERFOSFAT FABRIKS AKTIEBOLAGET (B.P. 306,103, 15.2.29. Swed., 15.2.28).—Raw phosphatic material is treated with a mixture of sulphuric and nitric acids in proportion such that, on subsequent treatment of the product with ammonium carbonate, or with ammonia and carbon dioxide, the solution contains $(\text{NH}_4)_2\text{SO}_4$: NH_4NO_3 :: 1:2. If desired, part of the sulphuric acid may be replaced by an equivalent weight of ammonium sulphate, and alkali sulphates may be added to the material to prevent too high a rise in temperature. After filtration to remove the insoluble constituents, which contain a high proportion of citrate-soluble phosphates, the solution is evaporated to dryness, yielding a residue containing ammonium sulphate nitrate, $(\text{NH}_4)_2\text{SO}_4 \cdot 2\text{NH}_4\text{NO}_3$, which may be used as a fertiliser alone or in admixture with the phosphatic material.

L. A. COLES.

Manufacture of soluble phosphatic fertilisers from tricalcium phosphates. J. A. POND (B.P. 300,965, 17.11.28. N.Z., 21.11.27).—Finely-ground phosphatic material, e.g., Nauru guano, is treated below about 140° in an air-tight rotating container with sufficient sulphuric acid, d 1.36—1.7, to convert the tricalcium phosphate almost entirely into mono- and di-calcium phosphates. During the heating, which is continued until the mixture is dry, the vapours and moisture evolved are withdrawn from the container by suction and/or by the introduction of heated air under pressure.

L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

The sugar palm. J. N. MILSUM and J. H. DENNETT (Malayan Agric. J., 1929, 17, 449—453).—Two samples of juice from 7-year-old sugar palms, *Arenga saccharifera*, Labill., contained, on the average, 6% of sucrose,

0.13% of invert sugar, 0.25% of carbohydrates not sugars, 0.005% N, 0.020% of ash, and had d_{20}^{25} 1.0315 and 1.0200. E. HOLMES.

Ash of Nipa [palm] juice and its preservation, using alcohol and heat as sterilising agents and copper sulphate and lime as preservatives. J. H. DENNETT (Malayan Agric. J., 1929, 17, 437—448; cf. B., 1928, 313).—Nipa juice from cultivated palms contained 0.036% N and 0.630% of total ash as compared with 0.047% and 0.575%, respectively, in juice from indigenous palms. The ash of the former contained more potash, viz., 0.277% as against 0.183%, less sulphates and magnesia, and approximately the same amounts of phosphoric acid, chlorides, and iron, aluminium, and calcium oxides than that of the indigenous palms. Fermentation and inversion of juice is largely inhibited by washing the collecting cups with 95% alcohol containing 0.6—1% of copper acetate or sulphate, and collecting at 12-hr. intervals. Lime at the rate of about 8 g. per litre of juice is also effective. E. HOLMES.

Manufacture of sugar from nipa sap. M. L. ROXAS (Philippine J. Sci., 1929, 40, 185—229).—As a source of sugar the nipa palm (*Nypa fruticans*, Wurm.) has several advantages over the sugar cane, as the juice is obtained without milling and is free from invert sugar when it leaves the plant. The collection of the juice, however, necessitates much more field labour than the sugar cane requires, and it has not been found practicable to prevent considerable deterioration of the sap during its collection. The best preservative for the sap is a condition of strong alkalinity, and the author describes various attempts to ensure that the sap is rendered alkaline as it drips from the cut stem into the attached receptacle, which is usually a bamboo tube. The method provisionally adopted was to coat the interior of the tube with milk of lime, mixed with sodium bisulphite in some cases. The yield of sap per tree per day is about $\frac{1}{2}$ litre, and the yield per hectare of nipa swamp is about 30,000 litres in a season of 90 days. A factory working 100 tons of sap per day would require about 1000 natives collecting from 400 hectares, besides 100 men in the factory itself. In an experimental factory plant sugar of 97.4 polarisation was produced from sap containing 12.5% of sugar, after liming and carbonatation; but very heavy liming was necessary, corresponding to 27 c.c. of 0.2N-alkali per 10 c.c. of sap. With addition of a bone-char filter, about 70% of the sugar in the sap could be obtained as white sugar of 99% polarisation. It is concluded that in spite of the heavy requirements of lime (for collection and purification of the sap) and the necessity of employing wood instead of bagasse as fuel, sugar can be produced from the nipa palm at a reasonable profit. J. H. LANE.

Starch slime. SPROCKHOFF (Z. Spiritusind., 1930, 53, 78—79).—From 10 to 15% of the starch output is lost in the form of starch refuse or slime, which consists of about 50% of very minute starch granules mixed with traces of proteins, fibre, iron, bacteria, etc. The formation of the waste is chiefly due to the prolonged exposure of the slow-settling starch grains to the attack of bacteria and enzymes. By their agency the protein is converted into a gelatinous material which covers

the surface of the starch grains and prevents their rapid separation. The amount of waste may be reduced to about 2% if the separation is speeded up by replacing the settling tanks with centrifuges, and by assisting more rapid separation of the starch by treatment of the starch paste in the final stage of manufacture with sodium hypochlorite. The sodium hypochlorite, which replaces the customary alkali and acid, removes the gelatinous protein coating more rapidly and also accelerates the separation of the grains from the liquid. C. RANKEN.

Tenacity of large and small granules of potato starch. SPROCKHOFF and PARLOW (Z. Spiritusind., 1930, 53, 62—64).—The viscosity, and consequently the tenacity, of starch pastes made from potato starch with very small granules is greater than that where the starch granules are large, provided the more finely-divided starch flour has been rapidly manufactured and is fresh. The inferior tenacity of smaller-grained starch found in practice is due to the longer treatment required for its preparation and separation. Not only is a more prolonged enzymic action permitted to take place at the surface, but the smaller starch granules offer a relatively greater surface area for the attack. C. RANKEN.

PATENTS.

Ultrafine, soft, granulated sucrose sugar. B. H. VARNAU and T. B. WAYNE (U.S.P. 1,751,298, 18.3.30. Appl., 23.7.27).—See B.P. 294,800; B., 1928, 832.

Manufacture of dextrose. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,750,939, 18.3.30. Appl., 17.1.25).—See B.P. 254,729; B., 1927, 122.

Grinding of gummy materials etc. (U.S.P. 1,739,761). **Digestors** (B.P. 325,760).—See I. Alcohol from starch (Austral.P. 6187).—See XVIII. Explosive (U.S.P. 1,741,146).—See XXII.

XVIII.—FERMENTATION INDUSTRIES.

Use of potato flakes in the manufacture of alcohol. KILP (Z. Spiritusind., 1930, 53, 71).—Optimum fermentation and the highest yield of alcohol are obtained by running the potato flakes very slowly into the water heated in the mash tun to 50—55°, and by keeping the mash in rapid motion by an efficient stirrer (80—90 r.p.m.). "Balling" and the subsequent necessity to raise the mash to 78—80° are avoided. C. RANKEN.

Boulard's process for arresting fermentation at a desired stage. C. SCHWEIZER (Mitt. Geb. Lebensm. Hyg., 1929, 20, 30—34).—As found by Malvezin (B., 1927, 612), "immunisation" of grape juice against *Saccharomyces ellipsoideus* by Boulard's method (A., 1926, 867) does not protect the material against other micro-organisms. W. J. BOYD.

Fusel oil obtained in the form of vapour. B. LAMPE (Z. Spiritusind., 1930, 53, 62; cf. B., 1929, 619).—Fusel oil which has been separated in the form of vapour instead of by the usual method showed a slightly lower content of amyl alcohol. In the three samples dealt with the amyl alcohol amounted to

50.2, 49.6, and 40%, whereas from the normally separated fusel oil the maximum and minimum amounts of amyl alcohol were 64 and 50%, respectively. When the fusel oils which were separated in the form of vapour were fractionated, the fraction collected up to 90–91° was very much greater than with the normally obtained oils. In addition, none of the oils so obtained was able to satisfy the official requirements, which demand that when 100 c.c. are distilled less than 30 c.c. shall distil under 100°, less than 50 c.c. under 120°, and at least 90 c.c. under 130°.

C. RANKEN.

PATENTS.

Manufacture of malt. O. SLEEMAN (B.P. 325,495, 19.11.28).—The malt house is divided into superimposed compartments by means of several perforated floors so hinged that they permit the even dropping of the grain from floor to floor. Air may be admitted into, or exhausted from, any or all of the compartments. A water sprinkler is provided for moving over and sprinkling the grain on each of the upper floors.

C. RANKEN.

Recovery of enzymes. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 325,831, 28.11.28).—Enzymes, such as the enzymes of rubber latex, blood, milk, etc., are separated from combined albuminous substances by degrading the albumin present by means of other enzymes, such as papayotin, which do not attack the desired enzymes under the conditions of working. The p_H of the solution should be adjusted to 6.8–5.0. The products obtained may be purified by dialysis or by selective adsorption.

C. RANKEN.

Apparatus for production of alcohol from substances containing starch. N. SACHOULIS (Austral.P. 6187, 28.2.27).—Starchy material is stirred for 1 hr. with warm 1% hydrochloric acid and then sterilised in a boiler into which steam and filtered air are blown. The mash is cooled to 40° and saccharified by inoculation with a fungus culture previously grown in rice flour with added aromatic substances, e.g., sage. The mash is then fermented with yeast in a large vessel and subsequently in four small closed vessels, and the alcohol obtained by distillation and rectification.

C. RANKEN.

Denaturing agents for alcohol. I. G. FARBERIND. A.-G. (B.P. 298,617, 12.10.28. Ger., 12.10.27. Addn. to B.P. 298,611; B., 1930, 119).—Neutral substances, e.g., ethyl or acetyl sulphide, acetonitrile, ethyl thioacetate or thiobutylate, are added to alcohol in addition to the agents described previously.

L. A. COLES.

Manufacture of yeast. K. A. JACOBSEN, ASST. to STANDARD BRANDS, INC. (U.S.P. 1,752,003, 25.3.30. Appl., 28.5.28. Denm., 8.6.27).—See B.P. 291,770; B., 1929, 262.

Sterilisation of liquids (B.P. 325,796).—See XXIII.

XIX.—FOODS.

Factors influencing the value of wheat in baking. SCHRIBAUX (Compt. rend., 1930, 190, 689–691).—The value of a sample of wheat for bread-making is a function of the gluten content, and as a rough guide to this the percentage of nitrogen is determined. It is

concluded that the variety of wheat is the most important factor and the climate the next. The mode of culture, particularly when nitrogenous fertilisers are used, affects the quantity but not the quality of the crop.

II. A. PIGGOTT.

Bleaching of flour. C. SCHWEIZER (Mitt. Geb. Lebensm. Hyg., 1928, 19, 223–235).—A résumé of the literature on bleaching of flour.

W. J. BOYD.

Detection of rye flour in wheaten flour. C. SCHWEIZER (Mitt. Geb. Lebensm. Hyg., 1929, 20, 119–122).—The method of Tillmans and co-workers (B., 1929, 70) is more sensitive than that of Geilinger and Schweizer (Mitt. Geb. Lebensm. Hyg., 1925, 16, 95).

W. J. BOYD.

Comparative sucrose determinations in sweetened condensed milk. A. DÜRING (Pharm. Zentr., 1930, 71, 49–51).—Comparison of the official method (Röttger, 1926, vol. 1, 5th ed., 407) with the modified method described below showed the latter to be the more rapid, convenient, and accurate. The condensed milk (10 g.) is mixed with 60 c.c. of hot water and treated with a suspension of 1 g. of calcium oxide in a little water. The mixture is shaken, heated on the water-bath at 80° for 1 hr., cooled, and treated successively with 10 c.c. of dilute sulphuric acid (1:4), 5 c.c. of lead acetate, and 5 c.c. of saturated sodium phosphate solution. After the precipitate has settled, the liquid is filtered by suction through a porcelain funnel covered with cloth and a thin layer of asbestos, and the residue on the filter is washed until free from acid. The filtrate is made up to 200 c.c., treated with 0.5 g. of ignited charcoal powder, and again filtered. The rotation of the solution in a 200-mm. tube at 20° (measured in the Laurent polarimeter) multiplied by 15 gives the weight of sucrose in 100 g. of sample.

W. J. BOYD.

The Vieth ratio [in determination of dried milk.] F. E. NOTTBOHM (Z. Unters. Lebensm., 1929, 58, 300–310).—Analyses of numerous samples of sound milk from lowland cattle both mixed and from single animals show that the Vieth ratio, lactose : protein : ash = 13 : 9 : 2, is the correct one. Analyses of samples of condensed and dried milk confirm this. Vieth's formula is also applicable to milk throughout lactation, except for 10–14 days at the beginning and for 4 weeks at the end of the period. For milk unsound through disease or bacterial infection a more correct ratio is found to be lactose : protein = 13 : 10.

W. J. BOYD.

Milk of the silver fox. O. LAXA (Ann. Falsif., 1929, 22, 598–600).—A method is given for the determination of the important constituents of milk on 8 g. of material. The result of the analysis shows that the milk of the fox is very rich in fat (12.25%, saponif. value 208) and protein (casein 9.15%, albumin and globulin 7.89%), and contains less lactose than cows' milk. The proportion of Vieth for protein, sugar, and ash gives the values 18.8 : 4.3 : 1.6 for foxes' milk as compared with 17.7 : 5.6 : 1.6 for dogs' milk.

A. SHORE.

Biochemistry of cheese ripening. C. BARTHEL (Svensk Kem. Tidskr., 1930, 42, 28–40).—The biochemical processes which take place in cheese during its ripening are discussed. An improved method has

been devised in connexion with the nitrogen determinations used in following the changes. The cheese is mixed with quartz sand of grain size 0.3–0.1 mm. in the proportion of 1 : 2, and the mass pressed to expel the liquid portion; the nitrogen content of the expressed fluid is determined, and the result given as a percentage of the total nitrogen present in the cheese. The figures obtained give a more satisfactory series in experimental work than those resulting from the usual determination of nitrogen in an aqueous extract of the cheese. Using the above method, the effect of the salt content of cheese has been investigated, and the results are tabulated. Even small amounts of salt exert a marked influence on the hydrolysis of the paracasein; furthermore, the hydrolysis of the cheese constituents and the content of lactic acid bacteria appear to bear a direct relationship to one another.

H. F. HARWOOD.

Margarine containing egg-yolk. E. VOLLHASE, H. J. STEINBECK, and E. DANIELSEN (*Z. Unters. Lebeusm.*, 1929, **58**, 342–352).—The following modification of Fendler's method of detecting egg-yolk in margarine (*ibid.*, 1903, **6**, 977) is recommended. 200 G. of margarine are melted at 50–60° and shaken at intervals during 15 min. with 100 c.c. of 2% salt solution also at 50–60°. The aqueous layer is separated, well cooled, and filtered. The clear filtrate (50 c.c.) is shaken with 8 c.c. of ether. If liquid egg-yolk is present the ether is coloured yellow; 0.25% of yolk in margarine gives, under these conditions, a coloration in the ether equal to that of a freshly prepared aqueous 0.0005% picric acid solution. In testing for dried egg-yolk the melted margarine is filtered and the filter is cut up and extracted with ether. The insoluble material is filtered off, washed with ether, dried, and extracted with 2% salt solution. The filtered extract is shaken with 8 c.c. of ether. (The melted margarine can be extracted directly with distilled water as it already contains sufficient salt.) As before, a yellow coloration of the ether indicates the presence of egg-yolk. A biological (precipitin) method of detecting 0.25% of egg-yolk in margarine, using only 50–100 g. of sample, is described. This is equally applicable for dried or liquid yolk.

W. J. BOYD.

Bacterial detection of foreign honey. C. SCHWEIZER (*Mitt. Geb. Lebensm. Hyg.*, 1928, **19**, 117–125). Honey, although acid in reaction, is liable to bacterial infection. Unheated honey may be free from bacteria. Strong bacterial infection indicates unhygienic conditions of production. Bacterial examination gives no reliable indication of the country of origin.

W. J. BOYD.

Purification and determination of pectin. G. SPENCER (*J. Physical Chem.*, 1930, **34**, 429–434).—The purification of pectin by reprecipitation by alcohol, electrodialysis, and washing by decantation with acidified alcohol is described. The second method is the least, and the last the most, satisfactory. By the decantation method the ash content was reduced to 0.108%. An apparatus for electrodialysis is described. The chief constituents of the ash left after purification are silicon and aluminium. The determination of pectin is discussed briefly.

L. S. THEOBALD.

Experiments with heat-precipitated horse-flesh

protein as precipitogen. C. SCHWEIZER (*Mitt. Geb. Lebensm. Hyg.*, 1929, **20**, 69–76).—Although Rosenberg's method (*Centr. Bakt.*, 1924, **91**, 318) of identifying boiled horse-flesh by the precipitin reaction, using boiled material for preparation of the antigen, has a scientific basis, its adoption in food analysts' laboratories is not recommended because of the unspecific, although less intense, reactions which the antiserum gives with extracts of the boiled flesh of other animals. The Rosenberg antiserum does not give better results than the ordinary Uhlenhuth antiserum.

W. J. BOYD.

Utilisation of blood. H. LÜTHJE (*Chem.-Ztg.*, 1930, **54**, 85–86).—The simplest method of utilising blood from a small slaughterhouse is by converting it into "blood-meal" in a jacketed cylindrical dryer, for which purpose it need not be defibrinated. On a rather larger scale a better return is obtained by making "water-soluble blood-powder" by atomisation of defibrinated blood in a hot-air dryer. The product should be 95% soluble in water.

C. IRWIN.

Abnormal taste and odour of preserved foods. G. BIDAULT and G. HINARD (*Ann. Falsif.*, 1930, **23**, 30–36).—These abnormal qualities of preserved food do not depend on the material preserved, but, though they may arise from various causes, are in the majority of cases due to the bad quality of the material forming the airtight joint. The smell of petrol arises from the use of defective gums, or notably from regenerated rubber when a devulcanising process has been used, and may also be due to rubber substitutes, where petrol, heavy oil, paraffin, vaseline, etc. have been used in their production. In some cases the odour may arise from defects in fixing the cover. Insufficient tinning of the receptacle may cause blackening of certain foods and facilitates attack of the iron by acid juices. Certain indefinite odours are regarded as due to accidental contamination of containers with dirt.

D. G. HEWER.

Use of the Mojonner milk tester for the routine determination of vanillin. Towt lead number. L. V. TOWT (*J. Dairy Sci.*, 1929, **12**, 469–472).—The extract (25 c.c.) and water (10 c.c.) are evaporated at 60° to 25 c.c., the evaporation being repeated after addition of 10 c.c. of water. The residue, in an 18-g. cream-test bottle calibrated to 50 c.c., is treated with 4 drops of glacial acetic acid, shaken, then treated with 10 c.c. of standard lead acetate solution, diluted to the mark, shaken, corked, and centrifuged for 5 min. Of the clear liquid 25 c.c. are placed in a fat-flask, and extracted with 60 c.c. of ether in three portions, centrifuging as in a test for fat. The ethereal extracts are evaporated at 55°; the residue being dried over sulphuric acid and weighed. The remainder is diluted to the 45-mark, corked, shaken, centrifuged, and the level of the lead precipitate, which is proportional to the lead number, is observed.

CHEMICAL ABSTRACTS.

Hardened oils.—UENO and others.—See XII.

PATENTS.

Treatment of coffee. D. J. KENNEDY (B.P. 325,468, 30.11.28. Cf. B.P. 325,039; B., 1930, 347).—The green

berries are steeped in citric acid solution for about 72 hrs. prior to roasting.

L. A. COLES.

[Apparatus for] making bread. J. E. WILSON, S. McCONNELL, and C. BROWN (B.P. 326,358, 18.3.29).

Freezing and storing fish. GOVERNOR & CO. OF ADVENTURERS OF ENGLAND TRADING INTO HUDSON'S BAY, and C. TOWNSEND (B.P. 325,947, 16.1.29).

Saturating and filling plants [for beverages]. C. WINTERWERB (B.P. 326,410, 17.5.29).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Percolation of alcoholic extracts; rational preparation of ergot extract. H. BREDDIN (Pharm. Ztg., 1930, 75, 336—337).—A method is described for preparing concentrated extracts by percolation, which is simple, economical in solvent, and gives a better exhaustion of the drug than the official procedure. Full details are given for obtaining ergot extract by this method.

H. E. F. NOTTON.

Manufacture of diethylbarbituric acid. J. SCHWYZER (Pharm. Ztg., 1930, 75, 337—340).—Working details on a 10—20-kg. scale are given for the conversion of chloroacetic acid into ethyl malonate, ethyl diethylmalonate, and diethylbarbituric acid, and for the preparation of carbamide from calcium cyanamide.

H. E. F. NOTTON.

Standardisation and stabilisation of veratrum preparations and the hydrogen-ion concentration factor. VI. E. E. SWANSON and C. C. HARGREAVES (J. Amer. Pharm. Assoc., 1930, 19, 122—127; cf. B., 1928, 284).—The influence of the hydrogen-ion concentration factor on the stabilisation and deterioration of fluid extracts and tinctures of veratrum has been extensively studied. The activity of the preparations is controlled by the hydrogen-ion concentration and pH 4—5 is apparently the most satisfactory value for the stabilisation of the active principles. The stability rapidly decreases with lessening values of pH . The chemical assay method is unreliable, and it is recommended that the lethal mouse method should be used. Also distilled water dilutions are more reliable than alcoholic dilutions.

E. H. SHARPLES.

Reactions of novocaine. L. EKKERT (Pharm. Zentr., 1930, 71, 198).—Ten drops of a solution of 1 g. of *p*-dimethylaminobenzaldehyde in 10 c.c. of concentrated sulphuric acid are added to 0.01—0.02 g. of novocaine hydrochloride, and the mixture is carefully heated until it becomes coffee-brown. After cooling and mixing with about 5 c.c. of water, an intense yellow colour tinged with green is formed. The nitrate behaves similarly. Holocaine, stovaine, alypin, cocaine, and tropacocaine all give the brown colour on heating, but no yellow colour develops on dilution (cf. B., 1928, 836).

E. H. SHARPLES.

Preparation of atropine. DUILIUS (Chem.-Ztg., 1930, 54, 182).—Dried belladonna root is crushed finely, moistened with sodium hydroxide solution, and dried again after addition of a small quantity of solid, anhydrous sodium carbonate. The powdered product is

extracted with ether to remove fat and the residue is dissolved in 5% acetic or sulphuric acid. After filtration the atropine and hyoscyamine are precipitated with ammonia. The crude alkaloids are dissolved in dilute acid and colouring materials and resins are removed by shaking with ether; the liquid is then covered with an ether layer and potassium carbonate added slowly until precipitation commences, an excess of the solid salt is then added and the mixture vigorously shaken. The ether layer is poured off, dehydrated with solid potassium carbonate, and poured quickly through a folded paper. The filtrate is distilled until crystals begin to form, when it is cooled rapidly with agitation; the ether distillate is used for further extraction of the aqueous solution. The purified bases thus obtained are mixed with 50 c.c./kg. of pure chloroform and the mixture is heated at 116° for $\frac{1}{2}$ hr. in an oil-bath to convert the hyoscyamine into atropine. After addition of 500 g. of acetone the crystals of atropine are collected on a vacuum filter, washed with acetone, and dried. For the preparation of the sulphate, 1 kg. of the base is dissolved in 1.3 litres of absolute alcohol and the solution nearly neutralised with an ice-cold solution of 170 g. of sulphuric acid (d 1.84) in 1.3 litres of absolute alcohol. The sulphate is subsequently precipitated by the slow addition of 10 litres of ether.

A. R. POWELL.

Critical remarks on the U.S.P. pancreatin assay. F. E. WILLSON (J. Amer. Pharm. Assoc., 1930, 19, 128—129).—The end-point in the U.S.P. pancreatin assay method is not definite; precipitation is too prolonged and discordant results are obtained with different observers. In the determination of the amylolytic activity, if after the addition of the digested starch paste to the iodine solution the mixture be shaken as the end-point is approached, the blue colour disappears, and whether or not the solution is shaken has a considerable influence on the assay result. It is recommended that the mixture should not be shaken. Divergent results are obtained by using iodine solutions of different temperature, and a definite figure should be adhered to throughout; 23° is suggested.

E. H. SHARPLES.

Assay of pancreatin. F. E. WILLSON (J. Amer. Pharm. Assoc., 1930, 19, 129—132).—The following method for the determination of the tryptic activity of pancreatin is better than the U.S.P. method in that a definite end-point is obtained, it is much shorter, and more consistent results can be obtained especially with the higher-strength samples of pancreatin. Two tubes, one containing 24 c.c. of distilled water and 25 c.c. of 4% casein solution, and the other 25 c.c. of each, respectively, are heated to 55° in a water-bath. To the first is added 1 c.c. of the pancreatin solution, and the mixture digested at 55° for exactly 20 min. Meanwhile 20 c.c. of the blank are mixed with 10 c.c. of neutral 40% formaldehyde and neutralised to phenolphthalein with 0.1*N*-sodium hydroxide; 20 c.c. of the test solution are treated similarly, and the net amount (in c.c.) of 0.1*N*-alkali necessary multiplied by 100 is the percentage strength of tryptic activity. The original casein solution should be maintained at between pH 7.0 and 8.0, and it may be preserved by the addition of chloroform.

E. H. SHARPLES.

Evaluation of digitalis. A. STASIAK and B. ZBORAY (Magyar gyógyszer Társ. Értes., 1929, 5, 257—272; Chem. Zentr., 1929, ii, 1951).—A study of the frog-sinus method. A standard preparation must always be tested at the same time; for the standard preparation powdered digitalis leaves are preferred. When these precautions are observed, the frog-sinus method gives results in accord with those obtained by the 6-hr. frog method and the cat method.

A. A. ELDRIDGE.

Estimation of carbolic acid in pharmaceutical preparations. J. RAE (Pharm. J., 1930, 124, 239—240).—A colorimetric method which has been found suitable for glycerin of carbolic acid, and for carbolic acid lozenges and pills, based on the colour developed on addition of sodium nitrite and sulphuric acid, is described. Substances normally present in the preparations do not interfere, but oxidising agents must be absent.

S. I. LEVY.

Determination of hypophosphorous, phosphoric, and glycerophosphoric acids in pharmaceutical mixtures of their salts. F. E. RAURICH (Anal. Fís. Quím., 1930, 28, 160—166).—Hypophosphite is determined by adding an excess of silver nitrate to the acidified solution and weighing the precipitated silver; sodium acetate is then added to the filtrate and the precipitate of silver phosphate, corresponding to the phosphate originally present and to the oxidised hypophosphite, is separated and weighed. Sucrose, if present in the preparation, is destroyed by fuming nitric acid, sulphuric acid, potassium permanganate, and hydrogen peroxide, and finally the glycerophosphate is determined by the usual molybdate method. Iodometric methods are not applicable on account of the organic material usually present, which may absorb a considerable quantity of iodine.

H. F. GILLBE.

Loss of volatile products during the curing and fermentation of tobacco. S. M. PETRIK (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 63, 24 pp.).—The loss of methyl alcohol during the fermentation and curing of yellow cigarette tobaccos (*Platana Arcadia* and *Tykkulak*) is less than in the case of cigar tobaccos. The longer the period of starving, the greater is the loss of methyl alcohol during the whole process, but the less is the loss during the actual fermentation. The loss of methyl alcohol during the fermentation of high-grade industrial tobaccos is very small as they are thoroughly starved. The methoxyl content of pectin obtained from *Platana Arcadia* and *Tykkulak* was 4.98% and 6.62%, respectively. The pectin is the chief source of methyl alcohol in tobacco. The air in the first fermentation warehouse in Krasnodar contained methyl alcohol, volatile oily products of unknown nature, and carbon monoxide, but no nicotine nor other volatile bases.

W. O. KERMACK.

Oil of frankincense. A. BLUMANN and L. SCHULZ (Annalen, 1930, 478, 303—306).—By treating the high-boiling fraction of oil of frankincense with sodium sulphite and bicarbonate, a ketone, $[\alpha]_D +172.4^\circ$, has been isolated which is identified as a mixture of *d*- and *l*-verbenone, since it has the same physical constants and yields an identical semicarbazone. Further frac-

tionation of the non-aldehydic portion, followed by separation of the alcohols by distillation with boric acid, does not yield verbenol, which is decomposed, but its presence is definitely shown by the isolation of verbenene dibromide by bromination of the mixture of hydrocarbons distilled from the borates. It is concluded that the olibanol of Fromm and Autin (A., 1914, i, 70) is a mixture of verbenone, verbenol, and other terpene alcohols. Indications have, however, been obtained of the presence of the olibanol of Haensel (A., 1908, i, 665) in the highest-boiling fraction of the alcohols.

R. K. CALLOW.

Colour reactions of essential oils. L. EKKERT (Pharm. Zentr., 1930, 71, 195—198; cf. B., 1927, 859).—Colour reactions of 62 essential oils mixed with alcoholic solutions of various phenols and "layered" with concentrated sulphuric acid are given. E. H. SHARPLES.

Tobacco-seed oil. PIATNITZKI.—See XII. **Growth, transpiration, and storage of ash in tobacco.** KALITAIEVA.—See XVI. **Determination of vanillin.** TOWT.—See XIX.

PATENTS.

Manufacture of pinene hydrochloride and synthetic camphor. W. A. GAWALOWSKI and L. S. SACHAROV (F.P. 597,063, 17.4.25).—Pinene, b.p. 155—162°, is treated with hydrogen chloride at 0—20°, and the hydrochloride is oxidised at 170—180° and 15 atm. pressure with oxides or peroxides of lead, manganese, nickel, cobalt, zinc, or tin, or especially "ferric acid" (FeO_2 or FeO_3), in presence of sodium carbonate or bicarbonate, lime, or acetic acid.

C. HOLLINS.

Manufacture of pinene hydrochloride and of camphor therefrom. SOC. D'ETUDES POUR LE CAMPHRE DE SYNTHÈSE (F.P. 621,398—9, 13.1.26).—(A) Dry hydrogen chloride is led over the surface of turpentine oil. (B) The hydrochloride crystals which sink to the bottom of the vessel are treated with milk of lime and the resulting camphene is oxidised with air. The camphor is freed from traces of chlorine by heating with caustic alkali at 150°.

C. HOLLINS.

Manufacture of camphor from turpentine oil. A. ADIDA (F.P. 606,748, 20.11.25).—*d*- α -Pinene is heated with picric acid in benzene at 120° for $\frac{1}{2}$ hr.; the resulting bornyl picrate is hydrolysed with caustic alkali and oxidised to camphor. Hydrolysis of the picrate from the mother-liquors gives fenchol and borneol.

C. HOLLINS.

Production of $\alpha\delta$ -diamino- β -ketobutane [*n*-butanone] dihydrochloride and 2-thiol-4(5)- β -aminoethylglyoxaline. BOOTS PURE DRUG CO., LTD., and F. L. PYMAN (B.P. 325,151, 22.3.29).— $\alpha\delta$ -Dibenzamidobutan- β -one is hydrolysed with aqueous alcoholic hydrochloric acid at 150° under pressure, and the diamine dihydrochloride, m.p. 221° (decomp.; picrate, m.p. 212°), is condensed with sodium thiocyanate (1 mol.) to give 2-thiol-4(5)- β -aminoethylglyoxaline [hydrochloride, m.p. 248—249° (decomp.); picrate, m.p. 225° (decomp.)], which may be converted by oxidation with ferric chloride into histamine.

C. HOLLINS.

Thiazine derivatives [antiparasitics]. E. ROTHLIN and F. MÜLLER, Assrs. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,737,475, 26.11.29. Appl., 16.12.27. Switz., 29.12.26).—Thiazines, *e.g.*, tetramethyldiaminophenthiazine, are converted into their bile acid salts, especially cholates, which have antiparasitic properties. C. HOLLINS.

Manufacture of organic mercury compounds [mercurisation of benzene]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,266, 15.11.28).—Mercuric acetate is heated under reflux with benzene in the presence of a non-reacting solvent, *e.g.*, nitrobenzene, *o*-dichlorobenzene, in quantity sufficient to maintain the b.p. at 130—135°; when the addition of sodium hydroxide to a sample no longer precipitates mercuric oxide, the solution is filtered and concentrated *in vacuo*. After crystallisation from water, the product, phenylmercuric acetate, has m.p. 148° (cf. Dimroth, B., 1898, 1173). L. A. COLES.

Production of serum for prevention and cure of tuberculosis. REISCHACH & Co., G.M.B.H. (B.P. 297,675, 17.9.28. Ger., 24.9.27).—The caseous degeneration tissues of cattle etc. are extracted and sterilised with a mixture of solutions containing 10 g. of sodium chloride in 100 c.c. of glycerin and 0.1 g. of Trypaflavine in 100 c.c. of 1% phenol solution; after four days the mixture is filtered through material which does not allow the germs to pass, *e.g.*, compressed asbestos paper, and the residue is dried and ground. The filtrate, after further filtration, may be used for preventive inoculation, or a mixture of it with the dried residue may be used for the inoculation of animals for the production of lymph, the local inflammation set up being maintained by the application of suction cups. L. A. COLES.

Surgical dressings, bandages, etc. BRIT. CELANESE, LTD. (B.P. 307,459, 6.3.29. U.S., 8.3.28).—A pad of raw cotton or fluffed cellulose acetate is enclosed in a fabric of cellulose acetate; other cellulose esters or ethers may be used. L. A. COLES.

Cigarette papers (B.P. 325,125).—See V.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Preparation of fine-grain plates. M. N. LEROY (Brit. J. Phot., 1930, 77, 125).—A mixture of 3 c.c. of a 10% solution of colloidal silver with 50 c.c. of a 5% gelatin solution is coated evenly upon glass and dried. The colloidal silver is converted into silver chloride by bathing in a solution containing sodium chloride 2 g., copper sulphate 2 g., water 100 c.c., and the plates are washed and redried. Bromide or iodide plates may be prepared by substituting potassium bromide or iodide for sodium chloride, but in the preparation of the iodising bath the cuprous iodide first formed must be removed by filtration. The sensitivity of the plates may be increased by bathing in a 0.02% solution of silver nitrate followed by rinsing and drying. The following developer is recommended for use with the plates: potassium bromide

7.5 g., diaminophenol hydrochloride 3 g., sodium sulphite (anhyd.) 40 g., water 1000 c.c.

J. W. GLASSETT.

Action of chromic acid on the sensitivity of solarised silver bromide plates. A. P. H. TRIVELLI and R. P. LOVELAND (J. Franklin Inst., 1929, 208, 731—766).—The desensitising curve representing the action of chromic acid on pure bromide emulsions in the solarisation region is irregular in form. This is found to be due to an abnormal distribution of silver in the developed grains of the untreated emulsion, the relative weight of silver increasing with decreasing density in the first reversal of the solarisation. By using silver weights instead of densities, the curves become simpler and show in the over-exposure region an increase and a second decrease of sensitivity after treatment with acid. After treatment, development of the grains of the emulsion proceeds in the usual way, the amount of silver being approximately proportional to the density. These results are discussed in relation to the view that the action of chromic acid on the sensitivity of photographic emulsions is constituted of a slow action on the silver halide, an action on the silver and silver sulphide of the sensitivity specks at different rates, and an action on the gelatin as a result of which a new sensitiser is produced and destroyed.

R. CUTHILL.

Desensitising and the latent image. LÜPPO-CRAMER (Brit. J. Phot., 1930, 77, 151—153).—It is shown that the latent image on exposed plates treated with desensitisers is readily destroyed by exposure to red light, and the suggestion is made that this destructive action of the red light is the responsible factor in many observed cases of latent-image destruction by desensitisers, the action of the desensitisers themselves being negligible. J. W. GLASSETT.

PATENTS.

[Photographically] transferring subjects to metal [zinc or copper] surfaces. H. L. KREBS (U.S.P. 1,742,710, 7.1.30. Appl., 29.3.28).—A silver print bleached by treatment with a solution containing potassium ferricyanide and, if desired, potassium oxalate or bromide, or lead nitrate and nitric acid if the image is to be transferred to copper, is applied to a sheet or roll of the metal and the back of the print is moistened with dilute ammonia or concentrated ammonium carbonate solution. The print is withdrawn after about 1 min., leaving an image which can be used, *e.g.*, as a foundation for etching or for the application of acid-proof inks. L. A. COLES.

Production of photographic images. E. GAY (U.S.P. 1,751,318, 18.3.30. Appl., 14.10.26. Fr., 23.10.25).—See B.P. 283,274; B., 1928, 213.

[Multi-colour filter] for colour photography and cinematography. C. NORDMANN (B.P. 309,504, 11.4.29. Fr., 11.4.28).

Ascertaining or comparing the density or translucency of photographic images and apparatus therefor. C. E. BELL and L. G. SCOTT (B.P. 326,201, 7.11.28).

Colorimeter (B.P. 324,351).—See I. **Decorative glass** (B.P. 325,924).—See VIII.

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal [explosive] powders. H. MURAOUR and G. AUNIS (Compt. rend., 1930, 190, 485—488; cf. A., 1928, 141, 770).—From the results of experiments carried out with a powder free from solvent and containing 13% N, in a bomb of 150 c.c. capacity, giving rise to pressures of 54—4310 kg./cm.², *K* (cf. B., 1926, 722) is calculated. Defining the intensity of combustion as $1/K$, this is shown to be a linear function of the inverse of the maximum pressure (save for the smallest charge). For a pressure of 70 kg./cm.² it is about double that for one of about 2000 kg./cm.²

C. A. SILBERRAD.

PATENTS.

Explosive. L. W. BABCOCK, Assr. to HERCULES POWDER Co. (U.S.P. 1,741,146, 31.12.29. Appl., 4.5.27).—Bagasse, comprising the pith and fibre of sugar cane, is ground to a desired fineness and boiled in water, so as to lower its density without increasing its absorbency. The treated product is used as a constituent of explosives, which thereby acquire a low density. In dynamite, in which the carbonaceous material, consisting of 6% of unboiled bagasse and 4% of coarse oat hulls, was replaced by 6% of boiled bagasse, the number of cartridges per 100 lb. was increased from 330 to 351, whilst with 8% of boiled bagasse the cartridge count was 365 per 100 lb.

W. J. WRIGHT.

Treatment of nitrocellulose. IMPERIAL CHEM. INDUSTRIES, LTD., and J. S. B. FLEMING (B.P. 325,254, 16.10.28).—The water-absorbent power of normal butyl alcohol, used for dehydrating wet nitrocellulose and wetting it to make it safe for transport, is increased by the addition of a small amount of ethyl alcohol; at 20°, 10% of the latter solvent increases the absorbency of the butyl alcohol from 23 to 30%, which is found to be sufficient in practice. A higher percentage of ethyl alcohol is undesirable when the nitrocellulose is used for the manufacture of lacquers, as it lowers the flash point too much.

W. J. WRIGHT.

Manufacture of cordeau. W. O. SNELLING and C. B. KOCH, Assrs. to TROJAN POWDER Co. (U.S.P. 1,741,380, 31.12.29. Appl., 30.9.25).—After a lead tube has been filled with molten trinitrotoluene, and the latter allowed to cool, the tube instead of being rolled or drawn is subjected over its entire circumference to the action of rapidly moving hammers, whereby its length is increased and its diameter decreased. The temperature of the trinitrotoluene must be kept below its m.p. during the operation, a current of air being directed against the tube. By this method the trinitrotoluene is reduced to a finer state of division and consequently becomes more sensitive, and the cordeau is smoother and stronger than when drawn or rolled.

W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Foaming and sludge digestion in Imhoff tanks. W. D. HATFIELD (Ind. Eng. Chem., 1930, 22,

172—174).—Foaming always occurred in Imhoff tanks at Decatur, Illinois (where the sewage is abnormal owing to the presence of a large proportion of starch-wastes), shortly after the plant had been recommissioned after a stoppage. Addition of lime repressed, but did not prevent, the tendency to foam, and the amount required when much sludge was present was prohibitive on the ground of cost. Once the foaming ceased there was no recurrence so long as the plant remained in commission. The digestion of fresh solids proceeds very rapidly during the first 24 hrs., probably owing to the high sewage temperature (20—32°) and the nature of the trade-waste present.

C. JERSON.

New chlorine-silver method of disinfecting [water] in combination with the chlorine-copper method. W. OLSZEWSKI (Pharm. Zentr., 1930, 71, 161—162).—The quantities of chlorine required and of silver dissolved are greatly diminished if cupric chloride be added to the water.

S. I. LEVY.

PATENTS.

Sterilisation of liquids. G. A. KRAUSE (B.P. 325,796, 21.11.28).—Water which has been partially sterilised by contact with oligodynamic metals becomes further sterilised on being kept for several hours out of contact with such substances. Untreated, non-sterile water when mixed with partially or completely sterilised water from this process becomes partially sterilised on keeping and may then receive a final contact to complete the sterilisation process. In this way the volume of water dealt with by a definite amount of the active metal may be substantially increased. Suitable apparatus is described and its application in the brewing industry is indicated (cf. B.P. 325,004; B., 1930, 350).

C. JERSON.

Purifying aqueous liquids, particularly waste water from factories. METALLGES. A.-G. (B.P. 305,657, 24.1.29. Ger., 9.2.28).—Waste water (e.g., from coking installations) containing products which it is desirable to recover may be treated with a suitable adsorption media, e.g., active carbon, and the adsorbed material extracted therefrom and subsequently separated by means of a solvent which is separable from water. The adsorption media is freed from traces of solvent and re-activated in any suitable manner, e.g., by scavenging with live steam. The process may be carried out in filters containing media to a depth of a metre or more and which are charged in an upward direction, whilst the extraction and re-activation are effected in a downward direction.

C. JERSON.

Sterilisation of water. O. and R. ADLER (U.S.P. 1,750,561, 11.3.30. Appl., 11.6.28. Czechoslov., 13.6.27).—See B.P. 292,099; B., 1928, 876.

Biological purification of waste water. F. SIMMER, Assr. to A./S. DANSK GAERINGS-IND. (U.S.P. 1,751,459, 18.3.30. Appl., 13.1.28. Austr., 2.3.26).—See B.P. 307,587; B., 1929, 418.

[Valve for] base-exchange water-softening apparatus. O. OLSSON (B.P. 325,886, 3.12.28).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 23 and 30, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Preheaters with curved tubes. F. HORNING (Chem. Fabr., 1930, 133—134).—Several types of preheater or economiser are described in which the tubes are bent on an arc of considerable radius. By curving the ends of the vessel or by similar means each tube end is at right angles to the outer shell. Difficulties caused by the thermal expansion of straight tubes are thus avoided, and the risk of fracture of a tube following on pitting, which may easily occur in the preheating of solutions, is reduced. C. IRWIN.

Prevention of boiler scale. R. ESCOURROU (Chim. et Ind., 1930, 23, 273—293).—Colloids available for use as boiler disinfectants may be classified as (i) those having a hydrated or liquid dispersed phase, such as starch, dextrin, tanning extracts, or soaps; (ii) non-hydrated colloids such as graphite. The last-named substance adsorbs the crystal nuclei and, because of its high conductivity, gives a mud which offers no risk of overheating. The action of various disinfectants of the first class was studied in a laboratory boiler of 2-litre capacity. A hard feed-water was used, and photomicrographs of scale formation with and without disinfectants were taken. Various tanning extracts and cellulose extracts are shown to be effective. Cellulose extracts in some cases tend to give acid vapours, and entrained colloidal sulphur may occur in the condensed water. Synthetic tannins are too acid for practical use. Sugars have no effect whatever. Sodium sulphuricinate and resinate destroy scale, but have certain disadvantages. The usual quantity of tanning extract recommended is an initial dose of 1—2 kg./m.³ and then 0.2—0.3 kg./m.³ per day. The optimum dose for a given water can be determined on an experimental boiler. Tests with varying p_H values for one disinfectant showed best results at neutrality. A number of miscellaneous commercial disinfectants were similarly tested. Disinfectants should be introduced directly into the boiler, not into the feed-water store. It is pointed out that the action of some disinfectants is greatly modified by increase of pressure and other variables and that, although the experimental boiler gives speedy information regarding scale prevention, such tests offer no security against possible corrosion. C. IRWIN.

Derivation of Dühring's rule. A. McL. WHITE (Ind. Eng. Chem., 1930, 22, 230—232).—Dühring's rule, which states that $(T_a - T'_a)/(T_b - T'_b) = m$, when the temperatures are those of equal vapour pressure, has been hitherto considered to be empirical. The Clausius-Clapeyron equation states that $d \log p / dT = \Delta H / RT^2$, where p = vapour pressure, T =

absolute temperature, and ΔH = heat of vaporisation. It may be integrated if approximations are made and the relation $(1/T_a - 1/T'_a)/(1/T_b - 1/T'_b) = n = \Delta H_b / \Delta H_a$ is derived, a and b being two different substances, the vapour pressures of which at T_a and T_b are equal. Combining this equation with the Dühring one, the slope of the Dühring line m can be calculated if the vapour pressures and heats of vaporisation of the two substances are known. Examples show that so long as the Clausius equation holds good this method of calculation gives results in agreement with experiment. The reverse process of calculation of heats of vaporisation from the slope of the Dühring line is also possible. C. IRWIN.

Nomograms for technical gas analysis. E. SCHWARZ VON BERGKAMPF (Z. anal. Chem., 1930, 80, 103—108).—The construction of nomograms for the calculation of the volume of a gas at N.T.P. and for calculating the pressure in the correction tube of a Jeller apparatus is described. A. R. POWELL.

Fractionating columns. CHILLAS and WEIR.—See II. **Insulating board from straw.** GIBSON.—See V. **Removal of incrustation from evaporator and heater tubes.** SPRINGER.—See XVII. **Metafiltration and brewing.** PICKARD.—See XVIII.

See also A., Apr., 447, **Rapid evaporation** (NAUMANN). **Vacuum-distillation apparatus** (FRIEDRICHS).

PATENTS.

Heat exchanger or condenser. BIRMINGHAM ALUMINIUM CASTING (1903) Co., LTD., and P. PRITCHARD (B.P. 326,278, 31.12.28).—A single baffling and dividing wall is formed as a corrugated cylinder, and is sandwiched between cylindrical walls which make contact with the crests of the corrugations, thus producing helical inner and outer passages for the two fluids.

B. M. VENABLES.

Heat-transferring devices such as air preheaters. W. A. WHITE (B.P. 326,381, 11.4.29. Addn. to B.P. 313,998; B., 1929, 739).—Means are described for securing in groups the unit heaters described in the prior patent.

B. M. VENABLES.

Drying and heating apparatus. H. KÜPPERS (B.P. 326,269, 19.12.28).—A flame thrower suitable for drying moulds or foundry ladles is described.

B. M. VENABLES.

Apparatus for liquation [of sulphur etc.]. J. THAME and W. E. LANGTON (B.P. 325,948, 16.1.29).—The material is conveyed on a perforated belt or chain of trays through a heated chamber. The fine solid matter that drops through the perforations with the

* The remainder of this set of Abstracts will appear in next week's issue.

liquid is separated from the latter on a rifled concentrating table underneath the conveyor.

B. M. VENABLES.

Means for the control of temperature. ELECTRO-FLO METERS Co., LTD. From REPUBLIC FLOW METERS Co. (B.P. 326,309, 13.2.29).—A purely mechanical gear for controlling oil burners, electric rheostats, etc. is described. The primary control is of the hit-and-miss type, and is sufficiently gentle to be operated by the pointer of a moving-coil instrument, which is clamped during the actual "hit."

B. M. VENABLES.

[Optical pyrometer for] measurement of high temperatures, specially of incandescent bodies. A. DORNFRIED, E. KARCHER, and A. SINGLE (B.P. 325,863, 28.9.28).—A filter of coloured glass or other material is graduated from a minimum density at one end to a maximum at the other, it is placed within a frame provided with a scale, and the incandescent body is observed through the filter by means of a rider provided with a collecting lens, which is moved to a position where the body is just invisible.

B. M. VENABLES.

Fine-grinding of dry material. C. PONTOPPIDAN (B.P. 326,366, 21.3.29).—In the fine-grinding of cement or the materials thereof it is found that in the later stages the material adheres to the grinding bodies, owing to frictional electricity, and prevents further reduction. To prevent this, 0.5–10% of one or more materials such as coal, coke, cinder, ore, or peat is added, or an atmosphere of, e.g., superheated steam or carbon dioxide may be provided. The effect of the added substance is to produce a *P.D.* which reduces that set up by the material.

B. M. VENABLES.

Apparatus for treatment of granular or pulverulent materials with gases. S. WRIGHT (B.P. 326,085, 3.5.29).—In an apparatus of the type where the gases rise against a stream of the material which is agitated and the fall is hindered by rotating retarders, the last-mentioned are made Z-shaped in section.

B. M. VENABLES.

Filtering apparatus. E. W. W. KEENE (B.P. 326,221, 8.11.28).—In a rotary drum filter the sections of cloth are supported on frames which form shallow filtrate compartments in conjunction with the impervious drum, and the openings for filtrate leading to the outlet channels come under the channel-shaped rims of the frames.

B. M. VENABLES.

Filtration of water or liquid. R. H. L. PENNELL and A. W. WYLIE (B.P. 325,937, 5.1.29).—A filter of the sand or gravel type, preferably operating with upward flow, is washed by a reverse current which is accelerated compared with the rate of filtration. In the case of a closed filter the rush of wash liquid is produced by the pressure of air trapped above the filter bed; in an open filter the wash-water outlet pipe is extended downwards as far as possible to produce a barometric suction.

B. M. VENABLES.

Centrifugal separators. G. JUNGBLUTT (B.P. 312,271, 14.5.29. Fr., 23.5.28).—A fluid from which a solid or liquid is to be separated is passed axially through a rotor which is surrounded by a fluid (preferably the

same) substantially in hydrostatic equilibrium. The material to be separated and some of the fluid (e.g., dust and air) pass through the circumference of the rotor to a settling device, or to an auxiliary separator such as a cyclone, from which the air with a little residual fine dust may be returned to the inlet of the rotor.

B. M. VENABLES.

Centrifugal sludge separator. A. NYROP (B.P. 312,997, 31.5.29. Denm., 4.6.28).—The separator bowl is in three parts, the upper part being single and rigidly connected to the separating discs or filters and to either the inner or outer part of the lower double bowl. The two lower walls slide on each other in a liquid-tight manner so that a circumferential sludge outlet between the upper and lower halves may be opened and closed. If the inner part is coupled to the upper, the sludge outlet is held closed by springs and hydraulic pressure is used to open it. The pressure is generated between the lower bowls by separated liquid overflowing inwards through passages near the axis, which the liquid reaches only when sludge has accumulated; the effect may be aided by an external supply of liquid through an axial passage. When the sludge discharges, a slight additional sliding movement uncovers discharge ports for the hydraulic liquor so that the bowl rapidly shuts again. If the outer part of the lower bowl is coupled to the upper, the sludge outlet is closed by the hydraulic pressure of an externally supplied liquid and opened by gravity, and when the separated liquid overflows inwards it causes an outlet for the other liquid to open, releases the pressure, and allows the bowl to open. An automatic gear is described for controlling the supply of feed and hydraulic liquor.

B. M. VENABLES.

Heating of liquids, using the sun's rays for the purpose. J. A. THOMSON (B.P. 325,928, 31.12.28).—The heat-absorbing element comprises a pair of thin, corrugated plates which are maintained a short distance apart by wires or metal strips which also serve the purpose of causing the internal liquid to take a sinuous path. The element is placed in a vertical or inclined position to catch the sun's rays, is preferably connected in closed circuit with a storage tank, and may be enclosed in a glass hut to minimise the loss by convection.

B. M. VENABLES.

Apparatus for cleaning articles by solvent vapour. CARRIER ENG. Co., LTD., and K. J. R. ROBERTSON (B.P. 326,299, 30.1. and 2.3.29).—The articles are conveyed through a bath of solvent vapour by means which makes a knee-like bend at both entrance and exit, under canopies in which "air cushions" are maintained to isolate the vapour from the ambient air; the escape of vapour is further prevented by refrigerating coils near the air cushions.

B. M. VENABLES.

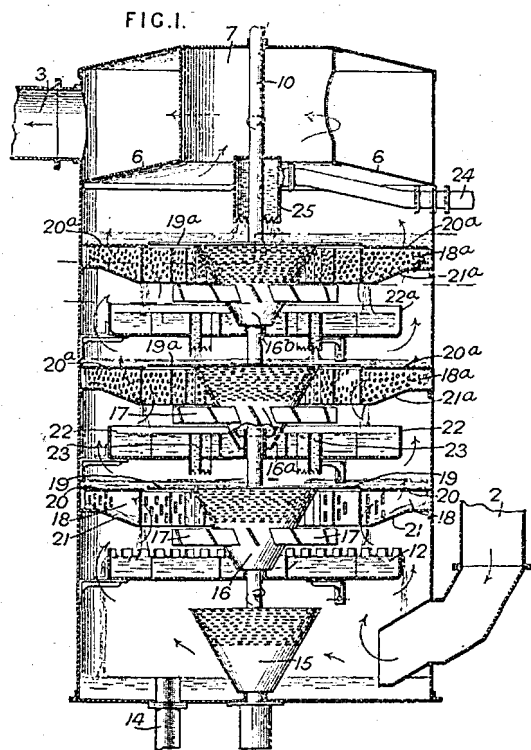
Condensation of vapours. I. G. FARBENIND. A.-G. (B.P. 301,430, 29.11.28. Ger., 29.11.27).—An apparatus which effects silently the condensation of steam or other vapour in a liquid comprises a number of downwardly directed jets of vapour surrounded by annular openings through which the liquid flows in the same direction. The downward flow tends to prevent detachment of vapour bubbles, with consequent noise

when they condense, and their formation is entirely prevented by immediately dispersing the mixture among Raschig rings, gravel, or other filling.

B. M. VENABLES.

Utilising the waste gases from furnaces. CARBORUNDUM CO., LTD. From CARBORUNDUM CO. (B.P. 326,205, 3.12.28).—The waste gases from a tunnel kiln are taken partly through a dryer and partly through an air preheater for the kiln. The goods in the dryer, *e.g.*, porcelain ware, are not in contact with the gases, but the latter are supplied to a lowermost distribution chamber, and thence, through adjustable openings in the ceiling, to an intermediate chamber with an impervious metal roof, through which the heat is conducted to the goods chamber at the top. B. M. VENABLES.

[Countercurrent] gas washers. C. A. JENSEN. From BARTLETT HAYWARD CO. (B.P. 325,300, 21.11.28).—The gas passes upwards and the liquid downwards through a tower as shown in the figure. The liquid is lifted from the trays and sprayed by the rotating



perforated cones, 15–16b, into the pockets, 18 and 19a, which are provided with perforated division plates about tangential to the rotating inclined blades, 17, by which the gas is projected into the pockets. The blades forming the pockets have, at the bottom, open centres and closed outer parts, and at the top, perforated outer parts and centres closed by the rotating discs, 19 and 19a, which also serve to spray the descending liquid. B. M. VENABLES.

Air washing and the like. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 325,884, 3.12.28).—The air is forced into the lower part of a tank of water, and while rising is caused to travel in a zig-zag manner by horizontal or sloping baffles, all of

which are drowned. Inclined louvre-like baffles are provided at the points where the gas passes round the edges of the main baffles. B. M. VENABLES.

Air filters. C. G. VOKES (B.P. 326,047, 28.3.29).—A supporting spider is described for a filter composed of pleated fabric. B. M. VENABLES.

Refrigerating apparatus. LIVERPOOL REFRIGERATION CO., LTD., and J. ALLAN (B.P. 326,827–8, 20.11.28).

Absorption refrigerating apparatus. ELECTROLUX, LTD. From PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 326,965, 22.2.29).

Absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 308,793, 11.3.29. Ger., 28.3.28).

Utilisation of waste liquors (B.P. 326,112).—See II. **Continuous kilns** (B.P. 309,547).—See VIII. **Boiler-feed water** (Austr.P. 109,459).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Chemical and microscopical investigations of the coals from the Witbank and Ermelo coal fields. P. E. HALL (J. Chem. Met. Soc. S. Africa, 1930, 30, 195–219).—Proximate and ultimate analyses, and the results of additional coking tests, of 22 samples of coal have been recorded; four of the coals were subjected to benzene pressure extractions, and thin sections of six were prepared and examined microscopically. The Witbank coals may be divided into three groups: (a) a non-banded, tough, homogeneous coal, composed of vegetable tissues which have suffered a partial aerobic decay with the formation of a “fusainised” type of material; (b) a finely-banded, striated coal with good caking qualities; and (c) a less strongly caking, banded coal with wider bright bands. The Ermelo coals are of a less mature nature and are non-caking. None of the coals contained any durain. They are all suitable for steam raising, and some for gas manufacture. Their high ash content, of at least 9%, and the unfavourable nature of the ash makes them unsuitable for the production of blast-furnace coke. A. B. MANNING.

Comparative tests of various fuels when burned in a domestic hot-water boiler. E. S. MALLOCH and C. E. BALTZER (Canada Dept. Mines Rept. No. 705, 1929, 92 pp.).—A number of fuels have been tested in the furnace of an experimental heating installation of the hot-water type, and of a size suitable for a house of eight or nine rooms. Three different rates of combustion were used; most of the tests were continued until 1000 lb. of fuel had been consumed, and an endeavour was made to keep all errors within the limit of 2%. The plant and method of making observations are described in detail. The fuels may be divided into three groups: (1) anthracites and cokes of 75–92% fixed carbon content, (2) semi-bituminous coals of 70–74% fixed carbon content, and (3) Alberta sub-bituminous and domestic coals of 40–51% fixed carbon content. One test was carried out with air-dried peat, and one with Welsh briquettes. The average thermal efficiencies obtained with the fuels of each of these groups were 72.9%, 65.8% and 60.8%, respectively. The quantities of fuel fired per therm delivered to the

water averaged 10.78 and 11.07 lb. for groups (1) and (2), respectively; for group (3) the figures varied from 14.77 to 19.06 lb. The furnace required little attention when burning fuels of group (1), considerably more with those of group (2), and more again with those of group (3). Of the 21 fuels tested Welsh anthracite was the most economical.

A. B. MANNING.

Role of the catalyst in the processing of coal. H. S. TAYLOR (Proc. II. Int. Conf. Bit. Coal, 1928, 1, 190—199).—"Oxide" catalysts (potassium carbonate, zinc, tungstic, or chromic oxide, etc.) are preferred to metallic hydrogenation catalysts on account of the presence of sulphide poisons. Thorium, aluminium, selenium, titanium, and zirconium oxides are good hydration-dehydration catalysts. The presence of small quantities of alkali is advantageous. Phenols inhibit the polymerisation of hydrocarbons; lead tetraethyl and iron carbonyl inhibit oxidation.

CHEMICAL ABSTRACTS.

Chlorination of coal. S. OCHI (J. Fuel Soc. Japan, 1930, 9, 27—29).—Treatment of coal with saturated chlorine water removes considerable amounts of the sulphur present, inorganic sulphur being the most affected. The reaction is facilitated by fineness of size of the coal, is almost unaffected by pressure, and conforms with an optimum temperature of 25°. The total sulphur content of Miike coal was reduced by chlorination from 3.73% to 1.36%. Treatment with chlorine water also reduces the amount of ash, especially acid-soluble ash. Miike coal which had been extracted with benzene yielded further benzene extracts after chlorination. These extracts can be separated into portions soluble or insoluble in ethyl alcohol. Ibaraki coal (of younger geological age) gave products insoluble in benzene but soluble in ethyl alcohol.

H. E. BLAYDEN.

Determination of phenols in waters from coking plants. A. TRAVERS and AVENET (Compt. rend., 1930, 190, 975—976).—The total phenol content in such waters is determined by distillation with superheated steam (350°), followed by oxidation of the sulphur compounds in the distillate with excess of hydrogen peroxide, removal of the excess of hydrogen peroxide by boiling with cobaltic oxide, and titration with bromide-bromate mixture in the presence of excess of 15% sulphuric acid.

A. I. VOGEL.

Influence of the reactivity of coke on water-gas formation. K. BUNTE and A. GIESSEN (Gas- u. Wasserfach, 1930, 73, 241—247).—The gasification of coke in steam was determined in an electrically heated quartz tube, packed at the bottom with quartz beads to preheat the gas, and then with a column of graded coke. Nitrogen saturated with water at 70° was passed through the furnace at known rates and the effect of changes in reaction temperature and in time of contact was determined. Undecomposed steam in the exit gas was measured by cooling and drying with calcium chloride and phosphorus pentoxide; permanent gases were collected over brine for analysis. Curves are given showing the results obtained at different temperatures and velocities. The reactivity of seven different cokes

or charcoals towards steam is found to vary in the same order as their reactivity to oxygen or carbon dioxide. An exact relation between ignition point, combustibility, and reactivity to steam is only obscured by secondary reactions, in which steam reacts with carbon monoxide or reverse changes are set up as the gases are cooled.

R. H. GRIFFITH.

Gum formation in cracked gasolines. C. R. WAGNER and J. HYMAN (J. Inst. Petroleum Tech., 1929, 15, 674—680).—The colour of a gasoline is no indication of its gum content, but on evaporation a "gummy" gasoline develops a yellow colour until a final residue is obtained varying in consistency from an oily liquid to a solid mass and from pale amber to deep brown in colour. Usually this is insoluble in light petroleum, partially soluble in benzene, and completely soluble in acetic acid and alcohol. It appears that "gummy" gasolines contain little or no gum in solution, but that gum-forming materials are present which become resinified by heat during evaporation. The common aldehydes are incapable of forming appreciable quantities of gum alone, but by the addition of crotonaldehyde to a straight-run gasoline and exposure to oxygen and light a gum was formed on evaporation which was practically identical with that from a "gummy" gasoline. A sample of gum-free, vapour-phase cracked gasoline containing at least 50% of olefines, after exposure to oxygen and light, gave a reaction for peroxides and left an appreciable residue of gum on evaporation. A sample of highly unsaturated gasoline that had been exposed to an oxidising atmosphere was distilled *in vacuo* to a 28% bottom, having an acid number of 0.75 and a saponif. value of 13.4. It gave a strong test for peroxides and contained 0.2% O. On distillation it gave a series of violent explosions and vapours of acetaldehyde were evolved. The residual 10% remaining undistilled at 172° was a heavy resinous mass which was separated into a light-red oily liquid (*d* 0.9027, acid value 4.49, saponif. value 32.41), and a black asphaltic resin (acid value 64.86, saponif. value 307.9). The rate of gum formation is slow at first, but after the "period of induction" it is rapid, especially at elevated temperatures. In the dark and at room temperature autoxidation is very much less pronounced. A trace of hydrogen chloride gas causes polymerisation in gum-free, vapour-phase cracked gasoline and promotes oxidation. Glacial acetic acid and α -crotonic acid show only faint catalysing action. Both quinol and ammonia decrease the rate of gum formation. A theory of gum formation is offered involving autoxidation, per-acids, and oxonium compounds.

H. S. GARLICK.

Deterioration of cracked spirits by gumming. E. W. J. MARDLES and H. MOSS (J. Inst. Petroleum Tech., 1929, 15, 657—673).—Engine tests carried out on a Delco engine with bouncing-pin equipment in the standard manner by comparison with fuels of known H.U.C.R. showed that for cracked spirits stored at ordinary temperatures a decrease of anti-knock value occurs even after 23 days. The results obtained after incubation of cracked spirits and blends at higher temperatures and under different conditions of storage are given. The addition of a small quantity of phenol reduces the gum

yield and partly prevents the fall in H.U.C.R. After deterioration there was only a partial return to the original anti-knock quality after reconditioning by alkali wash and redistillation. Results show that the copper or glass dish method with standardised conditions give results comparing favourably with those obtained by more elaborate methods, but that evaporation in steam tends to promote hydrolytic cleavage and steam-distillation. An apparatus is described in which 20 c.c. of fuel are evaporated by a stream of air, steam, or nitrogen, from a copper or glass dish supported in a chamber suspended in an oil bath. Results are lowest when a copper dish is used, and tend to show that the copper does not catalytically induce gum formation. The gumming tendency of a spirit is determined by the gum yields of samples withdrawn at regular intervals from a bulk sample incubated in contact with air or oxygen. The gum yields of various fractions of a vapour-phase cracked spirit vary considerably. The results of the investigation of the influence of a large number of substances on the gumming rates of several cracked spirits are given. Water and anti-oxygens exhibit a considerably inhibiting effect. H. S. GARLICK.

Determination of existent gum in cracked distillates. W. S. NORRIS and F. B. THOLE (*J. Inst. Petroleum Tech.*, 1929, 15, 681—685).—The present dish methods for the determination of existent gum in cracked distillates suffer from the following disadvantages: (1) the period of evaporation is long and differs for samples of varying volatility; (2) the degree of oxidation is not under control and the degree of heat polymerisation varies with the rate of evaporation; (3) the results are affected by the thermal conductivity of the material of the dish; (4) the material of the dish may exert a specific catalytic action; (5) the usual final temperature of the "gum" is about 100°, which differs considerably from the normal temperature of an inlet valve upon which gum deposits usually accumulate; (6) evaporation in an atmosphere of steam involves a considerable and non-standardisable period of heating and may cause chemical interaction. The authors consider that an ideal test would embody the following essentials: (a) instantaneous evaporation of the whole of the sample so that heat effects would be reduced to a minimum; (b) evaporation at a temperature about 200°, approximately equal to that on an inlet valve of an automobile engine; (c) exclusion of air to avoid oxidation effects; and (d) maintenance of an accurately controlled temperature during the test. A method of flash evaporation based on the above and giving concordant results consists in mounting a spiral tube of pyrex glass, 45 cm. long and 6 mm. internal diam., in an oil bath maintained at 200° or, preferably, in an air oven maintained at 220°. The inlet of the spiral is fitted with a cork carrying a tube through which nitrogen is passed at a rate of 120 c.c. per min., and a tap-funnel by means of which a measured sample of spirit (25 c.c.) is introduced at a rate of 3 drops per sec. Vapour from the exit of the spiral, mixed with nitrogen, is led to a convenient condenser. The spiral tube is weighed before and after the evaporation of the sample and is subsequently cleaned in chromic acid mixture. The results are considerably

lower than those obtained in a glass dish on the steam bath. H. S. GARLICK.

Estimation of gum in petrol and its significance. S. J. M. AULD (*J. Inst. Petroleum Tech.*, 1929, 15, 645—656).—The methods of determining preformed gum in cracked distillates are discussed in relation to the value of such fuels as motor spirits. No evaporation method is entirely satisfactory, but the steam-bath method, although giving results consistently higher than the steam-oven method, is considered the most suitable for works' control owing to its simplicity of operation and reasonable concordance of results. The results are comparative only, and are higher than the least quantity of preformed gum in a given sample. The copper-dish method gives disproportionately high results due to the catalytic action of the metal and the longer time necessary to evaporate 100 c.c. over that needed for 20 c.c. It is established that only preformed gum has any bearing on engine operation, but the nature of the gums and their solubilities in the various hydrocarbons or other motor fuel constituents has to be considered. As a measure of the stability of a motor fuel the gum content is determined by the steam-bath method on a 20-c.c. sample of motor spirit withdrawn every 24 hrs. from a bottle in which it is stored in contact with oxygen. H. S. GARLICK.

Design of fractionating columns, with particular reference to petroleum distillation. R. B. CHILLAS and H. M. WEIR (*Ind. Eng. Chem.*, 1930, 22, 206—213).—The problem of designing a plate-column still for separating crude oil into six fractions is not capable of mathematical solution, and the number of plates corresponding to each fraction is determined experimentally. A small quantity of the lower-boiling fraction present in each product is best removed by a "stripper" and returned to the main column. The maximum fractionating effect is believed to be obtained with a free space velocity of 4—6 ft./sec., provided that entrained liquid can be removed. This, however, entails a considerable pressure drop. The heat balance and volumes of vapour and liquid for a typical still are worked out. Large columns with single down-pipes and overflows should have low bubble-caps of less height than the overflow weirs or the liquid gradient will be too high. An example of the working out of plate dimensions for a velocity of 2.25 ft./sec. is given. The spacing demanded for the flow of reflux is not sufficient for the removal of fine spray, whilst in the case dealt with the theoretical space is 7 in., at least 18 in. with one set of baffles should be provided. The baffles are a series of blades set at 30° to the vertical. The trend of design is towards smaller diameters and higher vapour velocities. C. IRWIN.

Atmospheric oxidation of sub-Moscow coal at different temperatures. N. M. KARAVAEV and A. K. IVANOV (*Chim. et Ind.*, 1930, 23, 294—300).—See B., 1929, 344.

See also A., Apr., 424, **Extinction of methane flames by water vapour** (COWARD and GLEADALL). 429, **Catalytic oxidation of carbon monoxide in presence of hydrogen** (KUENTZEL). 467, **Action of aluminium**

chloride and metallic aluminium on hydroxy-compounds (POSTOVSKI and LUGOVKIN).

PATENTS.

Internal-combustion engines utilising the gaseous mixture obtained from the electrolysis of water. C. FIRPO (B.P. 325,866, 30.10.28).—An engine using electrolytic gas as fuel is directly coupled to a generator supplying current for the electrolysis of caustic soda solution. The mixed gases produced are passed to the cylinder through a carburettor, to which alcohol is supplied as an "auxiliary fuel." The cylinder head is movable, so that the dimensions of the combustion space may be altered. Firing may be by sparking plug or by spontaneous ignition due to compression.

T. A. SMITH.

Apparatus for carbonisation of coal and like material. ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (B.P. 325,810, 23.11.28. Cf. B.P. 244,837; B., 1926, 428).—High-temperature retorts are fitted with a special base-plate which acts as a support for removable, metal, low-temperature retorts. This enables the plant to be used for high- or low-temperature carbonisation as may be desired.

T. A. SMITH.

Processes of and ovens for carbonising coal. H. F. MAUREL (B.P. 325,869, 1.11.28).—Preformed coal briquettes are passed through an oven having two heating zones, in the first of which the briquettes are heated gradually to about 315°, and in the second are maintained at that temperature, the total heating period being about 4 hrs. The briquettes are supported on a conveyor which enters the oven at the bottom. Horizontal partitions divide the first heating chamber into a series of superposed passages through which the conveyor, supported by guide sprockets at each end of the chamber, carries the briquettes. The conveyor then delivers them into the second heating chamber, which is in the form of a hopper, and re-collects the finished briquettes from the bottom thereof. The conveyor enters and leaves the oven through a liquid seal. The briquettes are heated directly by the circulation of hot combustion gases through the oven; they may also be heated indirectly by the circulation of hot gases through some of the partitions in the first heating chamber, which are made hollow for that purpose.

A. B. MANNING.

Heating of coke ovens or the like. P. E. VERPEAUX, and UNION CHIM. BELGE SOC. ANON. (B.P. 326,094, 9.5.29).—Horizontal coke ovens having heat regenerators and vertical heating flues are provided with auxiliary flues by means of which air is distributed to various levels in the heating flues to produce supplementary combustion of the gas admitted to the lower portion of the heating flues. The quantity of hot air which can be drawn through the regenerators is increased by fitting injectors constructed of refractory material under the auxiliary flues. Air is supplied to the injectors at 100 mm., and further air is drawn into the auxiliary flues through the regenerators. By regulating the pressure at the nozzles the temperature of the air supplied to the auxiliary flue can be controlled.

T. A. SMITH.

Production of [carbonaceous] artificial materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,872, 3.11.28 and 16.1.29).—The solid residues obtained by the complete hydrolysis of the carbohydrates of vegetable materials with acids are mixed with sulphur and/or bituminous materials, and, if desired, with fillers and other binding agents etc., and are compressed while hot into suitable shapes. The products can be used as acid-proof coating agents, insulating materials, roofing materials, etc. A. B. MANNING.

Extraction of products from carbonaceous materials. J. W. H. AINSWORTH (B.P. 300,984, 19.11.28. Austral., 22.11.27).—The material in granular form is conveyed through an inclined retort on an endless belt or scraper conveyor, and passes therein through a series of zones of progressively higher temperatures. The zones are separated from each other by closures formed by the material itself, as it is drawn through sections of the retort, which are diminished in height. The gases and vapours in each zone are withdrawn separately through re-fractionating chambers arranged directly above the contractions in the retort, and so constructed as to prevent condensed liquid from falling back on to the material. A hopper with feed delivery means is fitted at one end of the retort. The conveyor enters and leaves the retort through a water-seal. At the outlet end the seal takes the form of a tank into which the residual solid falls, and from which it is removed at convenient intervals. A. B. MANNING.

Treating coal to remove smoke-producing constituents. F. LLOYD, R. V. WHEELER, and A. P. PEHRSON (B.P. 324,386, 23.10.28).—Bituminous coal is heated in a single stage at a temperature not much higher than its active decomposition point and not lower than that at which the smoke-producing constituents (hydrocarbons and resins) are distilled. The required temperature of distillation is dependent on the carbon content of the coal, and varies from about 250° to 365°. A. B. MANNING.

Distillation and utilisation of solid fuels. A. J. A. HÉRENG (B.P. 325,809, 23.11.28. Cf. B.P. 322,332; B., 1930, 176).—The dried, and preheated fuel is passed through a distillation chamber in a horizontal layer on an endless conveyor which is permeable to gases, the volatile products of distillation being removed from the under face of the moving layer. The roof of the chamber is formed by a radiating plate heated by a furnace wherein the gas produced by the plant itself may be burnt. A current of gas is circulated continuously through heating coils in the furnace, thence over and countercurrent to the moving layer of fuel, up through the dryer and preheater and through the cooling chamber for the coke, back to the heating coils. If desired, steam may be introduced into the gas-circulating system. The solid residue is cooled and recovered as a compact semi-coke, or may be conveyed directly to a combustion chamber and utilised for steam raising etc. A. B. MANNING.

Production of fuel from brown coal etc. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of R. HEINRICH (G.P. 457,613, 5.3.25).—To utilise the carbon content of

flue gases from industrial furnaces, finely-divided brown coal or other low-grade fuel is injected into the flues under conditions which prevent its combustion and the gases are passed through an electrical precipitation plant. The resulting flue dust has a higher calorific power than the original brown coal, and is suitable for direct use in coal dust-fired furnaces. A. R. POWELL.

Conversion of coal, tars, mineral oils, etc. into fuels of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,862, 28.9.28).—The destructive hydrogenation of the starting material is carried out in stages and the carbonaceous substances of high mol. wt., which would otherwise choke the catalysts used in the later stages, are removed, *e.g.*, by conversion into products of lower mol. wt. in the earlier stages of the process, or by condensation or adsorption before the material enters the second or later stages. [Stat. ref.]

A. B. MANNING.

Preparation of active carbon. A.-G. F. STICKSTOFFDÜNGER (B.P. 301,330, 26.11.28. Ger., 26.11.27).—Ashes carried over into the flue as a result of the combustion of coal etc. are sieved or graded, purified by boiling with hydrochloric acid, washed with water, dried, and ground.

H. ROYAL-DAWSON.

Gas producer for the gasification of ligneous fuel. K. SCHMIDT (B.P. 325,616, 5.12.28).—In order to facilitate the removal of the ash from a producer with downward combustion, in which ligneous fuel, *e.g.*, rice-hulls, are gasified, the lower part of the producer is provided with a vertical grating in addition to the horizontal bottom grating, and the lower part of the fuel bed is kept in constant motion by means of a stirring device.

A. B. MANNING.

Generation of water-gas from powdered fuel. M. HELLER (B.P. 308,972, 27.3.29. Ger., 2.4.28).—The powdered fuel and steam are injected into an externally heated chamber in such a manner that the fuel and steam circulate therein and the particles of fuel are brought repeatedly into contact with the hot walls. The chamber may be circular in vertical cross-section, the fuel and steam being injected tangentially in at the bottom and the water-gas and ash withdrawn through a central discharge conduit. A number of such chambers, alternating with heating chambers, may be arranged in the form of a battery. The gas and air are introduced into the heating chambers so that a circulating flame is produced therein, and the waste gases are withdrawn from the centre. In another form of apparatus the fuel and steam circulate vertically in a shaft containing a vertical dividing wall which leaves passages at the top and bottom. The fuel and steam are injected at the bottom and the water-gas is withdrawn from the top.

A. B. MANNING.

Grids for gas purifiers. A. BEUTHNER (G.P. 456,371, 27.1.27).—The grids comprise a number of rotatable, horizontal shafts carrying two series of grid rods, one above the other, fixed to the shafts in such a way that the rods of the lower series are all on the left-hand side and those of the upper series on the right-hand side of the shafts. In this way blocking of the passage ways between the rods may be avoided by occasional gentle rotation of the shafts.

A. R. POWELL.

Discharge conduits for moisture-laden gases. E. MEYER and E. FICHTE (B.P. 325,793, 20.11.28).—Waste gases from gas heaters or gases containing a high percentage of steam or other condensible substances are discharged into a conduit made from a non-combustible plastic mass comprising moist wood shavings, Portland cement, sand, and water; the material may be rendered water-repellent by a coating of dilute aluminium acetate solution.

H. ROYAL-DAWSON.

Removal of ammonia and sulphuretted hydrogen from gases. C. J. HANSEN, Assec. of H. KOPPERS A.-G. (B.P. 299,765 and Addn. B.P. 301,061, [A] 18.9.28, [B] 19.9.28. Ger., [A] 31.10.27, [B] 24.11.27).—(A) The gases are scrubbed with iron thiosulphate liquors $[\text{FeS}_2\text{O}_3 + 2\text{NH}_3 + \text{H}_2\text{S} = (\text{NH}_4)_2\text{S}_2\text{O}_3 + \text{FeS}]$, and any hydrogen sulphide in excess of the ratio $2\text{NH}_3 : 1\text{H}_2\text{S}$ is removed by a separate liquor, preferably either an iron polythionate solution or an iron hydroxide suspension. The order in which the operations are carried out may be varied; *e.g.*, the ammonia may be removed first by condensation, then part of the hydrogen sulphide by means of the iron polythionate solution, and finally the remainder of the hydrogen sulphide, in the presence of all or part of the ammonia previously removed, by the iron thiosulphate liquor. The spent liquor containing iron sulphide is regenerated either by treatment with sulphurous acid to give thiosulphate, or by blowing in air to give iron hydroxide. (B) The process may be further modified by subjecting the gases to a preliminary washing with the spent thiosulphate liquor, and subsequently adding this ammoniacal liquor to the iron thiosulphate liquor in the main scrubbing operation. Other minor modifications of the process are described.

A. B. MANNING.

Separation of hydrogen from gaseous mixtures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,968, 30.1.29).—The gaseous mixtures are washed under pressure and at elevated temperatures with a liquid, *e.g.*, a tar or mineral oil, the solvent power of which for hydrogen increases with a rise in temperature. On cooling the liquid without releasing the pressure, a gas rich in hydrogen is evolved. Suspensions of coal in oil, or tars etc. which are to be hydrogenated, may be used as washing liquids under such conditions of temperature and pressure that they take up the quantity of hydrogen necessary for their subsequent hydrogenation, without any appreciable alteration in their chemical nature.

A. B. MANNING.

Separation of unsaturated hydrocarbons from gaseous mixtures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,817, 26.11.28. Cf. B.P. 325,309; B., 1930, 406).—Unsaturated hydrocarbons, and in particular acetylene, are recovered from gaseous mixtures by washing the latter, preferably under pressure, with a liquid ketone containing more than three carbon atoms, *e.g.*, diethyl ketone, acetophenone, cyclohexanone, etc.

A. B. MANNING.

Manufacture of hydrocarbons from tars, mineral oils, and like liquid hydrocarbon materials. I. G. FARBENIND. A.-G. (B.P. 301,059, 3.9.28. Ger., 24.11.27).—The tars, oils, etc. are subjected to destructive hydrogenation at elevated temperatures and pressures by

passing the material, together with hydrogen, or gases supplying hydrogen, through more than two reaction vessels in series, so arranged that no appreciable cooling occurs between the separate reaction vessels. The material and the hydrogen may be preheated before entering the first reaction vessel, and the desired temperatures in the system maintained by suitably lagging the reaction vessels and connecting pipes, the reaction being exothermic. A. B. MANNING.

Refining of low-temperature tar, tar oils of any origin, crude benzene, and products obtained by the destructive hydrogenation of carbonaceous materials. F. HOFMANN and C. WULFF (B.P. 301,420, 1.11.28. Ger., 29.11.27. Cf. B.P. 298,484; B., 1930, 406).—The crude materials are washed with a solution of sulphuric or phosphoric acid in an oxygen-containing organic solvent, *e.g.*, ethyl alcohol. A. B. MANNING.

Preparation of artificial resin, wax, and other complex hydrocarbons from coal-distillation residues. E. RAHMAN (B.P. 325,816, 27.11.28).—Coal tar and pitch are distilled in the presence of mixtures of the chlorides and oxides of iron, aluminium, calcium, or magnesium. Steam at 3–8 atm. is admitted through a pipe carried down to the bottom of the liquid and ending in a distributor having a large number of small apertures. Hydrogen may be admitted with the steam to bring about the formation of softer products, and crude montan wax may be added to the pitch. The waxes and resins produced are useful in the manufacture of varnishes and impregnating materials. The product distilling between 200° and 300°, on treatment with sodium or sodamide at 150°, yields fluorene. Pyrene, fluoranthene, retene, and picene may be obtained by fractionating the product distilling above 360°. T. A. SMITH.

Purification and/or utilisation of waste liquors. F. C. BUNGE and H. MACURA (B.P. 326,112, 18.6.29).—The phenol-containing liquors obtained in the treatment of coal tar or wood tar are purified by treatment with aldehydes (formaldehyde or furfuraldehyde). Resins are formed in a state of fine division. Before or during the treatment coagulating agents such as aluminium sulphate or lead acetate may be added. The recovered resins are useful for paint manufacture, and, when lead acetate has been used for coagulation, have a useful lead content. The bulk of the aldehyde resins may be removed by filtration and the residue by flotation treatment with coal sludge. The purified water is practically free from hardness, and may be used as boiler-feed water. T. A. SMITH.

Conversion of mineral oils into hydrocarbons of low b.p. E. D. KAMM, R. C. ODAMS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 325,487, 15.11.28).—Heavy mineral oil is hydrogenated to produce chiefly middle oil, heavy residue from this process being returned for further hydrogenation. This middle oil is cracked and any heavy residues produced are also returned to be rehydrogenated. By this method there is no loss of liquid carbonaceous material, and the formation of incondensable gases is low, if suitable catalysts (such as ferric oxide) are used in the hydrogenation step. Thus, by hydrogenating a heavy mineral oil, 86% of which

boils above 300°, for 1 hr. at 200 atm. and 450° in the presence of 5% of hydrated ferric oxide, 20% of petrol and 30% of middle oil is obtained. The heavy oil is returned for hydrogenation. The middle oil is cracked for 15 min. at 450° and 100 atm., yielding 37% of petrol, 45% of middle oil, and a pitch residue which is rehydrogenated. Coal pastes may be similarly treated. The preheated heavy oil or paste is pumped to a hydrogenation tower from which it passes to a catch-pot maintained at 350°. Residue from this pot is returned for hydrogenation, while the vapours consisting of benzene and middle oil are passed to a fractionating condenser, where the middle oil portion is condensed and retained in a catch pot maintained at 200°. From this catch pot the middle oil is transferred to a cracking unit all the products from which are passed to the heavy-oil catch pot. The heavy portions are thus returned for hydrogenation and the middle oil recycled for further cracking. The combined benzene vapours from all stages are condensed and the gases decomposed to provide hydrogen for the process. T. A. SMITH.

Cracking of oils. S. SEELIG (B.P. 296,000, 18.8.28. Ger., 22.8.27).—Coke formation is prevented during cracking by passing preheated cracking stock into an asphalt-removing chamber, where it comes in intimate contact with the oil which has been through the cracking unit, whereby all but the asphalt is vaporised. The asphalt is run off and the vapours are conducted to a second separator maintained at such a temperature that cracking stock is condensed and light vapours are removed to a condenser. The refined cracking stock is pumped from the second dephlegmator to the cracking unit, which consists of a metal bath maintained at cracking temperature. The oil is fed in below the surface of the molten metal. A uniform mixture of new refined cracking stock and recycled material is obtained. T. A. SMITH.

Mineral oil distillation. A. E. PEW, JUN., and H. THOMAS, ASSRS. to SUN OIL Co. (U.S.P. 1,742,933, 7.1.30. Appl., 17.3.24).—A stream of oil is passed into, through, and out of a series of confined spaces in which the oil is distributed over surfaces heated by mercury vapour. This vapour is generated from a separate supply of mercury and flows through multiple paths in heat-exchange relation but out of contact with the oil, thereby effecting by heat exchange and by its condensation the vaporisation in each confined space of the desired oil fraction. Condensed mercury is returned to the main supply, and the oil vapours are removed and led to separate condensers. Cracking is minimised by maintaining in one or more of the confined spaces an absolute pressure on the oil of less than 25 mm. Hg, by maintaining so thin a layer of oil on the heated surfaces as substantially to eliminate hydrostatic pressure, and by regulating the pressure and temperature of the condensation of the mercury vapour at each locus of heat-exchange independently. H. S. GARLICK.

Treatment of mineral oils. A. H. ACKERMAN, ASSR. to CATALYTIC CHEM. Co. (U.S.P. 1,742,020, 31.12.29. Appl., 14.12.25. Renewed 15.4.29).—In order to prevent the formation of, or to break up, carbon clusters in petroleum products, the oil is agitated with a composi-

tion comprising anthracene (10 pts.), sulphuric acid (2 pts.), oil of myrbane (4 pts.), sodium hydroxide (1 pt.), and naphthalene (200 pts.), with or without the addition of phosphate rock, sodium phosphate, ammonium carbonate, sugar, arsenious acid, arsenic sulphide, and golden antimony sulphide. H. S. GARLICK.

Manufacture of lubricating oils. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,832, 28.11.28).—An alkylated naphthalene hydrocarbon substituted by one or more alkyl groups, or a mixture thereof with a customary lubricating oil, is allowed to flow slowly at atmospheric or reduced pressure through an ozoniser and is there subjected to the action of the silent electric discharge produced by an alternating current of 500—3500 or more periods/sec. H. S. GARLICK.

Separating oily emulsions. G. W. COGGESHALL and A. REILLY, Assrs. to JEFFERSON CONSTRUCTION & OIL TREATING Co. (U.S.P. 1,742,648, 7.1.30. Appl., 1.7.27).—Emulsions to be treated are mixed at ordinary temperatures with dispersions or solutions of the sulphonic acids or their alkaline salts, recovered from the sludge resulting from the treatment of mineral oils with acid, in the proportion of approx. 1.4 lb. per 100 gals. of emulsion. Sufficient mineral acid is then added to cause substantially all of the sulphonic acid salts to be converted into free sulphonic acid within the emulsion, and sufficient time is allowed for settling to take place. H. S. GARLICK.

Demulsifying soap. J. F. TURNER, D. H. MATTHEWS, and M. F. ROSS, Assrs. to VACUUM OIL Co. (U.S.P. 1,742,623, 7.1.30. Appl., 30.11.27).—The acid sludge resulting from the treatment of petroleum oils with concentrated or fuming sulphuric acid, after being separated from the reacting oil, is washed with water to remove unspent acid and then treated with open steam. After settling and removing the aqueous portion, the acid sludge is neutralised, preferably with caustic soda solution, and then treated with "white water," obtained by washing with water a mineral oil that has been treated with acid and alkali, after which the layer containing in solution and in suspension the demulsifying soap is removed from the supernatant layer containing colouring matter. H. S. GARLICK.

Manufacture of motor fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,858, 29.8.28).—A non-knocking motor fuel having final b.p. 185° is obtained by mixing a knocking gasoline, b.p. below 150°, with an anti-knocking hydrocarbon fraction, b.p. 150—180°, which may be obtained by (a) distillation of aromatic tars or aromatic petroleum; (b) condensation of unsaturated compounds; (c) destructive hydrogenation or dehydrogenation of aliphatic or hydroaromatic compounds, aromatic middle oils, such as anthracene oils, coal-tar products, residue obtained by cracking and high-temperature tars; or (d) by the extraction of products of appropriate boiling range with sulphur dioxide. H. S. GARLICK.

Manufacture of highly concentrated paraffin wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,941, 7.1.29).—Crude paraffin wax is treated in the solid state with an aqueous solution of a wetting or

emulsifying agent, e.g., aqueous solutions of potassium, ammonium, or sodium soaps, and in particular of polynuclear and alkylated sulphonic acids, thereby displacing the oil from the wax. H. S. GARLICK.

Apparatus for manufacture of combustible gas. W. J. MURDOCK, E. E. LUNDGREN, and O. B. EVANS, Assrs. to PIER PROCESS CORP. (U.S.P. 1,743,725, 14.1.30. Appl., 28.8.26. Renewed 21.11.29).—See B.P. 240,440; B., 1927, 246.

Burner for pulverulent or gaseous fuels. ALLGEM. ELEKTRICITÄTS GES. (B.P. 304,142, 14.1.29. Ger., 13.1.28).

Drying and heating apparatus (B.P. 326,269). **Control of temperature** (B.P. 326,309).—See I. **Acetic acid from pyroligneous acid** (B.P. 316,284).—See III. **Self-combustible mixtures** (B.P. 299,396).—See XXII.

III.—ORGANIC INTERMEDIATES.

Drying of organic solutions in volatile solvents. A. LUND (Chem. Umschau, 1930, 37, 29).—Such solutions are easily dried without loss of solvent by shaking with, e.g., anhydrous sodium sulphate in a glass-stoppered separating funnel fitted with a fused-in, porous, glass filter disc just above the tap. The solution is drained off through the filter and tap, and the dehydrating agent is easily washed with fresh, dry solvent.

E. LEWKOWITSCH.

Detection of organic compounds. L. ROSENTHALER (Pharm. Ztg., 1930, 75, 272—273).—Acetone and alcohols, even when completely free from aldehyde, produce a red colour with Schiff's reagent unless the total quantity present is less than 20% of methyl alcohol, 5% of ethyl alcohol, 10% of propyl and isopropyl alcohols, 2.5% of *n*-butyl alcohol, and 5% of acetone. Previous addition of hydrochloric acid prevents the formation of the red colour in the Schiff reaction, but if the acid be added after the formation of the colour this changes to blue and gradually disappears. Denigès' reagent oxidises methyl, ethyl, *n*-propyl, *n*-butyl, and isobutyl alcohols to the corresponding aldehydes and is itself reduced to mercurous sulphate. The aldehydes may be characterised by formation of their *p*-nitrophenylhydrazones. α -Naphthol may be differentiated from β -naphthol by the character of the precipitates produced by mixing equal amounts of their saturated solutions and a 2% solution of mercuric acetate; β -naphthol gives a white precipitate soluble in glacial acetic acid and α -naphthol gives a yellow viscous liquid from which flocks separate on keeping. With β -naphthol the reaction is sensitive at a dilution of 1:5000, and with α -naphthol 1:250,000. The difference in behaviour is most marked when examined under the microscope. E. II. SHARPLES.

Determination of phenols. TRAVERS and AVENET. **Xylene from light oils.** BESPOLOV.—See II. **Separation of fatty acids.** GROSSFELD.—See XII. **Absolute alcohol from raw spirit.** SCHLAGE. **Waste from butyl alcohol manufacture.** HATFIELD.—See XVIII.

See also A., Apr., 501, **Higher alcohols of fermentation** (SWENARTON).

PATENTS.

Catalytic hydrogenation and dehydrogenation of organic compounds. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 305,198, 3.12.28. Ger., 2.2.28).—Catalyst masses are prepared by using a suitable granulated carrier, *e.g.*, magnesia or lime, as a precipitant for the oxide or hydroxide of the catalyst metal. A solution of a salt of the metal or metals, *e.g.*, copper formate, copper and manganese nitrates, or nickel nitrate, is poured over the granulated carrier. C. HOLLINS.

Manufacture of ethylene [from acetylene]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,695, 22.2.29).—Aluminium powder, cerium, or ceria is used as catalyst in the hydrogenation of acetylene to ethylene at 280–320° and preferably at ordinary pressure.

C. HOLLINS.

Dehydration of vapour mixtures containing acetic anhydride and water. I. G. FARBENIND. A.-G. (B.P. 300,563, 14.11.28. Ger., 14.11.27. Addn. to B.P. 285,090; B., 1929, 671).—In the process of the prior patent a dephlegmator is placed at the top of the separating column, and the solvent is injected only at the bottom.

C. HOLLINS.

Extraction of acetic acid from pyroligneous acid. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 316,284, 15.3.29. Belg., 28.7.28).—Tars are removed from pyroligneous acid, prior to the extraction of acetic acid, by treatment with *C*-alkylated cresols, *e.g.*, the *C*-butylcresol obtained by condensing ordinary cresol with butyl alcohol in presence of zinc chloride.

C. HOLLINS.

Manufacture of glacial acetic acid from aqueous acetic acid. I. G. FARBENIND. A.-G. (B.P. 300,923, 19.11.28. Ger., 19.11.27).—A halogenated hydrocarbon (trichloroethylene) is used as entraining liquid to remove water from dilute (85–95%) acetic acid.

C. HOLLINS.

Production of keten. KODAK, LTD., Assecs. of H. T. CLARKE and C. E. WARING (B.P. 309,577, 15.3.29. U.S., 13.4.28).—Acetone (etc.) is vaporised into a chamber containing a network or sponge of copper, free from iron and nickel, at 650–700°, the time of contact being less than 5 sec. A yield of 80% is claimed.

C. HOLLINS.

Production of esters of α -hydroxycarboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,531, 20.10.28. Addn. to B.P. 300,040; B., 1929, 11).—In the process of the prior patent alcohol and the necessary amount of water are added before or after the formation of the cyanhydrin, an inert solvent being present and the esterification being performed without isolation of the intermediate products. The process is suitable for alcohols of high mol. wt. and for viscous aldehydes and ketones. The preparation of cyclohexyl lactate, b.p. 95–110°/7 mm., and β -chloroethyl α -dihydroxyvalerate, b.p. 120–135°/10 mm., and cyclohexanol-2-carboxylate, b.p. 134°/6 mm., is described.

C. HOLLINS.

Preparation of [polymerisation] products of acrylic acid or its derivatives. ROHM & HAAS A.-G. (B.P. 304,681, 2.11.28. Ger., 24.1.28).—Acrylic acid or

a derivative (*e.g.*, ethyl acrylate) is polymerised (and in part oxidised) by heating, preferably in acetone solution, in a current of oxygen or air. Solid products or their solutions are obtained which are useful as adhesives and for films, lacquers, etc.

C. HOLLINS.

Preparation of diarylthiocarbamides. SILESIA VER. CHEM. FABR. (B.P. 307,906, 4.1.29. Ger., 17.3.28).—Excess of aniline (twice the theoretical amount) is warmed for 4–5 hrs. with carbon disulphide at 45–50° under reflux. A 90% yield of very pure thiocarbamide, m.p. 153–154°, is claimed.

C. HOLLINS.

Manufacture of *N*-[hydr]oxyethyl derivatives of nuclear substitution products and homologues of 4-amino-1-oxybenzene [*p*-aminophenol]. I. G. FARBENIND. A.-G. (B.P. 300,503, 7.11.28. Ger., 12.11.27. Addn. to B.P. 280,873 and 290,997; B., 1929, 38, 236).—Nuclear substituted *p*-aminophenols are condensed with ethylene chlorohydrin or oxide. The preparation of *N*-mono- and *N*-di- β -hydroxyethyl derivatives of 2-chloro-*p*-aminophenol, m.p. 67–68° and 112°, respectively, 5-aminosalicylic acid, m.p. 206° (decomp.) and 183° (decomp.), respectively, and of 6-amino-*m*-cresol, m.p. 80–82° and 108–109°, respectively, is described.

C. HOLLINS.

Manufacture of derivatives of naphthalene [from *p*-benzoquinones and α -butadienes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 324,661, 26.9. and 1.10.28).—A *p*-benzoquinone substituted in position 2 and/or 3 is condensed with an α -butadiene; if no 2- or 3-substituent is present, mild conditions are employed. *p*-Benzoquinone and 0.85 pt. of α -butadiene at 60–65° under pressure yield a product which is converted by acid or alkali at 50–60° into 1:4-dihydroxy-5:8-dihydronaphthalene, oxidisable by way of a quinhydrone to α -naphthaquinone. Other 1:4-dihydroxy-5:8-dihydronaphthalenes described are the 2-methyl, 6:7-dimethyl, 6-methyl, 2-chloro-, 2:3-dichloro-, and 2:3-dichloro-6:7-dimethyl derivatives.

C. HOLLINS.

Recovery of β -naphthol and other difficultly filterable organic compounds. R. F. STEWART, R. J. VENN, and DORR CO., LTD. (B.P. 325,772, 1.7.29).—Fine sand is added to the suspension of β -naphthol, which is then filtered, and the filter-cake is levigated to obtain a concentrated aqueous suspension of β -naphthol, from which the naphthol is separated as an oil after raising to the requisite temperature.

C. HOLLINS.

Manufacture of oxythionaphthens [thioindoxyls]. I. G. FARBENIND. A.-G. (B.P. 298,955, 17.10.28. Ger., 17.10.27).—4-Alkylthioindoxyls are obtained by heating the corresponding *o*-cyano- or *o*-carboxy-arylthioglycollic acids with water at 180°. The preparation of 6-chloro-4-methyl- and 6-chloro-4:7-dimethyl-thioindoxyls is described.

C. HOLLINS.

Manufacture of *OO*-diacetyldiphenolisatin. F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 314,518, 17.5.29. Ger., 29.6.28. Addn. to B.P. 221,976; B., 1925, 26).—Diphenolisatin diacetate is obtained in one operation by heating isatin and phenol in acetic acid at 80° in presence of a little sulphuric acid, adding acetic anhydride, and continuing the heating until acetylation is complete.

C. HOLLINS.

Manufacture of 1-halogeno-2-aminonaphthalene [naphthylamine]sulphonic acids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,387, 12.2.29).—A β -naphthylaminemono- or di-sulphonic acid is acetylated, halogenated in the α -position, acidified, and hydrolysed. The preparation of 1-chloro- β -naphthylamine-5-, -6-, and -8-sulphonic and -5 : 7-disulphonic acids, and of 1-bromo- β -naphthylamine-7-sulphonic acid, is described.

C. HOLLINS.

Manufacture of diphenyl. FEDERAL PHOSPHORUS Co., Assees. of T. J. SCOTT (B. P. 312,902, 8.2.29. U.S., 2.6.28).—Benzene vapour passes through a metal coil immersed in a lead bath at 600—650° (i.e., below the temperature at which diphenyl is formed) and bubbles up through the molten lead into a similar arrangement of metal coil and lead at 750—800°. The diphenyl produced is passed at high velocity into a water-cooled condenser.

C. HOLLINS.

Manufacture of halogen derivatives of the dibenzanthrone and isodibenzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,525, 21.8.28, 23.3, 22.5; and 4.6.29).—Dibenzanthrone or isodibenzanthrone is brominated in an inorganic medium, especially chlorosulphonic acid. The products are usually isomerides of the known bromo-compounds, and when chlorosulphonic acid above 50° is used they contain both chlorine and bromine. Halogen carriers should preferably be present. Dibenzanthrone yields a chloro-dibromo-compound which is a marine-blue vat dye of greater fastness than the starting material; 26 other examples, leading to violet or blue vat dyes, are given.

C. HOLLINS.

Manufacture of derivatives of pyranthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,250, 9.10.28).—The nitro-group in mononitropyranthrone (cf. B.P. 23,361 of 1912; B., 1913, 746) is exchanged for halogen by the action of benzoyl chloride, or is reduced and exchanged for halogen or a hydroxyl, thiol, cyano-, or thiocyno-group by the Sandmeyer reaction. Hydroxypyranthrone may be etherified, for example with β -chloroethyl-*p*-toluenesulphonate. The products are either dyes or intermediates.

C. HOLLINS.

Separation of unsaturated hydrocarbons (B.P. 325,817). **Complex hydrocarbons from coal** (B.P. 325,816).—See II.

IV.—DYESTUFFS.

Fastness of dyestuffs in ultra-violet light. G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 50—59).—Pieces of filter paper, soaked in filtered aqueous or alcoholic solutions of the dyestuffs and dried in the dark, were exposed at about 50 cm. distance to the light of a 2000-c.p. mercury-vapour lamp without an intervening nickel oxide glass screen. Effects due to the heat or ozone generated were minimised by ventilation. The dyestuffs examined, belonging to the nitroso-, nitro-, and azo groups, were thus divided into four classes, in accordance with their fastness under these conditions.

T. H. POPE.

Relation of media p_H to the bacteriostatic action of dyes. S. E. OWEN (Amer. J. Pharm., 1930, 102, 154—158).—The action of 13 dyes on 4 types of bacteria has

been examined. Of the 10 food dyes investigated, 8 of them showed no bacteriostatic action in a p_H range of from 5.4 to 10.4. The p_H value of the media is an insignificant factor in the selective action of gentian-violet, basic fuchsin, and probably methylene-blue. Erythrosin inhibits the growth of *B. anthracis* within the range studied (4.4—10.4) and guinea-green inhibits the growth of the same organism at p_H 7.4 and under.

E. H. SHARPLES.

See also A., Apr., 473, **Behaviour of Pechmann dyes with amines** (BOGERT and GREENBERG). 477, **Acyl-p-quinones and the "Pechmann dyes" problem** (BOGERT and HOWELLS).

PATENTS.

Manufacture of nitrogenous vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,550, 15.10.28. Addn. to B.P. 318,180; B., 1929, 51).—The process of the prior patent is extended to condensation of halogenated (etc.) polynuclear heterocyclic compounds with two or more primary or secondary amines of which at least one is vattable and at least one contains a 4-ring residue. Examples are α -aminoanthraquinone and aminodibenzanthrone with chlorobromoanthraquinone-acridone (grey), or the dichlorinated vat dye from pyrazolanthrone (by alkaline melt and ethylation) (red-violet) or with dibromo-6 : 6'-dichloro-4 : 4'-dimethylthioindigo (grey-black) or with hexabromodihydroxydinaphthazine (green-grey); tribromoanthraquinonenaphth-acridone with 1-amino-4-methoxyanthraquinone and aminopyranthrone (grey), etc.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,267, 15.11.28. Cf. B.P. 305,082; B., 1929, 277).—The process of the earlier patent is applied to the carbazolisation of 1 : 1'-dianthraquinonylamines containing two acridone rings in the 4 : 4'-position and no arylamino-groups. Trichlorinated anthraquinone-2 : 1-acridone is condensed with the aminodichloroanthraquinone-acridone obtain by the action of oleum on the product from 4-bromo-1-aminoanthraquinone-sulphonic acid and 2 : 6-dichloroanthranilic acid (followed by removal of the sulphonic group) and the resulting dianthraquinonylacridone is carbazolised to give a fast green vat dye.

C. HOLLINS.

Manufacture of vat dyes [of the isodibenzanthrone series]. I. G. FARBENIND. A.-G. (B.P. 303,123, 28.12.28. Ger., 28.12.27).—1 : 4-Dichloro-8-arylnaphthalenes yield vat dyes when treated with alkaline condensing agents, 4 mols. of hydrogen chloride being removed from 2 mols. of the starting material. 1 : 4-Dichloro-8-benzoylnaphthalene with alcoholic potassium hydroxide at 100—150° gives isodibenzanthrone; a bluer violet is obtained from the *o*-chlorobenzoyl compound, a redder-violet from the *p*-isomeride. Dibenzoisodibenzanthrones obtained from 1 : 4-dichloro-8- α - and - β -naphthoylnaphthalenes are, respectively, reddish-blue and pure blue vat dyes.

C. HOLLINS.

Manufacture of derivatives of mono-aminodibenzopyrenequinones. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,222, 8.10.28. Cf. B.P. 324,964; B., 1930, 411).—Aminodibenzopyrenequinone

is condensed with benzoyl chloride (orange-red), *m*-methoxybenzoyl chloride (orange-red), 1-chloro-anthraquinone-2-carboxylic chloride (orange), α -chloroanthraquinone (orange-brown), cyanuric chloride (red-brown), 1-nitro- or 1-amino-anthraquinone-2-carboxylic chloride (brick-red), oxalyl chloride (yellow), bromo- or nitro-dibenzopyrenequinone (brown), bromobenzene (brown), octobromocyclohexane (green-grey), trichlorinated anthraquinone-2 : 1-acridone (brown), benzotrichloride (orange), methyl iodide (green-grey), tetrabromindigo (brown), dibromoanthanthrone (brown), nitropyranthrene (grey), nitrodibenzanthrone.

C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 300,504, and 300,557, [A], 9.11.28, [B] 14.11.28. Ger., [A] 12.11.27, [B] 14.11.27).—(A) A halogenated *m*-5-xylydine is diazotised and coupled in substance or on the fibre with an arylamide of 2 : 3-hydroxynaphthoic or acetoacetic acid. Examples are: 2-bromo-*m*-5-xylydine with 2 : 3-hydroxynaphthoic *p*-anisidide (bluish-red); 4-chloro-*m*-5-xylydine with the 5-chloro-*o*-anisidide (scarlet) or bisacetoacetylolidine (reddish-yellow), etc. Amongst the new bases used are 2-chloro-*m*-5-xylydine, m.p. 58–59°, 2-bromo-*m*-5-xylydine, m.p. 66–67°, 4-bromo-*m*-5-xylydine, m.p. 27–28°, b.p. 261–263°; 2 : 4-dichloro-*m*-5-xylydine, m.p. 71–72°; 4 : 6-dichloro-*m*-5-xylydine, m.p. 88–89°, and 2 : 4 : 6-trichloro-*m*-5-xylydine, m.p. 188–189°; (B) 2-Amino-*p*-cymene or a derivative is diazotised and coupled with a 2 : 3-hydroxynaphthoic arylamide. Examples are: 2-amino-*p*-cymene with 5-chloro-*o*-anisidide (scarlet); 5-chloro-2-amino-*p*-cymene with 5-chloro-*o*-toluidide (bluish-red); 6-nitro-2-amino-*p*-cymene with *p*-anisidide. [Stat. ref.]

C. HOLLINS.

Manufacture of azo [acid pyrazolone] dyes. SOC. CHEM. IND. IN BASLE (B.P. 301,096, 24.11.28. Switz., 24.11.27).—Dichloroaniline is diazotised and coupled with a halogenated sulphaaryl methylpyrazole, e.g., 2 : 5-dichloroaniline \rightarrow 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone (yellow on wool or silk). [Stat. ref.]

C. HOLLINS.

Manufacture of yellow [pyrazolone] azo dyes [for wool, silk, and lakes]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 325,229, 13.11.28).—A pyrazolone is coupled with a diazotised aminoaryl-sulphonaryalkylamide; e.g., *m*-aminobenzenesulphonmethylanilide, $\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{SO}_2 \cdot \text{NPhMe}$, is diazotised and coupled with 1-*p*-sulphophenyl-3-methyl-5-pyrazolone for a yellow wool dye.

C. HOLLINS.

Manufacture of [mordant] azo dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,485, 12.11.28).—An aminosulphosalicylic acid (5 : 4- or 3 : 4-) is diazotised and coupled with *p*-cresol, quinol monomethyl ether, *m*-4-xylol, *p*-hydroxydiphenylmethane, *p*-acetamidophenol, 6-chloro-*m*-cresol, or other *p*-substituted, non-carboxylated, monocyclic phenols, to give chromable dyes for wool.

C. HOLLINS.

Mordant disazo dyes. J. R. GEIGY SOC. ANON. (J. R. GEIGY A.-G.) (B.P. 306,448, 8.1.29. Ger., 20.2.28).—Arylamine-mono- or -di-sulphonic acids containing no chelate groups are diazotised and coupled with

α -naphthylamine or Cleve acid, then rediazotised and coupled with a salicylic acid to give chrome-printing browns. Examples are: Laurent acid \rightarrow Cleve acid \rightarrow salicylic acid; α -naphthylamine-4 : 7-disulphonic acid \rightarrow Cleve acid; α -naphthylamine \rightarrow salicylic acid.

C. HOLLINS.

Manufacture of [green] polyazo dyes. I. G. FARBENIND. A.-G. (B.P. 300,987, 21.11.28. Ger., 22.11.27).—An aromatic amine is diazotised and coupled, through a suitable α -naphthylamine-middle component, with a cyanuric compound, $\text{X} \cdot \text{Cy}(\text{A}) \cdot \text{R}$, in which A is a 1 : 8-aminonaphthol residue (substituted or not), R is an aminoazo residue, and X is halogen or an amino-, thiol, or ether residue. Examples are: *m*-4-xylydine-5-sulphonic acid \rightarrow 2-ethoxy-Cleve acid \rightarrow condensation product from cyanuric chloride and H-acid, *p*-aminobenzenesalicylic acid, and aniline or H-acid (green on cotton); *p*-aminoacetanilide-*m*-sulphonic acid \rightarrow 1-amino-2-naphthol β -sulphatoethyl ether \rightarrow condensation product from cyanuric chloride and ethyl-H-acid, 4-amino-4'-hydroxyazobenzene-2 : 3'-dicarboxylic acid, and thio-*p*-cresol (green); 4-chloroaniline-2-sulphonic acid \rightarrow 2-methoxy-Cleve acid \rightarrow condensation product from cyanuric chloride and H-acid, *o*-nitro-*p*-toluidine, and dimethylamine, reduced, \rightarrow 1-*o*-carboxyphenyl-3-methyl-5-pyrazolone (yellowish-green). Many other cyanuric coupling components are mentioned.

C. HOLLINS.

Manufacture of o-hydroxyazo dyes and of metal compounds therefrom. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,208, 7.8.28).—A sulphonated *o*-aminophenol is diazotised and coupled with *p*-substituted phenols; the dyes may be converted into metal compounds. Examples are: 4-nitro-*o*-aminophenol-6-sulphonic acid \rightarrow *m*-4-xylol (yellow-brown on wool, deeper brown when chromed); 4-chloro-*o*-aminophenol-6-sulphonic acid \rightarrow ψ -cumenol (brown-red on wool, dark brown when chromed). Carboxylated components are excluded, as are aminophenols and naphthols as coupling components. [Stat. ref.]

C. HOLLINS.

Manufacture of [azo] dyes containing metal. SOC. CHEM. IND. IN BASLE (B.P. 297,003 and 325,204, 10.9.28. Switz., 10.9.27).—(A) A disazo or polyazo dye is treated with a solution containing several metals, such as chromium, nickel, copper; or an azo dye is converted into a metal compound and then coupled with a diazo component, or the metal compound is diazotised and coupled with a coupling component, in either case further metals being introduced, if desired. Examples are: 4-chloro-*o*-aminophenol (1 mol.) \rightarrow di-J-acid, pre-coppered, coupled with diazotised 4-chloro-*o*-aminophenol, and further treated with copper sulphate; 3 : 3'-dichlorobenzidine \rightarrow 2 mols. of J-acid in acid solution, coupled alkaline with 2 mols. of diazotised 4-chloro-*o*-aminophenol, and treated with copper sulphate and chromium fluoride solution. (B) Chromable monoazo dyes having J-acid or an *N*-substituted J-acid as end-component are treated with a number of metal solutions; e.g., 5-nitro-*o*-aminophenol \rightarrow phenyl-J-acid is boiled with copper sulphate, nickel sulphate, and chromium fluoride to give a pure blue dye for cotton.

C. HOLLINS.

Solutions of azo dyes in drying oils. J. R. GEIGY, A.-G. (B.P. 301,726, 6.11.28. Ger., 3.12.27).—Non-sulphonated dyes derived from tetrazotised diamino-diarylmethanes are easily soluble in linseed oil and produce a quick-drying printing oil. Examples are: diamine from benzaldehyde and *p*-xylidine \rightarrow 2 mols. of β -naphthol; diamine from benzaldehyde and *o*-toluidine \rightarrow 2 mols. of *p*-cresol; diamine from *o*-chlorobenzaldehyde and *p*-xylidine \rightarrow 2 mols. of phenylmethylpyrazolone etc. C. HOLLINS.

Manufacture of sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,519, 20.11.28. Addn. to B.P. 315,910; B., 1929, 309).—Halogenated dinaphthylene dioxides are heated with sulphur, with or without sodium sulphide, to give brown dyes; deca-chlorodinaphthylene dioxide yields a grey. C. HOLLINS.

Manufacture of acid wool dyes. I. G. FARBENIND. A.-G. (B.P. 299,721, 29.10.28. Ger., 29.10.27).—A sulphamic or a sulphonated amino-derivative of naphthalic anhydride is condensed with ammonia or a primary amine (including *o*-diamines); or the sulphamic or sulphonie group is introduced into the product from naphthalic anhydride. The sulphamic acid obtained from 4-aminonaphthalic anhydride, chlorosulphonic acid, and pyridine is heated with *o*-phenylenediamine to give an orange-yellow wool dye. Instead of the diamine there may be used ammonia (yellow) or aniline (greenish-yellow). The product obtained by sulphonating 4-aminonaphthalic anhydride with oleum at 50–60° gives with *o*-phenylenediamine an orange wool dye. A yellow results from sulphonation of 4-aminonaphthalimide with oleum at 110°. C. HOLLINS.

Manufacture of dyes of the safranine series. I. G. FARBENIND. A.-G. (B.P. 300,178, 6.11.28. Ger., 7.11.27).—A 4-acylamino- or 4-nitro-3'-amino-diphenylamine-2-sulphonic acid and a *p*-diamine are oxidised together, and, if desired, the acyl group is removed by hydrolysis or the nitro-group is reduced. The preparation is described of safranines from: 3'-amino-4-acetamido-4'-methyldiphenylamine-2-sulphonic acid and *p*-aminodiethylaniline-3-sulphonic acid (hydrolysed, reddish-violet) or 4-amino-4'-methoxydiphenylamine-2-sulphonic acid (hydrolysed, violet); 4-nitro-3'-dimethylaminodiphenylamine-2-sulphonic acid (reddish-blue) or 4-amino-4'-methoxydiphenylamine-2-sulphonic acid (blue). C. HOLLINS.

Derivatives of dibenzanthrone etc. (B.P. 325,525). **Derivatives of pyranthrone** (B.P. 325,250).—See III. **Colouring photographic prints** (G.P. 458,989). **Pictures from dyes** (B.P. 298,979).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Kendyr [seed-hair] down. N. L. KARAVAIEV and P. N. ODINZOV (Papier-Fabr., 1930, 28, 133–136).—The properties of the Kendyr (*Apocynum venetum*) seed-hairs have been investigated in order to ascertain whether the material is suitable for use in the cellulose industries. The hairs are very soft, of mean thickness 14 μ , wall-thickness about 2 μ , of round cross-section, very brittle, and have little tensile strength and extensibility. On chemical analysis they were found to

contain (%): moisture 11.9, ash 2, protein 2.4, wax, fat, etc. 2.7, pentosan 20.8, lignin 30.4, pure cellulose 37%. On boiling with sodium hydroxide under pressure for times up to 6 hrs. they give 25–44% of very impure technical cellulose (on the wt. of dry material). Comparison is made between the properties of this and of other cellulose materials such as straw, wood, cotton, etc., and it is found that the down resembles straw in its cellulose content and yield of soda-cellulose. It is useless for the manufacture of cellulose nitro-products, and though it might be of use in some ways, e.g., as heat-insulating material, its prospects are not good. On the other hand, a certain future may be predicted for the true Kendyr fibres from the stem of the plant as raw material for textiles, and perhaps for paper.

B. P. RIDGE.

Digestion of maize stalks with neutral sulphites. E. HORVÁTH (Papier-Fabr., 1930, 28, 239–241).—Small-scale digestions of maize stalks with sodium sulphite are described. The product obtained by digestion with a 10–20% solution of the salt differs only slightly from that obtained with water alone, and the use of a 30–40% solution does not yield a much improved product. The addition of caustic soda gives a somewhat brighter product with a lower lignin content. The ash of all products is high, and the pentosan content is not appreciably affected by variation of the concentration and temperature of digestion. The best product obtained is much inferior to that obtained by the usual process of alkaline digestion. T. T. POTTS.

Structure of the vegetable cell-membrane. M. LÜDTKE (Papier-Fabr., 1930, 28, 129–133).—The cellulose lamella of vegetable fibres is divided by membranes into co-axial cylinders, and the latter are subdivided by other radial membranes into strips which are often inclined at a small angle to the axis of the fibre and produce a spiral structure. The strips thus formed are themselves built up of fibrils. At intervals along the fibre these membranes are joined to transverse elements. The membranes and transverse elements consist of non-cellulose material, which is probably a furfuroid. The bead-necklace effect observed on treating vegetable fibres with cuprammonium, shortening of the fibres in mercerisation, absorption and swelling phenomena, increased absorption and attack by chemical reagents at mechanically damaged places, etc. are shown to result from this structure. The effects of different conditions of nourishment during growth on the characteristics of plants are shown by study of the straw from wheat plants grown under various conditions of phosphorus, potash, and nitrogen manuring. Excess of nitrogen or of phosphorus causes an increase in the percentage of α -cellulose obtained from the dry material. Variations in conditions of nourishment or environment account for differences in the properties of pulps prepared from the same kind of plant under identical conditions. B. P. RIDGE.

Cooking-heat and its economical use in cellulose works. W. SCHMID (Papier-Fabr., 1930, 28, 138–141).—Further economy lies in making the most of the cooking-heat and in shortening the time of circulation in the cooker by using preheated fresh liquor. The

Waldhof works use the cooking-heat directly for heating the fresh liquor, but the economy thus effected is counterbalanced by increased cost of plant, since large liquor reservoirs are necessary and preheating must be effected under pressure in acid-resistant vessels. Further, the liquor is heated only to about 70°, instead of to 105°, which is necessary if the time required for bringing it to the boil is to be reduced to zero. Heat requirements and costs involved in producing the desired conditions are discussed, and it is shown that by the use of a steam reservoir and two steam ejectors, used instead of the liquor pumps, the liquor may be heated to the required temperature (105°) in two stages, without any great increase in cost of plant. A fuel economy of 18% and shortening of the time of circulation for the cooker of 25%, *i.e.*, twice the corresponding values obtained by the Waldhof method, are claimed.

B. P. RIDGE.

[Heat]-insulating board from straw. A. G. GIBSON (Ind. Eng. Chem., 1930, 22, 223—226).—Only a very small proportion of the straw produced in the U.S.A. is put to any useful purpose, the main problem being collection cost. A plant manufacturing insulating board from straw came into production in 1928. Straw is cut to short lengths, sprayed with water, and digested with water under 60—75 lb. steam pressure for 8 hrs. The cooked straw is drained, seasoned, and disintegrated. Water is added and the mixture passed to pulp grinders consisting of rotating discs of high-silicon iron. These last 3—4 weeks; other steels are worn out in a few hours by the abrasive action of the straw. The pulp, with water, alum, and resin size added, is beaten and passed to the forming machines and mixed with 5—10% of old newspapers to give the necessary fine aggregate. The board is formed on a cylinder machine, pressed to remove some of the water, and dried. The dried board as it leaves the dryer is sprayed with concentrated liquor from the digesters; this contains furfuraldehyde and resin, and acts as a preservative and binder. Straw board has a heat conductivity only slightly greater than that of cork.

C. IRWIN.

Sugar-factory filter-cloth. HONG.—See XVII.

See also A., Apr., 417, Behaviour of cotton cellulose and regenerated cellulose in sodium hydroxide solution (NEALE). 418, Elastic properties of wool in organic liquids (SPEAKMAN).

PATENTS.

Manufacture of hydrophilic cotton wool. A. CHARBONNEAU (F.P. 633,719, 11.9.26).—The raw cotton is boiled with caustic soda and lime, washed, treated with sodium hyposulphite solution at 80°, washed, and bleached with chlorine in very dilute sulphuric acid solution. The product is then again treated with sodium hyposulphite solution and finally immersed in sulphuric and nitric acids.

A. R. POWELL.

Improvement of cellulose raw material. ZELLSTOFFFABR. WALDHOF, V. HOTTENROTH, and O. FAUST (B.P. 301,088, 23.11.28. Ger., 24.11.27).—Cellulose raw material is treated with 9—18% caustic alkali solution, and, after removal of the excess of the latter

by pressure, is allowed to ripen for a period and at a temperature in accordance with the desired reactivity and is afterwards washed. [Stat. ref.] F. R. ENNOS.

Treatment of cellulose material. EUROMERICAN CELLULOSE PRODUCTS CORP., Assees. of B. DORNER (B.P. 312,634, 23.4.29. U.S., 29.5.28).—Maize stalks, after being leached, disintegrated, and washed, are treated with dilute alkali to remove silica (*e.g.*, with 0.1% caustic alkali at 120° and 1 atm.) and are then subjected to a second alkali treatment (*e.g.*, with 0.6% caustic alkali at 138° and 2.5 atm.), conditions in both treatments being such that the organic non-cellulosic material is not caramelised.

F. R. ENNOS.

Manufacture of viscose. I. G. FARBENIND. A.-G. (B.P. 306,971, 28.2.29. Ger., 28.2.28).—The deleterious effect of small quantities of heavy metals in the constituents used is more or less avoided by addition of 1—10 g. of oxalic acid or alkali oxalate to each litre of viscose produced, at any stage in its manufacture or when in the finished condition.

F. R. ENNOS.

Production of artificial filaments or threads. BRIT. CELANESE, LTD., H. DREYFUS, E. KINSELLA, J. BOWER, and W. I. TAYLOR (B.P. 326,232, 7.12.28. Addn. to B.P. 300,998; B., 1929, 203. Cf. also B.P. 304,674; B., 1929, 353).—A solution of cellulose acetate or other cellulose derivative is spun from a nozzle comprising a line of extrusion orifices, while a collector device, having a shape appropriate to that of the spinning nozzle, constrains substantially the whole of the evaporative medium to pass through the immediate vicinity of the orifices.

F. R. ENNOS.

Production of artificial filaments, yarns, or threads. BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and W. I. TAYLOR (B.P. 326,777—8, 13.9.28).—Regular or systematic variations in denier are imparted to the extruded filaments by (A) varying the speed of the pumps feeding the spinning solution to the nozzles, or (B) producing pulsations in the delivery of the spinning solution to the nozzles by the vibration or reciprocating action of a diaphragm or piston in contact with it. (Cf. B.P. 321,762; B., 1930, 98.)

F. R. ENNOS.

Production of artificial silk threads. G. S. WALLIS (B.P. 326,963, 21.2.29).—Spun threads of wool, cotton, etc. are subjected to a spray of cellulose solution while traversing a perforated tube which passes through a vessel containing the cellulose solution under pressure.

F. R. ENNOS.

Drawing or stretching process in the manufacture of cuprammonium artificial threads, filaments, and yarns. LONZA-WERKE ELEKTROCHEM. FABR. G.M.B.H. (B.P. 299,022, 19.10.28. Ger., 19.10.27).—Threads spun from cuprammonium cellulose solution, which have been coagulated in a strongly alkaline liquid, are treated with warm water or dilute aqueous solutions containing no free acid (*e.g.*, magnesium sulphate solution) until they become soft and rubber-like without losing their copper content; while in this condition they are stretched by suitable means to as much as twice their original length, and are finally fixed in an acid bath.

F. R. ENNOS.

Manufacture of artificial silk [from synthetic

resin]. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 303,867, 11.1.29. Ger., 11.1.28).—The raw material, consisting of a condensation product of a polyhydric alcohol (glycerin) and a polybasic acid (phthalic acid) or its anhydride together with a plasticiser if desired, is extruded under heat into the air at a temperature of 150–200° or from a solution in acetone into a coagulating bath, the filaments being subsequently baked. F. R. ENNOS.

Cellulose films. FILM OZAPHANE (SOC. INDUSTRIELLE D'APPLICATIONS PHOTOGRAPHIQUES) (B.P. 301,878, 6.12.28. Fr., 7.12.27).—Cellulose films regenerated from viscose are rendered unflammable by impregnation after manufacture with a solution of commercial ammonium phosphate (about 10% of phosphate) or one containing not less than 8% of ammonium sulphate or chloride. The films may be sensitised before, during, or after the impregnation with a mixture of diazo compounds and phenol, which can be developed by the action of ammonia etc. F. R. ENNOS.

Manufacture of cellulose derivatives. H. DREYFUS (B.P. 326,861, 23.11.28).—Cellulosic materials are treated at 140–200° with esters of organic acids in the presence of tertiary organic bases (e.g., dialkyl-anilines); the product is afterwards esterified by any suitable method. F. R. ENNOS.

Manufacture of plastic masses from cellulose derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,952, 12.2.29).—A cellulose ester of a saturated fatty acid containing at least eight carbon atoms (e.g., cellulose laurate), a solvent for the ester, and a resin are mixed with suitable fillers.

F. R. ENNOS.

Improvement of acylcellulose. I. G. FARBENIND. A.-G. (B.P. 299,326, 22.10.28. Ger., 22.10.27).—After precipitating the acylcellulose from its crude solution and washing, it is stabilised by heating up to 100° with dilute aqueous or alcoholic sulphuric acid, under such conditions that the solubility in a specific solvent is not altered. F. R. ENNOS.

Manufacture of mixed ethers of carbohydrates. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,865, 19.12.28).—Cellulose is heated at about 120° with a mixture of methyl or ethyl chloride and an alkyl chloride containing three or more carbon atoms (e.g., butyl chloride), in the presence of caustic alkali; the films produced therefrom are very little affected by water. F. R. ENNOS.

[Apparatus for] manufacture of imitation fur or the like. G. ROMANE (B.P. 314,517, 14.5.29. Fr., 30.6.28).

Production of [compound] yarns or threads of fibrous substances. A. GREEN (B.P. 326,786, 19.9.28).

Cellulose ester compositions (B.P. 297,679).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fastness of dyestuffs. BRAVO.—See IV.

PATENTS.

Bleaching of cellulose derivatives. K. ROOS (B.P. 326,471, 11.12.28).—Coloured threads and films

of nitrocellulose or cellulose acetate are rapidly bleached at 50° by means of active chlorine (chlorine gas or sodium hypochlorite) after a preliminary swelling in 1–20% acetic acid. A. J. HALL.

Manufacture of materials for use in dyeing or printing. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,563, 23.10.28).—Preparations needing no alkali for dissolution are made by mixing an alkali-starch compound with a phenolic coupling component (or its alkali salt), with or without diluents. E.g., the sodium salt of 2:3-hydroxynaphthoic *o*-toluidide or 5-chloro-*o*-toluidide is ground with 3–8% of alkali-starch; or the *p*-anisidide (30 pts.) is mixed with the diazoamino-compound from diazotised 4-chloro-*o*-anisidine and 4-sulphoanthranilic acid (70 pts.) and alkali-starch (5 pts.). C. HOLLINS.

Dyeing of viscose silk. I. G. FARBENIND. A.-G. (B.P. 301,754, 3.12.28. Ger., 2.12.27).—Uniform shades are obtained by dyeing viscose silk with secondary disazo dyes, $A \rightarrow M \rightarrow E$, in which *A* is an aromatic monoamine not containing a nitro-group or the salicylic acid grouping, *M* is a middle component, and *E* is an *ortho*-coupling naphthol or naphthylamine other than an *N*-substituted 2:8-aminonaphtholsulphonic acid. Examples are: *p*-toluidine-3-sulphonic acid \rightarrow 2-ethoxy Cleve acid \rightarrow N.W. acid (blue); aniline-*o*-sulphonic acid \rightarrow Cleve acid \rightarrow Schäffer acid (violet); *m*-aminobenzoic acid \rightarrow *o*-anisidine \rightarrow methyl- β -naphthylamine-7-sulphonic acid (violet); aniline-2:5-disulphonic acid \rightarrow 1-*m*-aminophenyl-3-methyl-5-pyrazolone \rightarrow 1:3:6-naphtholdisulphonic acid. C. HOLLINS.

Dyeing of artificial silk of regenerated cellulose. SOC. CHEM. IND. IN BASLE (B.P. 300,916, 19.11.28. Switz., 19.11.27).—Azo dye compounds containing two or more metal complexes give level shades on viscose silk. Amongst the examples are: 4-nitro-*o*-aminophenol (2 mols.) \rightarrow di-J-acid, with chromium and copper (grey); 4-chloro-*o*-aminophenol (2 mols.) \rightarrow phosphogenated J-acid, with copper and chromium (ruby); 5-nitro-*o*-aminophenol \rightarrow *o*-tolyl-J-acid with 25% of copper and 75% of chromium (blue). C. HOLLINS.

Dyeing or colouring higher fatty acids. I. G. FARBENIND. A.-G. (B.P. 299,790, 31.10.28. Ger., 31.10.27).—The fatty acids are converted partly or completely into their hydroxyalkylamine salts, especially β -aminoethyl alcohol and "triethanolamine" salts, before, during, or after application of the dye, or the dye may be pretreated with the hydroxyalkylamine; metal salts may be added to modify the colour. In the examples the following colouring matters are used with stearic acid and ceresin: benzeneazodimethylaniline (yellow); 1-di-(β -hydroxyethyl)amino-4-hydroxyanthraquinone (bluish-red); cupric chloride (green); ferric chloride (yellow); *o*-toluidine \rightarrow phenylmethylpyrazolone and cobalt nitrate (brown); Victoria-blue B; methyl-violet B; *p*-nitroaniline (2 mols.) \rightarrow H-acid, reduced, \rightarrow 2:4-tolylenediamine (2 mols.) (black); patent-blue A; alizarin-blue B; 5-chloro-*o*-aminophenol \rightarrow S-acid (violet); alizarin and aluminium chloride (bluish-red); 4:6-dichloro-*o*-aminophenol \rightarrow phenylmethylpyrazolone and cupric chloride (clear brown); thioindigo-red. C. HOLLINS.

Preparation of dyed effect threads. CHEM. WORKS, FORMERLY SANDOZ (B.P. 324,680, 31.7.28. Addn. to B.P. 280,493; B., 1928, 812).—Cellulose dyed with direct dyes suitably resistant to acetylation is mono- or di-acetylated by the process of the prior patent, and may then be used for effect threads since the dyeing has become nearly as fast as vat dyeings to washing and cross-dyeing. C. HOLLINS.

Production of pattern effects upon textile fabrics or other materials. BRIT. CELANESE, LTD., G. H. ELLIS, and W. B. MILLER (B.P. 324,650, 29.9., 3.11., and 3.11.28, and 29.4.29).—In the colouring of textiles, especially of acetate silk, by impregnation with amines followed by diazotisation on the fibre and development with a coupling component, resist or discharge effects are obtained by local application of chlorates, chromates (or other oxidising discharges), sulphonylates, hyposulphites (or other reducing discharges), which prevent diazotisation of the amine. For coloured discharge effects a colour which resists the particular discharge applied is added; e.g., for acetate silk, acetylated aminoanthraquinones. Amongst the examples are: acetate silk is padded with 5-nitro-*o*-anisidine, printed with sodium chlorate, diazotised and coupled with 2:3-hydroxynaphthoic acid for a white pattern on a pink ground; acetate silk is padded with dianisidine, printed with Durindone-red Y, alkali, anthraquinone, hyposulphite, and Formosul, steamed, diazotised, and developed with 2:3-hydroxynaphthoic acid for a red pattern on a navy-blue ground. C. HOLLINS.

Colouring of textile materials [resist effects with oxidation dyes]. BRIT. CELANESE, LTD., G. H. ELLIS, and W. B. MILLER (B.P. 324,683, 24.8.28).—A reducing agent, e.g., formaldehydesulphoxylate, hyposulphite, or an oxalate, is applied locally before or after the amine to be oxidised on the fibre. A coloured resist is obtained by adding a vat dye to the printing paste. The process is particularly suitable for acetate silk. C. HOLLINS.

Manufacture of discharge basic dyeings [with synthetic mordants]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 325,562, 23.10.28).—Basic dyeings on a synthetic mordant are discharged by means of oxidants, e.g., sodium chlorate. C. HOLLINS.

Manufacture of non-dyeing mordants, and dyeing of basic dyes on cotton. G. B. ELLIS. From CHEM. WORKS, FORMERLY SANDOZ (B.P. 325,388, 13.2.29).—A phenol containing no nitrogenous groups is boiled under reflux with an aqueous suspension of sulphur and lime for 30–50 hrs. The precipitated calcium salt is converted into the sodium salt and applied as mordant for basic colours. C. HOLLINS.

Printing fabrics in [ice] colours on aniline-black. I. G. FARBENIND. A.-G. (B.P. 305,476, 4.2.29. Ger., 2.2.28).—Fabric padded as usual for aniline-black, e.g., with aniline, aniline hydrochloride, ferrocyanide, and chlorate, is dried and printed with a coupling component (an arylamide of 2:3-hydroxynaphthoic or acetoacetic acid), the printing paste containing zinc oxide and sufficient caustic alkali to neutralise the acid of the aniline padding. The fabric is then steamed

in a Mather-Platt and the resist is developed by passing the cloth through a diazo solution. Examples are: β -naphthol and 2:5-dichloroaniline (yellow-orange); bisacetoacetylolidine and 2:5-dichloroaniline (yellow); 2:3-hydroxynaphthoic anilide and 6-chloro-*o*-toluidine (orange); the *o*-toluidide and 4-chloro-*o*-toluidine (red), phenyl 4-hydroxy- α -naphthyl ketone and *m*-chloroaniline (orange). C. HOLLINS.

Manufacture of vegetable [immunised] effect threads. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 322,556, 15.9.28).—Sodium cellulose is treated with an aromatic or heterocyclic sulphonyl chloride of basic character, e.g., dimethylaniline-*p*-sulphonyl chloride, *m*-nitrobenzenesulphonyl chloride (followed by reduction with stannous chloride), etc. Resistance to ironing and fastness to alkali are claimed for the products. C. HOLLINS.

Preparations for stiffening and sizing textile yarns and fabrics. A. F. GALVIN (B.P. 302,358, 12.12.28. Fr., 15.12.27).—A sizing preparation which dries very rapidly and allows the production of a matt or semi-lustrous finish which does not break, yellow, or harden with age comprises a solution in benzene of a wax, stearine, an animal fat, a benzene-soluble soap, and a resin soluble in benzene, or gum dammar, or carnauba wax. A. J. HALL.

Mothproofing composition. R. M. RITTER (B.P. 327,009, 25.3.29. U.S., 31.10.28).—Animal fibres are impregnated with a solution containing alkaloids extracted from the seeds of *Strychnos Nux Vomica* or *Strychnos Ignatii*, together with saponins of quillaia, metal mordants, and phenyl salicylate. A. J. HALL.

[Apparatus for] cleaning of garments and the like. CARRIER ENG. CO., LTD., and K. J. R. ROBERTSON (B.P. 325,646, 31.12.28. Addn. to B.P. 322,797; B., 1930, 197).—The apparatus described in the prior patent is provided with an additional vacuum plant in which textile materials, after treatment with the solvent and while being carried on a brattice, are brushed by rotary brushes and the loosened dirt is removed by suction. A. J. HALL.

Dyeing machines. A. J. HALL (B.P. 327,168, 30.11.28).

Machines for treating fabrics by drying, dry-cleaning, dyeing, or washing. H. PIERCE (B.P. 311,222, 15.12.28. U.S., 7.5.28).

Apparatus for printing on fabrics. T. BROWN (MANCHESTER), LTD., and T. BROWN (B.P. 327,088, 9.7.29).

Products from fatty acids (B.P. 295,024).—See XII.
Decoration of fabrics (G.P. 460,309).—See XXI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Action of sulphur dioxide on natural phosphates. G. CALCAGNI (Annali Chim. Appl., 1930, 20, 74–79).—The action of sulphur dioxide on finely-divided tricalcium phosphate, apatite, and pebble and Kosseir phosphates, moistened with water, gives products in which up to 99.12, 32.32, 35.75, and 55.37% of the

total phosphoric acid is in the soluble form; if the phosphates are heated during the treatment, the degree of solubilisation is lowered considerably. This treatment may serve to replace the more expensive treatment with sulphuric acid for the manufacture of phosphatic fertilisers.

T. H. POPE.

Volatilisation of phosphorus from phosphate rock. R. O. PIKE (Ind. Eng. Chem., 1930, 22, 242—245).—Experiments on the volatilisation of phosphorus (as P_2O_5) from a mixture of Idaho phosphate rock, sand, and carbon at temperatures below the fusion point showed that addition of carbon raised the fusion point from 1300—1400° to above 1650°. Both carbon and silica are necessary for a satisfactory reaction, though not in any very definite proportion. Effects of time, temperature, and grain size were investigated. These experiments were made in crucibles. An attempt to repeat the results in a rotary kiln failed at 1300—1400° and the phosphorus was only driven off by the use of an oxygen-enriched blast giving a temperature of about 1700°. A viscous slag was then formed, but the temperature is too high for any lining to withstand. The failure of the kiln at the lower temperature is ascribed to the action of the carbon dioxide from the combustion of the oil fuel used penetrating the porous charge and producing the endothermic reaction $C + CO_2 = 2CO$.

C. IRWIN.

Method of analysis of calcium citrate to replace the official method. G. ROMEO and N. SCIACCA (Annali Chim. Appl., 1930, 20, 80—90).—In view of the divergent and erroneous results yielded by the official method of analysis used for trading purposes, the author suggests the use of the following procedure for the analysis of calcium citrate. The sample is decomposed by sulphuric acid, the pectic substances and calcium sulphate being then precipitated by alcohol. In the aqueous alcoholic solution determinations are made of (1) the total acidity; (2) acidity due to the excess of sulphuric acid by the benzidine process; and (3) acidity due to hydrochloric and phosphoric acids from the chlorides and phosphates present—colorimetrically. The content of citric acid is obtained by difference.

T. H. POPE.

Separation of iodine by the thiosulphate method. J. J. DE UGARTE (Caliche, 1929, 11, 49—51; Chem. Zentr., 1929, ii, 2484—2485).—The reduction of sodium iodate to iodine by means of sodium thiosulphate and sulphuric acid (*ibid.*, 1927, 8, 515) is best effected at 22° and is technically advantageous in that it avoids nitrous fumes and surface losses of iodine.

A. A. ELDRIDGE.

[Helium] inflation of the metal-clad airship ZMC-2. A. R. CARR and A. C. GOOD (Ind. Eng. Chem., 1930, 22, 227—230).—This airship is constructed of "Alclad" alloy sheets, 0.0095 in. thick, the seams of which are sealed with bituminous material and riveted to a supporting frame. Owing to the difficulty of separating helium from air, the air was first displaced upwards by carbon dioxide and then the latter downwards by helium. The carbon dioxide-helium mixture was passed through a caustic soda scrubber and the helium returned. As no dryer was used the final gas was saturated with

water vapour. This was removed by recirculating the gas through the scrubber converted into a dryer and charged with solid caustic soda. Blankets prevented the entrainment of the latter. The final gas contained over 92% He and 0.67% CO_2 . 100 Cub. ft. of helium per 24 hrs. are required to compensate for outward leakage through the hull. There is no inward leakage of air.

C. IRWIN.

Decomposition of sodium sulphate by silica. BOGITCH. See VIII. **Conductivity of chromic acid solutions.** CHERRY. See XI. **Solution of arsenious and mercuric iodide.** ACTON. See XX.

See also A., Apr., 423, **Electrolytic production of hydrogen** (BAARS). 430, **Sulphuric acid catalysis** (NEUMANN and JÜTTNER). 435, **Crystalline sodium sulphide** (PECKER). **Extraction of rubidium and caesium from carnallite** (JANDER and BUSCH). 438, **Preparation of pure hafnium salts** (DE BOER and BROOS). **Preparation of pure nitric acid** (MISH-SCHENKO). **New phosphonium salt** (ÉVRARD). 498, **Toxicity of lead compounds** (BUCK and KUMRO).

PATENTS.

Preparation of chemicals in a granular form. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of E. GEISEL (G.P. 453,366, 6.3.25. Addn. to G.P. 424,193).—The material is fused and agitated with a proportion of an unfused substance. In examples, sodium sulphide is melted in its water of crystallisation and the liquid is agitated with a quantity of the anhydrous salt while cooling, fused potassium nitrate is stirred with calcium phosphate to obtain a granular mixed fertiliser, or fused borax is mixed with about 1% of wood charcoal to obtain a flux for soldering.

A. R. POWELL.

Manufacture of ammonia salts. "MONTECATINI" SOC. GEN. PER L'IND. MINERARIA ED AGRICOLA, Assees. of G. FAUSER (B.P. 313,446, 10.6.29. It., 11.6.28. Addn. to B.P. 292,129; B., 1929, 978).—The vapour formed by the reaction of the atomised acid in an atmosphere of ammonia is removed by passing it through the acid in the tank from which the atomiser is supplied, thus retaining the ammonia contained therein and avoiding the use of a fan and rectifying column.

H. ROYAL-DAWSON.

Recovery of alkali phosphate and nitrate separately from solutions containing both. [Decomposition of phosphate rock.] I. G. FARBENIND. A.-G., Assees. of R. GRIESSBACH, K. RÖHRE, R. GOLDBERG, and K. O. SCHMITT (G.P. 459,187, 20.11.25).—The rock is treated with nitric acid and an alkali sulphate simultaneously or successively and the filtered solution is neutralised to the monoalkali phosphate stage, whereby the greater part of the alkali phosphate separates on cooling. The mother-liquor is either acidified with nitric acid or treated with an excess of alkali carbonate, evaporated, and cooled to recover alkali nitrate.

A. R. POWELL.

Preparation of hydrogen or hydrogen-nitrogen mixtures. H. BOMKE (G.P. 460,422, 14.12.26).—Coal gas and steam are passed over a catalyst to obtain a mixture of hydrogen and carbon monoxide and, after addition of sufficient oxygen or air to convert the latter

into dioxide, the gases are passed first over heated dolomite, then through an absorbent for carbon dioxide. The resulting mixture of hydrogen and nitrogen is suitable for the manufacture of synthetic ammonia.

A. R. POWELL.

Liquation of sulphur etc. (B.P. 325,948).—See I. Internal-combustion engines (B.P. 325,866). Ammonia and hydrogen sulphide from gases (B.P. 299,765). Hydrogen from gaseous mixtures (B.P. 325,968).—See II. Metallurgical furnace (U.S.P. 1,742,441).—See X. White lithopone (B.P. 325,641).—See XIII. Material containing calcium nitrate (B.P. 308,717). Fertilisers (B.P. 326,814 and 326,529).—See XVI. Self-combustible mixtures (B.P. 299,396).—See XXII.

VIII.—GLASS; CERAMICS.

Effect of continued remelting of cullet in sillimanite and fireclay vessels. A. A. CHILDS, V. DIMBLEBY, H. W. HOWES, and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 296—303 T).—The repeated remelting at 1400° in open pots in a gas-fired furnace of a glass of initial approximate composition SiO_2 74%, CaO 8.5%, Na_2O 16.5%, ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) as impurity, caused a lowering of the alkali content in one series of nearly 1.5% after 9 meltings. This represented an actual loss by volatilisation as contrasted with the "loss" due to the fall in percentage values as more and more iron and aluminium oxides were taken up from the pot. Fireclay pots behaved similarly, but the pot attack was much more pronounced than in the case of the sillimanite pots. These facts relative to the corrosion of pots are considered noteworthy as this was usually ascribed to melting batch rather than to the attack of the molten glass itself, and in these meltings batch was used to prepare only the first lot of glass, which was then melted and remelted for subsequent trials. M. PARKIN.

Effect on properties of soda-lime-silica glass of continued remelting in platinum. V. DIMBLEBY, H. W. HOWES, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1929, 13, 304—321 T).—The effect of pot corrosion was cut out by making 9 remelting tests, similar to those dealt with in the preceding abstract, in platinum vessels heated in an electric furnace. Careful analysis showed that, save for a very slight tendency for the alkali content to fall, there was no certain evidence that any change in chemical composition was thus caused. The densities, determined by the powder method in a sp. gr. bottle and also by a flotation method, using bromoform and pentachloroethane, and the expansions and annealing temperatures, all showed a similar constancy within the (narrow) limits of experimental error. It is pointed out that with such small samples no test of brittleness or "working range" could be made. M. PARKIN.

Devitrification of glass. I. Relative stabilities of different glasses. II. Time-temperature curves of visible devitrification. III. Proposed classification of glasses. C. W. PARMELEE and A. J. MONACK (J. Soc. Glass Tech., 1929, 13, 322—336 T).—I. A number of glasses representative of various types were slowly cooled after heating in crucibles of 15 c.c. capacity

to approx. 1100°, the process taking 12—15 hrs. They were then examined for crystal formation. If necessary the heating and cooling were repeated till devitrification set in. In lead optical glasses a silica content greater than 50—55% appeared to be dangerous (contrast Peddle's 65%). A crown glass containing SiO_2 20.2, CaO 13.4, MgO 1.1, alkalis 14.9%, was unexpectedly stable, as 5 thermal treatments did not cause devitrification. A high-zinc oxide glass and a good bottle glass were also of good stability, whilst Fourcault window glass, plate and wire glass, and a light barium crown optical glass devitrified with only one treatment. A borosilicate crown optical glass, although devitrified on the surface, was quite clear inside, even after several treatments, and pyrex glass was practically completely devitrified as a result of a single treatment.

II. Time-temperature curves of visible devitrification were taken, the glass being wrapped in platinum foil suspended by the thermocouple 2—3 mm. below the junction. The quenched samples were viewed under a magnification of 380 diam. to find crystals; if none was found a fresh sample was heated at the same temperature for a longer time and so on till devitrification did set in. These time-temperature curves taken for a light flint optical glass, plate glass, and wire glass showed the value of the method.

III. It is suggested that the area of the rectangle lying between the 0 and 2 hrs. ordinates and 600° and 1200° abscissæ, and outside the curve be taken as a measure of the tendency to devitrify. The greater this area the less is the tendency, provided the curves are plotted to a standard time and temperature scale. A devitrification constant $V = A/(a \times b)$, where A is the area referred to, a the linear measure in cm. of 100° on the temperature axis, and b that in cm. of 1 hr. on the time axis, is suggested as a means of classification. On this basis glasses of class (i) would have $V = 12$ or less, of class (ii) $V = 12$ —30, and of class (iii) $V =$ above 30. M. PARKIN.

Velocity of crystallisation of soda-lime-silica glasses. A. DIETZEL (Sprechsaal, 1929, 62, 506—509, 524—525, 543—544, 562—568, 584—585, 603—604, 619—621, 638—639, 657—660; Chem. Zentr., 1929, ii, 2487—2488).—The speed of crystallisation is not affected by the thermal history. The curve of the speed is acute for the "short" glasses rich in lime, and flatter for "long" glasses rich in silica. In glasses containing 18% Na_2O , calcium oxide up to 8% has a favourable effect on the vitreous property; for 12% Na_2O the favourable effect of lime is observed up to 12%. In a spatial conception of the maximum crystallisation speed isotherms there is a "valley" extending from 18% Na_2O , 9% CaO to 12% Na_2O , 13.7% CaO ; the equation of this line is $y = 23.3 - 0.79x$, where x is the content of soda and y that of lime. A corresponding line on the devitrification time isochronal diagram is $y = 27.7 - (62x - x^2 - 422)^{\frac{1}{2}}$ A. A. ELDRIDGE.

Velocity of crystallisation of soda-lime-silica glasses. K. TABATA (J. Soc. Glass Tech., 1929, 13, 350 T). E. ZSCHIMMER (*Ibid.*, 350—351).—Zschimmer's results (B., 1929, 472) claiming that alteration of the conditions, e.g., time of heating, alters the nature of

the results obtained are criticised. Tabata suggests that longer heating periods at the lower temperatures should be employed.

In reply ZSCHIMMER refers Tabata to the more detailed work (Zschimmer and Dietzel, *Sprechsaal*, 1927, 58, 110), with which Tabata's results were not in disagreement. A more important point was that recently much higher equilibrium temperatures than those given by Morey and Bowen or by Dietzel had been obtained. Zschimmer considers it important to decide whether devitrification should be induced under falling or rising temperature conditions. M. PARKIN.

X-Ray investigation of feldspar glasses. G. L. CLARK and C. R. AMBERG (*J. Soc. Glass Tech.*, 1929, 13, 290—296 r).—Samples of clear silica glass and fused soda feldspar were subjected to X-ray analysis using a lead button to exclude the main beam and so cut out the halation which obscured the rings close to the centre. In every case, no matter at what temperature between 1165° and 1700° the feldspar was fused, two diffuse rings were obtained, the inner being the more intense. The values found denoted a silica molecule 7·15 Å. long, as against 6·42 Å. from the values of Pauling and Goldschmidt, and 2·52 Å. wide as against 2·80 Å. The molecules were arranged in chains with gaps 0·222 Å. between each. The addition of other oxides as in feldspar glass caused a modification of this arrangement resulting in decreased spacings. M. PARKIN.

Diffraction of X-rays by ordinary glass subjected to various treatments. C. W. PARMELEE, G. L. CLARK, and A. E. BADGER (*J. Soc. Glass Tech.*, 1929, 13, 285—290 r).—The X-ray patterns of various commercial glasses were photographed to see whether sharp line patterns were produced. Selenium and copper rubies showed only diffuse rings, as did also a cryolite opal glass. Devitrified pyrex glass gave well defined cristobalite lines, whilst glasses coloured by materials in true solution, viz., amber (Fe_2O_3), blue (CoO), green ($\text{CuO} + \text{Cr}_2\text{O}_3$), gave only diffuse ring patterns. When a piece of soda-lime glass (from a bottle-glass tank), which exhibited the blue opalescence of incipient devitrification, was tested it gave the diffuse ring, but when heat-treated to produce definite crystals and again tested, the same ring was present plus two annuli bounding this, which with the sharp lines formed the pattern of the devitrification product. In consequence, diffuse rings were taken as a sign of incipient crystal formation. Next, a bead of soda-lime glass in a furnace was tested as the temperature was progressively raised in 100° steps to 1300°, with 12-hr. exposures, but no material change in pattern and no devitrification took place. Compressing the specimens did not produce patterns. The X-ray method was thus not suitable for detecting strain in opal glasses. M. PARKIN.

Coloration of glass by X-rays. C. W. PARMELEE, G. L. CLARK, and A. E. BADGER (*J. Soc. Glass Tech.*, 1929, 13, 279—285 r).—Published work dealing with the coloration of glasses by X-rays and those from radium is reviewed, and the results of experiments in which borax beads containing a series of 45 substances to be tested were subjected to the radiation from a tungsten target X-ray tube (70,000 volts, 4 ma.) for 33½ hrs. are

tabulated. Of the compounds tested only those of chromium, lead, thallium, bismuth, and manganese gave pronounced changes of colour, these yielding brownish-yellow, brown, dark brown, orange-yellow, and violet hues respectively. M. PARKIN.

Manufacture of blue glass and the decomposition of sodium sulphate by silica. B. BOGITCH (*Compt. rend.*, 1930, 190, 794—796).—The fusion of sodium sulphate and silica results in the formation of two superposed liquid layers of the molten salts containing small quantities of silica and sulphuric acid, respectively. The sodium sulphate (upper) layer is destroyed above 1250°, especially if air, nitrogen, or carbon dioxide is bubbled through the melt, whilst carbon dioxide containing more than 10% CO produces coloured (yellow to orange) glasses owing to the progressive transformation of sulphate into sulphur. Addition of sufficient iron silicate to produce a glass containing 0·75% Fe gives a blue glass with 9·5% CO, and a green glass with more than 12·5% CO. The fact that blue glasses are not always obtained in practice under these conditions is due to the slow rate of reaction (cf. A., 1929, 1409). J. GRANT.

Influence of chemical composition on the physical properties of glazes. F. P. HALL (*J. Amer. Ceram. Soc.*, 1930, 13, 182—199).—A study was made of the factors affecting that type of crazing which is caused by unequal expansion or contraction of body and glaze. The physical properties of a large number of glazes in the form of homogeneous glass rods were determined, and a method is indicated for calculating from the chemical composition the approximate values for tensile strength, modulus of elasticity, and the mean coefficient of linear expansion of the glaze. F. SALT.

Possible use of certain California clays in vitreous china sanitary-ware bodies. W. F. DIETRICH and W. W. MEYER (*J. Amer. Ceram. Soc.*, 1930, 13, 202—217).—The possibility of substituting Californian clays for English china clay and ball clay in a standard sanitary-ware body composed of 32·5% of English china clay, 7·5% of English ball clay, 10% of Tennessee ball clay, 25% of Californian feldspar, and 25% of silica was studied. Chemical and physical data on the various clays used are given. Five reasonably good bodies were developed. F. SALT.

Determining the water content of clays. C. L. DEEDS (*J. Amer. Ceram. Soc.*, 1930, 13, 200—201).—Four kg. of water are added to 2 kg. of a representative sample of the clay being tested, and the mixture is blunged. A 250-c.c. flask is filled with this slip and weighed. The amount of dry clay is found from the equation: $D = (P - 250)[G/(G - 1)]$, in which D = wt. of dry clay, P = wt. of slip, and G = sp. gr. of dry clay. From this the percentage of water in the clay is readily obtained. F. SALT.

Rapid method for determination of moisture in grog, semi-porcelain pastes, and granulated blast-furnace slag. P. P. BUDNIKOV (*Chem.-Ztg.*, 1930, 54, 202—203).—The material (20 g.) is heated at 110° in a container through which a current of dry air can be aspirated by means of a filter pump.

H. F. HARWOOD.

Refractory cements. McMULLEN.—See IX. **Spalling furnace.** BAUMANN.—See XI. **Clay formation and the weathering of felspars.** TAMM.—See XVI.

See also A., Apr., 443, **Determination of alkalis in silicates** (MALJAROV).

PATENTS.

Stratified bodies such as strengthened glass. TRIPLEX SAFETY GLASS CO., LTD., W. R. LYTTLETON, J. WILSON, and H. W. DICK (B.P. 326,229, 5.12.28, 18. and 27.3.29).—A sheet of glass is coated on its unexposed side first with gelatin and then with a celluloid enamel, and, after softening this surface and also that of a sheet of transparent cellulose ester by spraying with a liquid solvent or plasticiser, the two sheets are firmly united by pressure and heat.

F. R. ENNOS.

Manufacture of strengthened glass. TRIPLEX SAFETY GLASS CO., LTD., and J. WILSON (B.P. 326,259, 13.12.28).—Two or more sheets of glass are treated on their unexposed surfaces with a water-soluble coating, e.g., gelatin, with or without a further coating of celluloid enamel, and are firmly united to an interposed transparent layer of celluloid by removing the coating from the marginal portions, applying a plastic luting material such as a solution of rubber and/or an ester gum in a solvent which is also a solvent for the central layer, and subjecting the whole to pressure.

F. R. ENNOS.

Continuous kilns of the Hoffman type. F. NOVELLI (B.P. 309,547, 11.4.29. It., 12.4.28).—The heat is provided by solid fuel burnt among the material, e.g., bricks, being fired. The bricks are stacked so as to form a series of gratings of which the apertures decrease in size downwards, and the fuel is charged through an opening in the top of the chamber, small pieces first, largest lumps last, so that the distribution of heat will remain uniform as the fuel burns away. The bricks actually supporting the fuel usually have to be previously fired, and are arranged as pillars or retorts below the charging openings, but they are surrounded by new bricks.

B. M. VENABLES.

Waste gases from furnaces (B.P. 326,205).—See I.

IX.—BUILDING MATERIALS.

Portland cements of high iron oxide content. I. S. NAGAI and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 47—49 B).—Extensive experiments on the preparation and physical properties of Portland cements containing 6—8% of iron oxide are described.

S. K. TWEEDY.

Testing refractory cements. C. McMULLEN (J. Amer. Ceram. Soc., 1930, 13, 171—173).—An electrically-heated furnace is described which attains a maximum temperature of 1500°, with the aid of which the transverse strength of bars of cement can be tested at high temperatures. The results of modulus of rupture tests on cements carried out at temperatures between 20° and 1400° and also cold after having been heated to various temperatures are presented. The effects of ignition of the temporary bond, the expulsion of the water of hydration in the clay, and of incipient fusion of the clay are noted.

F. SALT.

Small-piece testing on strength of cement mortars. III. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 46 B).—The relation between C and c (cf. B., 1930, 241) for 1 : 3-cement-sand mortars is $(C - C') = B \times (\log c - \log c')$, where B is a constant, which is large in the case of mixed Portland cements, blast-furnace slag cements, "solidit," "neo-solidit," etc. The influence of increase in the water-cement ratios on the decrease of c was also studied. The difference between various kinds of cements is reflected in the different values of the "constants of strength" for these cements.

S. K. TWEEDY.

Paints and wood surfaces. GARDNER.—See XIII.

See also A., Apr., 428, **Velocity of dehydration and subsequent hydration of gypsum** (BUDNIKOV).

PATENTS.

Manufacture of artificial masses [for covering floors and walls]. SOC. DES PROD. BELTON, and M. FAVRESSE (F.P. 633,754, 29.3.27).—Vegetable residues, e.g., leaves, seaweed, are treated with hypochlorite solution, then with 10% sulphuric acid, and, after neutralisation with soda, the mass is dried, pulverised, and mixed with regenerated rubber, resins, gelatin, drying oils, etc. The material containing rubber is formed into shape after plasticising with sulphur chloride and carbon tetrachloride. A. R. POWELL.

Fine-grinding of material (B.P. 326,366).—See I. **Artificial materials** (B.P. 325,872). **Conduits for gases** (B.P. 325,793).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Passivity of metals. IV. **Influence of acids in passivity and corrosion.** U. R. EVANS (J.C.S., 1930, 478—492; cf. A., 1930, 29).—Owing to the strong oxidising and weak base-dissolving properties of chromic acid it causes iron to become passive when alone, but increases the rate of dissolution of iron when sulphuric acid is also present. The rapid destruction of the oxide film usually present on iron by immersion of the metal in sulphuric acid is due to cathodic reduction of the film to ferrous oxide owing to formation of the cell iron|acid|ferric oxide. Addition of chromic acid prevents this reduction, so that the film remains intact for many hours in 0.1M-sulphuric acid. A preliminary anodic oxidation of the metal has the same effect as addition of chromic acid, hence the passivity of iron in acid solution during anodic treatment is due to the presence of an oxide film, contrary to the contention of Müller (A., 1928, 1319). The action of carbon dioxide during the rusting of iron has been further investigated, and it is concluded that many of the phenomena hitherto attributed to its agency are in reality caused by the presence of sulphur dioxide or hydrogen chloride. Thus droplets of condensed water on iron develop rust only in places where the iron is non-homogeneous, irrespective of the presence or absence of carbon dioxide; in fact, droplets on a non-rusty surface may be dried up in a current of carbon dioxide without the development of rust; a trace of hydrogen chloride, however, immediately causes general rusting. A solution of carbon

dioxide first causes slight corrosion of iron of the hydrogen-evolution type, but the acidity of the solution rapidly decreases, the evolution of hydrogen ceases, and the ferrous hydrogen carbonate formed is oxidised at the water-line to ferric hydroxide, which then tends to prevent diffusion of oxygen and consequent corrosion of the oxygen-absorption type. Waters saturated with carbon dioxide are therefore accelerators of the corrosion of iron, chiefly on account of the fact that they have a high solvent action on calcium compounds, and therefore prevent or hinder the formation of a protective scale on the surface of the metal. A. R. POWELL.

Properties of corrosion-resisting alloys. P. S. MENOUGH (Blast Furnace Steel Plant, 1929, 17, 1648—1651, 1810—1815).—A discussion, with tables of comparative properties, of chromium and nickel-chromium steels. CHEMICAL ABSTRACTS.

Reproducibility in corrosion work. U. R. EVANS (Amer. Electrochem. Soc., May, 1930. Advance copy. 11 pp.).—Even when the conditions are as uniform as possible, incomplete reproducibility is to be expected in corrosion work owing to the fact that corrosion begins at a number of isolated points on the surface of the metal. When the number of these points is small a wide variation in the behaviour of individual specimens can be predicted on a purely probability basis, but when the number of points of attack per unit area is large, complications arise because corrosion at one point may hinder that at neighbouring points or even prevent it from starting. The area protected in this way decreases with decreasing conductivity of the solution, indicating that the action is one of cathodic protection. Under such conditions the reproducibility will evidently be greater than that predicted from the probability theory on the assumption that the points of attack can be regarded as independent of one another.

H. J. T. ELLINGHAM.

Relation between the liquidus lines of cast iron and its chemical composition. F. YAMADA (J. Iron Steel Inst., Japan, 1929, 15, 184—186).—It is assumed that in the determination of the liquidus the influence of elements other than carbon can be reduced to that of carbon; the carbon equivalents are $0.387 \times (P \%)$, $0.215 \times (Si \%)$, and $0.077 \times (Cr \%)$.

CHEMICAL ABSTRACTS.

Arc-welding of cast iron with metallic electrodes. S. SATOH (Rev. Mét., 1930, 27, 37—48).—Excellent welds in cast iron have been obtained by the use of soft iron welding rods covered with a paste of 40—60% of graphite and 60—40% of carborundum with 1% of barium carbonate, bonded with sodium silicate solution (d 1.3). The welding rod should be connected to the negative terminal of the dynamo and used with 150 amp. at 20—22 volts; no preheating of the work is necessary. Analyses, hardness figures, and micrographs are given of welds produced with these rods, and the effect of additions of 48 other compounds instead of barium carbonate is discussed. A. R. POWELL.

Determination of carbon in open-hearth practice. K. HONDA and T. KASÉ (J. Study Met., Japan, 1929, 6, 49—52).—Since the hardness-carbon content curve is steep, the carbon content can be determined

indirectly by measurement of the (Rockwell) hardness. The results accord with those obtained by chemical analysis.

CHEMICAL ABSTRACTS.

Determination of molybdenum in steels and in ferromolybdenum. W. WERZ (Z. anal. Chem., 1930, 80, 109—112).—The steel (3—5 g.) is dissolved in 40 c.c. of hydrochloric acid, the solution evaporated to dryness, and the residue heated at 130°, cooled, and dissolved in 30 c.c. of hydrochloric acid and 10 c.c. of 1:1 nitric acid. After filtering to remove silica, the solution is evaporated to expel most of the acid, diluted with hot water, and poured into a boiling solution of 70 g. of sodium hydroxide in 300 c.c. of water. The mixture is cooled, diluted to 750 c.c., and 600 c.c. of this solution are filtered through a dry paper, rendered just acid to methyl-orange by addition of nitric acid, heated to boiling, and treated with 10—15 c.c. of 10% lead nitrate solution. The precipitate of lead molybdate is collected in a glass filter-crucible, washed with hot water, dried at 200—300°, and weighed. If the steel contains tungsten, 3—5 g. are dissolved in hydrochloric acid, the solution is oxidised with nitric acid, evaporated, diluted, and poured into an excess of hot sodium hydroxide solution. An aliquot part of the alkaline liquor is filtered, made just acid with sulphuric acid, and boiled with an excess of ammonia. The precipitate is removed and the solution boiled with 20 c.c. of 30% tartaric acid, ammonia to alkalinity, and 50 c.c. of freshly prepared ammonium sulphide solution; acidification with 50 c.c. of 1:1 sulphuric acid effects precipitation of molybdenum sulphide free from tungsten. The sulphide is collected, washed, and converted into trioxide for weighing. Ferromolybdenum is analysed by the first of the above methods after decomposition by fusion with peroxide.

A. R. POWELL.

Determination of beryllium in aluminium-free steel. F. SPINDECK (Chem.-Ztg., 1930, 54, 221).—Steel filings (1 g.) are dissolved in hydrochloric acid (d 1.12), oxidised with nitric acid, evaporated, and again evaporated with hydrochloric acid. The residue is dissolved in a little hydrochloric acid and silica removed by filtration. The diluted filtrate is heated and sodium acetate added to precipitate iron and chromium. The mixture is made up to 1 litre and 500 c.c. of the clear solution are evaporated to 30—40 c.c., filtered, and the slight iron precipitate washed on the filter with sodium acetate solution. The filtrate is treated with hydrochloric acid, boiled for 2 min., and precipitated with ammonia at 20—30°. The precipitate is collected, washed, ignited, and weighed as beryllia. With high-chromium steels it is advisable to dissolve in sulphuric acid and oxidise the chromium with silver nitrate and ammonium persulphate solution. Silver is removed by hydrochloric acid; iron and beryllium are precipitated with ammonia, dissolved in hydrochloric acid, and separated as before. J. H. BIRKINSHAW.

Effect of heat-treatment on certain silver-zinc and silver-cadmium alloys. L. GUILLET and J. COURNOT (Rev. Mét., 1930, 27, 1—7).—On annealing at 600° zinc-silver alloys with 69.77% and 62.28% Ag and quenching, a homogeneous solid solution is obtained which decomposes on tempering at 210° with the

separation of Zn_3Ag_2 in a highly dispersed form in the first case and in relatively coarse crystals in the second; in both cases appreciable hardening takes place. The cadmium-silver alloy with 60% Ag has a homogeneous structure when quenched from 600° ; on subsequent annealing at 150 – 320° a new constituent appears along the grain boundaries and as needles in the grains themselves, but practically no change occurs in the hardness. On the other hand, an alloy containing 49.25% Ag has a hardness of 119 after annealing at 500° , 86 after quenching from 500° , 107 when subsequently annealed at 150° , and 80 when annealed at 320° . The microstructure shows corresponding complex changes during these heat treatments, and it appears that the accepted equilibria in this section of the diagram do not represent the facts. A. R. POWELL.

Roasting and sintering of galena in a rotating furnace. N. C. KYRIACOU (Rev. Mét., 1930, 27, 49–53).—The theory of the roasting and sintering of galena for blast-smelting is explained and the advantages of a rotating furnace over the usual Huntington-Heberlein and Dwight-Lloyd roasting furnaces are discussed. Finally the operation of various types of rotating furnaces in operation at several lead smelting works (mostly American) is described briefly. A. R. POWELL.

Purity of lead at the time of the Nativity. A. EBELING and H. ADAM (Wiss. Veröff. Siemens-Konz., 1929, 8, [3], 203–209).—A specimen of Roman lead pipe found at Pompeii was encrusted with lead carbonate containing 0.5% Sn and consisted of coarse crystals of lead cemented together with lead oxide. After removing the corrosion products with 30% acetic acid the lead crystals contained 0.451% Sn, 0.066% Cu, 0.075% Sb, 0.006% As, 0.004% Ag, 0.005% Fe, 0.003% Zn, and 0.004% Sb. A specimen of Roman lead from Spalato-Salona, Dalmatia, contained 0.119% Sn, 0.036% Cu, 0.007% Sb, 0.001% As, 0.004% Zn, 0.001% Ni, and 0.004% Si. The complete absence of bismuth in both samples is noteworthy. A. R. POWELL.

Physics and metallography. W. ROSENHAIN (Z. Metallk., 1930, 22, 73–78).—A lecture to the Deutsche Gesellschaft für Metallkunde, in which the value of a knowledge of the lattice structure in the study of metals and alloys is discussed. A. R. POWELL.

Crystallographic analysis by X-rays. A. ROUX and J. COURNOT (Rev. Mét., 1929, 26, 655–661; 1930, 27, 8–18).—Simultaneous electrodeposition on iron or aluminium of cadmium and silver, tin, or nickel, or of copper and zinc yields deposits which are shown by X-rays to consist of complex mixtures of solid solutions and compounds and not of mixtures of the pure metals. The heat-treatment of silver-zinc alloys with 69.77% and 62.28% Ag has been followed röntgenographically. After quenching from 500 – 600° , the alloys consist of a homogeneous solid solution with a face-centred cubic lattice, $a = 4.054$ and 4.367 Å, respectively. Annealing at 210° results in hardening due to deformation of the lattice into the tetragonal system, owing to the precipitation of a second constituent in a finely-divided form, undetectable by X-rays. The tetragonal lattice of the alloy with the higher silver content has $a = 4.054$ Å, and $b/a = 1.02$, and that of the other alloy has $a = 4.175$ Å.

and $b/a = 1.06$. Hardening of the aluminium-copper alloy (10% Al) after quenching and tempering takes place in a similar manner. The mechanism of deformation and recrystallisation during cold-working and annealing of aluminium and duralumin and the effect of impurities in restraining the grain growth of copper and aluminium during recrystallisation are illustrated by numerous röntgenographs taken at various stages of the treatment. A. R. POWELL.

Moisture in blast-furnace slag. BUDNIKOV.—See VIII. **Active oxygen as anti-rust agent.** HEBBERLING. **Rust-preventive paints.** HOEPKE. **Pigments and rust prevention.** WAGNER.—See XIII. **Corrosion of canned-food containers.** BOGATSKY and others.—See XIX.

See also A., Apr., 402, **Tellurium-platinum thermo-elements** (TEICHMANN). 419, **Iron-nitrogen system** (EPSTEIN and others). **System nickel-chromium** (NISHIGORI and HAMASUMI). 445, **Spectroscopic determination of bismuth in copper** (LOMAKIN).

PATENTS.

Metallurgical furnace. F. A. J. FITZGERALD and J. KELLEHER, Assrs. to TITANIA CORP. (U.S.P. 1,742,441, 7.1.30. Appl., 24.7.28).—An electric furnace for the production of titanium nitride consists of a chamber of refractory material provided with an inclined grate to support the charge and a resistance heating element above the charge. Nitrogen is caused to circulate through the charge. Gas-tight charging and discharging hoppers are provided. C. A. KING.

Ignition furnace. METALLGES. A.-G. (B.P. 318,197, 19.6.29. Ger., 30.8.28).—The width of the exit slit of an ignition furnace may be varied by a lateral movement of the side walls of the furnace. C. A. KING.

Heating or melting of metals or alloys by induced currents of electricity. C. TAMA, and ELECTRIC FURNACE CO., LTD. (B.P. 325,940, 7.1.29).—A charge to be melted in an induction furnace is rendered conductive by chemical or electrochemical treatment so that currents of comparatively low frequency may be used throughout the heating operation; e.g., finely-divided material is pressed into coherent masses and the surface of materials coated with oxide is cleaned electrolytically. C. A. KING.

Manufacture of steel. J. K. SMITH, Assr. to GRANULAR IRON CO. (U.S.P. 1,742,487, 7.1.30. Appl., 11.2.28).—Iron is charged into an open-hearth furnace, covered with a mixture of basic slag, ground limestone, and carbonaceous material, and melted. C. A. KING.

Heat-treatment of steel and iron. GES. F. INDUSTRIEGASVERTUNG M.B.H. (B.P. 297,796, 23.8.28. Ger., 28.9.27).—Iron or steel heated to within the usual limits of annealing temperatures is cooled continuously to normal temperature by immersing it in a liquefied gas of low b.p., e.g., liquid air. It is claimed that thereby the hardening proceeds much more slowly than by a single immersion in water, but a similar effect may be obtained by interrupting the cooling in water or oil so that the successive immersions approximate to the conditions of a single immersion in liquid air. C. A. KING.

Steel. W. C. HAMILTON and C. E. SIMS, Assrs. to AMER. STEEL FOUNDRIES (U.S.P. 1,742,857, 7.1.30. Appl., 3.12.27).—Steel suitable for car wheels consists of a high-manganese, pearlitic steel containing 1–2.5% Mn and 0.5–2% Cu. C. A. KING.

Surface-hardened material and its production. Case-hardening. A. B. KINZEL, Asst. to ELECTRO METALLURGICAL Co. (U.S.P. 1,736,919 and 1,736,920, 26.11.29. Appl., [A] 28.7.27, [B] 20.8.27).—(A) The surface layer of iron or steel is "aluminised" by heating the article in contact with powdered aluminium. It is claimed that the presence of aluminium in iron greatly accelerates the action of case-hardening by nitrogenous substances, *e.g.*, ammonia. (B) The presence of more than 0.3% V in solid solution in iron accelerates nitrogenisation of steel by ammonia and similar case-hardening reagents. Aluminium may also be present with advantage as in (A). C. A. KING.

Hard [iron] alloy. R. PAQUET (Dutch P. 17,526, 13.10.25).—An alloy of 67% Fe, 0.3% Ni, 20% Cr, 9.5% Mo, 0.3% W, 1% V, 2% Mn, and 1.7% C is claimed. A. R. POWELL.

Hard [tungsten] alloy and its manufacture. P. M. McKENNA, Asst. to VANADIUM ALLOYS STEEL Co. (U.S.P. 1,737,255, 26.11.29. Appl., 22.1.29).—A hard alloy suitable for machine tools consists of tungsten, up to 5.5% Be, and not more than 7% (preferably less than 3%) C. The alloy is prepared by heating a mixture of tungsten and carbon black in a neutral atmosphere and grinding the product with powdered beryllium in a ball mill for 8 hrs.; the powder is then pressed hydraulically, heated, cut to shape, and sintered. C. A. KING.

Age-hardening aluminium alloys. ALUMINIUM-IND. A.-G. (Swiss P. 124,793–4, 5. and 19.11.26. Ger., [B] 21.8.26).—(A) The alloy comprises a nearly saturated solid solution of zinc or copper in aluminium to which is added not more than 4% of a constituent which will produce age-hardening, *e.g.*, tin, cadmium, silicon, or magnesium silicide. (B) The alloy comprises aluminium with 10–15% Zn, 1.5–3% Cu, 0.05–0.2% Mg, 1–2% Fe, and small quantities of manganese, nickel, chromium, or titanium. Part or all of the iron may be replaced by silicon. A. R. POWELL.

Manufacture of vanadium-aluminium-silicon alloys. VANADIUM CORP. OF AMERICA, Assces. of B. D. SAKLATWALLA (B.P. 305,202, 20.12.28. U.S., 2.2.28).—See U.S.P. 1,727,180; B., 1929, 945.

Moulds for casting metals. A. E. WHITE. From ALUMINIUM, LTD. (B.P. 325,553–4, 21.8.28).

Drying and heating apparatus (B.P. 326,269).—See I. Metallising bands (U.S.P. 1,731,261).—See XI. Uniting rubber to metal (B.P. 307,056 and 310,461).—See XIV.

XI.—ELECTROTECHNICS.

[Electric] furnace for high temperatures. S. REINER (Chem. Fabr., 1930, 101–102).—The furnace comprises a highly refractory tube embedded in a mass of "kryptol" packed inside a rectangular prismatic brick structure. The kryptol acts as the heating resistance,

and for heating a tube 10 cm. in diam. and about 40 cm. long a current of 100 amp. is required. Temperatures of 2000° can be maintained for some time and an oxidising atmosphere is obtained by passing a current of air through the tube. A. R. POWELL.

Electric spalling furnace. H. N. BAUMANN, JUN. (J. Amer. Ceram. Soc., 1930, 13, 167–170).—A description is given of an electric furnace which is heated by a graphite (or silicon carbide) resistor. Twelve standard refractory bricks can be heated simultaneously to $1350^{\circ} \pm 20^{\circ}$. In the spalling test one brick from each side of the furnace is removed at a time and subjected to an air-blast for 10 min. F. SALT.

Safety devices for electric light and power plant in chemical works. G. A. SCHMIDT (Chem. Fabr., 1930, 115–116).—An automatic cut-out for use in place of the usual fuses for electric motors in chemical works is described and illustrated in various modifications. A. R. POWELL.

Some conductivity characteristics of chromic acid and chromic acid chromium-plating solutions. R. H. CHERRY (Amer. Electrochem. Soc., May, 1930. Advance copy. 9 pp.).—Approximate data have been obtained for the specific conductivity of chromic acid solutions of various concentrations at various temperatures. At 25° the conductivity reaches a maximum at about 3.75*M*, and at 45° at about 4.5*M*. The conductivity of a 2.5*M*-solution is a linear function of temperature. The presence of chromic sulphate has only a small effect on the conductivity, but with 2.5*M*-chromic acid solution a maximum conductivity is reached when the ratio of the chromic acid molarity to the sulphate normality is 55.5. From the conductivity data for chromic acid solutions, values of the degree of dissociation and the hydrogen-ion concentration are calculated on the basis of the classical theory of electrolytic dissociation. H. J. T. ELLINGHAM.

Testing refractory cements. McMULLEN.—See IX. **Arc-welding of cast iron.** SATOH.—See X. **Determination of crystal in massecuites.** SANDERA. **Electrodialysis of molasses.** KAMEYAMA and KATO.—See XVII. **Electrodialysis of wort and beer.** WINDISCH and others.—See XVIII.

See also A., Apr., 391, **Photo-cell** (LANGE). 402, **Measurement of weak alternating currents** (TEICHMANN). 422, **Standardisation of antimony electrodes** (ITANO). **Biilmann's quinhedron electrode** (ITANO and ARAKAWA). 423, **Overvoltage in the electrolytic production of hydrogen** (BAARS). 433, **Action of silent discharge on oils, fats, and fatty acids** (IWAMOTO). 446, **Apparatus to measure colour temperature of lamp filaments** (SHARP). 451, **Action of silent discharge on saturated fatty acids** (IWAMOTO).

PATENTS.

Electrolytes for electric cells. L. A. LEVY, and ALMEIDA ACCUMULATORS, LTD. (B.P. 324,918, 5.11.28).—An electrolyte for secondary cells of the type described in B.P. 320,916 (B., 1930, 153) consists of zinc chloride and potassium bromide, or chlorides and bromides of potassium and zinc, a small amount of hydrochloric acid,

and an organic acid or salt, *e.g.*, zinc or potassium acetate, or acetic acid.

J. S. G. THOMAS.

Production of an electrical connexion of high conductivity between a layer formed of a metal compound and a conductor. SIEMENS-SCHUCKERT-WERKE A.-G. (B.P. 305,171, 29.1.29. Ger., 1.2.28).—A layer of finely-divided graphite, *e.g.*, Aquadag, is sprayed upon the metal compound, *e.g.*, a metallic oxide rectifier, before the application of a metal coating.

J. S. G. THOMAS.

Metallising the surfaces of insulating bands particularly for use in electrical condensers. E. PFIFFNER, Assr. to RADIO PATENTS CORP. (U.S.P. 1,731,261, 15.10.29. Appl., 16.12.25. Ger., 24.11.24).—A flexible band of insulating material is passed by means of rollers through a series of baths consisting alternately of a solution of a metal compound, *e.g.*, ammoniacal silver oxide solution, and of a solution of a reducing agent, *e.g.*, formaldehyde, whereby first one side of the band and then the other is coated with a conducting film of metal.

A. R. POWELL.

Apparatus for generating actinic rays. V. H. M. A. DANGERFELD (B.P. 327,122, 24.12.28. Addn. to B.P. 275,596).

Solid electric conductor, especially for high and maximum voltages. METALLGES. A.-G. (B.P. 314,340, 21.6.29. Ger., 25.6.28).

Control of temperature (B.P. 326,309).—See I. **Internal-combustion engines** (B.P. 325,866). **Lubricating oils** (B.P. 325,832).—See II. **Metallurgical furnace** (U.S.P. 1,742,441). **Heating of metals or alloys** (B.P. 325,940).—See X. **Separation of rubber from latex** (B.P. 307,747 and 325,401). **Plastic composition** (B.P. 325,312).—See XIV. **Treatment of milk** (B.P. 325,470).—See XIX.

XII.—FATS; OILS; WAXES.

Preservation of fats. G. W. FIERO (Amer. J. Pharm., 1930, 102, 146—154).—The preserving action on lard of a large number of substances has been examined. Complete preservation was effected by resorcinol (1%), clove oil (0.38%), guaiacol (0.06%), thymol (2.5%), and creosote (0.25%). Using the following substances, the lard remained free from rancidity (Kreis test) for four weeks in a warm, light room, or for 10 weeks in a cool, dark room, but rancidity developed after 8 and 25 weeks, respectively: saffrole (0.5%), sodium benzoate (1%), sodium benzoate dissolved in water and emulsified with the fat by means of neutral soap (0.1%). "Methenamine" completely prevented rancidity, but the fat became yellow on keeping. Mixtures of lard with the following substances did not develop rancidity: hydrogenated oil (25%), white wax and petrolatum (25% of each), white wax and liquid paraffin (25% of each), paraffin and liquid paraffin (25% of each).

E. H. SHARPLES.

Fractional saponification of fatty substances. I. E. DE'CONNO and L. FINELLI (Annali Chim. Appl., 1930, 20, 67—73).—The results of experiments on drying, semi-drying, and non-drying vegetable oils, fish oils, animal and vegetable solid fats show that

partial saponification, by modifying considerably the constants of the unsaponified part (especially the Tortelli heat number) and of the saponified part (mean mol. wt. of the non-volatile insoluble acids), may furnish useful indications concerning adulteration of edible fats.

T. H. POPE.

Colorimetry of oils and fats. F. PALLAUF (Chem. Umschau, 1930, 37, 21—22).—Observations of Greitemann (B., 1929, 564) are criticised. In view of the current English standard practice (Brit. Stand. Spec., No. 258), the standardisation of the iodine colour value in terms of mg. of iodine per 100 c.c. of solution and the use of a layer 10 mm. thick (except in the case of very pale edible oils) is advocated; further, these conditions lead to more convenient numerical values for the colour index.

E. LEWKOWITSCH.

Free [fatty] acid important in cotton-seed value index. G. S. MELOY (Oil & Fat. Ind., 1930, 7, 135—138).—Calculations and formulæ are described for determining the value index of an actual cotton-seed in relation to quotations for a "basis cotton-seed": the latter is taken as a seed which on analysis gives oil 19%, ammonia 3.5%, tare 11%, and free fatty acids not more than 2% at the time of purchase. A table is given for the corrections to be applied to the index of seed containing excess of free fatty acids.

E. LEWKOWITSCH.

Separation of solid and liquid fatty acids and detection of hardened fats in cacao butter. J. GROSSFELD (Chem. Umschau, 1930, 37, 3—13, 23—28).—Appreciable losses of solid unsaturated acids may occur by the recrystallisation of the insoluble lead salts from 95% alcohol, such loss being favoured by the presence of potassium acetate and reduced by the presence of excess of lead acetate; it is, however, notable that the losses of isooleic acid become relatively much greater as the total amount of the acid present increases, so that even small quantities of hardened oils can be detected in admixture with cacao fat. By dilution of the alcoholic solution with water it is possible to recover far greater quantities of isooleic acid: at first no appreciable amount of lead oleate is precipitated with it, but after a certain degree of dilution the precipitation of the oleate increases rapidly, but may be modified by the presence of acetic acid or lead acetate, or by temperature conditions. A new procedure is detailed for the fractional crystallisation of the lead salts from aqueous alcohol, by which oleic and other liquid acids are quantitatively removed. *E.g.*, 2.5 g. of fat are saponified with 1 c.c. of 50% potash and 25 c.c. of 95% alcohol and decomposed with 100 c.c. of lead acetate solution (50 g. of crystals and 5 c.c. of 96% acetic acid made up to 1 litre with 80 vol.-% alcohol). The salts are completely dissolved by warming, 20 c.c. of boiling water are added, and the mixture is kept overnight; the precipitate thus formed is collected on a glass Gooch crucible and washed with 50 c.c. of 70% alcohol and the crucible is fitted in an inverted position in an extractor, 3 c.c. of 96% acetic acid are poured over the plate, and the salts are dissolved, by extraction, in 100 c.c. of the lead acetate solution, then 15 c.c. of hot water are added with shaking and the solution is cooled overnight. The lead

salts are collected and washed as before, and are then dissolved in 5 c.c. of acetic acid and 10 c.c. of 96% alcohol and decomposed when warm with 5 c.c. of nitric acid (d 1.2), the flask being filled to the neck with water and warmed until a clear fatty acid layer is formed. After cooling, the acid cake is removed, washed with cold water, dried in the steam oven, and returned to the dried-out flask and dissolved in chloroform preparatory to the determination of iodine value. It is preferable not to weigh the solid fatty acids, but to express the iodine value found in terms of the percentage of *isooleic* acid in the original oil (1 c.c. of 0.1*N*-thiosulphate = 0.565% of *isooleic* acid). A certain amount of *isooleic* acid lead salt passes into solution (23–34% of *isooleic* acid was recovered from hardened oils containing actually 36–43%), but the presence of as little as 0.3% of hardened arachis oil in cacao butter may be detected with certainty by this method. Traces of water in the fatty acids had no deleterious effect in the Hanus determination, nor was any oxidation of *isooleic* acid observed on drying the acids in the steam oven. Increase of weight due to esterification did not amount to more than 0.1–0.3%. It is probable that the fatty acids of hardened arachis oil contain two *isooleic* acids, differentiable by solubilities of their lead salts, one of which is so insoluble as to be precipitated practically quantitatively. E. LEWKOWITSCH.

Determination of tung oil. J. MARCUSSEN (Farben-Ztg., 1930, 35, 1203).—The author replies to criticism of his method by Wolff and his co-workers (B., 1930, 292). Coagulation of tung oil by iodine-chloroform is hindered by the presence of turpentine and lead driers. If these be removed (by shaking out with alcohol and acetic acid, respectively), the method gives satisfactory results. S. S. WOOLF.

[Determination of tung oil.] H. WOLFF and J. RABINOWITZ (Farben-Ztg., 1930, 35, 1203; cf. preceding abstract).—Polemical against Marcussen. The authors reiterate that the method is unreliable. S. S. WOOLF.

***Hydnocarpus anthelmintica* seed from Ceylon.** ANON. (Bull. Imp. Inst., 1930, 28, 6–8).—The seeds (wt. 2 g., 70% of shell) of *H. anthelmintica*, from an experimental plantation in Ceylon, contain 30% of kernel (wt. 0.6 g., 7.3% of moisture) which on extraction yields 60.1% of oil having α (in chloroform) + 47.2°, α of fatty acids (in chloroform) + 48.8°, and solidifying point of fatty acids 37.2°. *Hydnocarpic* acid [m.p. 60°, α_D + 67.9°, iodine value (Wijs, 3 hrs.) 100.0%, 29.9% Ag in silver salt] and *chaulmoogric* acid (m.p. 67–68°, α_D + 60.7°, iodine value 89.3%, 27.5% Ag in silver salt) have been isolated from the oil. E. H. SHARPLES.

Determination of the oil content of palm kernels. A. GEHRKE (Chem. Umschau, 1930, 37, 1–3).—Possible reasons for the discrepancies observed between English and German determinations of the fat content of palm kernels on sampling shipments are discussed: it is recommended that the jute bags now used should be replaced by containers impervious to water and dust. E. LEWKOWITSCH.

[Analysis of] sulphonated oils. Report of a Committee of the American Leather Chemists' Association. G. W. PRIEST (J. Amer. Leather Chem. Assoc., 1929, 24, 570–576).—Two methods of determining the neutral fat in a sulphonated oil by extraction with light petroleum and methylated ether, respectively, have been compared in the case of 4 samples of sulphonated castor oils. The results obtained with light petroleum were too low, and by the ether-extraction method are much lower than those given by the official method of the American Leather Chemists' Association.

D. WOODROFFE.

Determination of neutral fat in sulphonated oils. R. HART (J. Amer. Leather Chem. Assoc., 1929, 24, 576–582; cf. B., 1929, 403).—The saponification value of the total fatty matter of the sulphonated oil is determined as follows. The oil (2–2.5 g.) is boiled for 30 min. under reflux with 25 c.c. of 0.5*N*-alcoholic potash, 50 c.c. of neutralised alcohol are added, the mixture is boiled gently to expel all ammonia, the excess of alkali titrated with 0.5*N*-acid, and the mg. of alkali, F_0 , absorbed by 1 g. of the fat is calculated therefrom. The percentage of neutral fat is determined from the formula $100F_n/V_n$, and particulars are given for the calculation of F_n and V_n from F_0 and the other figures obtained in the official method of analysis. The method has been tested on mixtures of known composition and on sulphonated oils, and the accuracy of the method is proved. The results obtained by this saponification method on the oils used by the Committee of the American Leather Chemists' Association (cf. preceding abstract) are about twice those obtained by extraction with methylated ether and much greater than those given with light petroleum. The extraction of the original undecomposed oil by solvents does not yield all the neutral fat, since some of it must be sulphonated, and therefore partly soluble in water.

D. WOODROFFE.

Drying of organic solutions. LUND.—See III. **Fats and carbonation.** STANEK and VONDRÁK.—See XVII.

See also A., Apr., 407, Adsorption of Japanese acid clay (UENO). 433, Action of silent discharge on oils, fats, and fatty acids (IWAMOTO). 434, Oxidation of oils in presence of irradiated sterols (COUTURE). 451, Action of silent discharge on saturated fatty acids (IWAMOTO). Fatty acids of wheat starch (LEHRMAN). Soaps of fatty acids of the oleic acid series (HIROSE and SHIMUMURA). 477, Colour reaction for ergosterol (MEESEMAECKER). 489, Microchemical saponification process (ROSENTHALER). 500, Decomposition of oils by fat-decomposing enzymes (EZOL). 507, Fruit of *Gleditschia triacanthos* (HARRIS).

PATENTS.

Products derived from soap-forming fatty acids, their glycerides or sulphonic acids. E. I. DU PONT DE NEMOURS & Co. (B.P. 295,024, 27.7.28. U.S., 4.8.27).—The hydroxylated alkylamine (especially "triethanolamine") salts of soap-forming fatty acids (e.g., oleic and stearic acids) or sulphonated oils (e.g., Turkey-red oil), obtained by mixing the base and the acid or

by heating the base with a fatty oil, are organic soaps useful as dyeing assistants and dispersing agents.

C. HOLLINS.

Drying and conditioning of household soap [for stamping]. TOMLINSONS (ROCHDALE), LTD., and J. N. TOMLINSON (B.P. 324,459, 21.11.28).—The soap is transferred on movable supports into a chamber where it is heated by circulating hot air, and then cooled by cold air, circulated first in one direction and then in the reverse, the total process occupying about 50 min.

E. LEWKOWITSCH.

Manufacture of cod-liver oil and like oils. C. FOWLER (B.P. 325,529, 15.10, 1.11, and 17.12.28).—The cooking or settling tanks are fitted with draw-off devices providing orifices at adjustable levels or (especially for floating factories) with flexible draw-off tubes, to be lowered either mechanically or automatically by the provision of floats, so that the orifice is maintained just below the surface of the liquid.

E. LEWKOWITSCH.

Recovery of oil from fish livers and other similar materials. A. W. OWE (U.S.P. 1,742,666, 7.1.30. Appl., 25.2.26. Norw., 5.3.25).—Fish liver in a finely-divided condition is heated to 60° or over and maintained at this temperature for such time only as is necessary to cause the liver to give off oil, after which it is cooled suddenly to 50° or below in contact with non-gaseous cooling means.

S. S. WOOLF.

Manufacture of linoleum. LINOLEUM MANUF. CO., LTD., and A. A. GODFREY (B.P. 326,763, 12.12.28).

Wax from coal (B.P. 325,816). Demulsifying soap (U.S.P. 1,742,623).—See II. **Dyeing of fatty acids (B.P. 299,790).**—See VI. **Sealing wax (B.P. 325,014).**—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ovens and furnaces for the paint and varnish industry. H. SPEICHER (Farben-Ztg., 1930, 35, 1259—1261).—A general dissertation on the use of electricity, or gas in place of coal, coke, etc. in varnish cooking, pigment manufacture, etc., the advantages of each type of heating agent being considered.

S. S. WOOLF.

Viscosity of paints containing water. H. WOLFF (Farben-Ztg., 1930, 35, 1100—1101).—If water be introduced into white lead paints, the exponent n in the equation $kp^n = V - v$ (which expresses the viscosity V of oil paints of various pigment concentrations, p being the percentage concentration of the pigment by vol., v the viscosity of the oil, and k and n constants characteristic of the pigment; cf. B., 1929, 825) increases with the quantity of water added. In the case of lithopone paints the exponent is hardly altered and, if anything, is lowered. The white lead curves for different water contents intersect at an oil content of approx. 34.5% by wt., whilst the lithopone curves do not intersect over the range for which the equation holds. As a working hypothesis, these differences are attributed to the fact that in the case of white lead the water is emulsified in the oil, whilst lithopone particles are surrounded by a water sheath. It is considered that these experiments indicate the value of the "turboviscometer" in

fundamental investigations (thus exceeding the original functions of the apparatus).

S. S. WOOLF.

Durability of exterior paints on wood surfaces. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Feb., 1930, Circ. 361, 180—234).—The causes of failure of exterior paints are discussed and illustrated, and methods for their prevention are indicated. Subjects treated include chalking, alligatoring, or checking, gas discoloration, copper stains, spotting effects, brown staining, soot collection, washing effects, types of priming, flaking, cracking, blistering, and miscellaneous construction defects.

S. S. WOOLF.

"Active oxygen" as anti-rust agent. H. HEBBERLING (Farben-Ztg., 1930, 35, 1256—1257).—The ascendancy of red lead as a rust-preventive pigment is due to its liberation of nascent oxygen in films. This is not bound up with lead peroxide content only, grade of dispersion being of great significance in this connexion. The suitability of other pigments, e.g., zinc chromate, is discussed from this aspect.

S. S. WOOLF.

Testing of rust-preventive paints. F. HOEPKE (Farben-Ztg., 1930, 35, 1153—1154).—A dissertation, the subjects dealt with comprising opacity, viscosity, drying, permeability, elasticity, tensile strength, hardness, gloss, swelling and shrinkage, micrography, durability, including artificial weathering, etc. Numerous results obtained by these methods are tabulated and discussed, among the conclusions drawn being the following: films of highly-dispersed red lead in linoleate-boiled oils are stronger than similar films containing resinate-boiled oils; iron oxides of high ferric oxide content are inferior to red lead in boiled oils, but superior in resinate-stand oils; boiled linseed oil absorbs more water and has poorer durability than linseed oil-tung oil blends; ratings of paints by normal and by accelerated weathering tests show concordance.

S. S. WOOLF.

Iron oxide pigments and rust prevention. H. WAGNER (Farben-Ztg., 1930, 35, 1151—1152, 1204—1206).—The influence of the physical and chemical properties of iron oxide pigments on water absorption by paint films derived therefrom and on rusting of painted iron plates was studied by the immersion in water and subsequent exposure of plates coated with paints based on 27 such pigments representing types differing considerably in composition, particle shape and size, electrolyte content, variation from neutrality, etc. It was shown by X-ray photographs that the hæmatite space-lattice persists in all iron oxide pigments, even highly dispersed artificial oxides showing it, and it is considered that amorphous iron oxide pigments are non-existent. Swelling of the paint films is influenced mainly by the oil content and by the soluble non-ferrous constituents, soap-formation being entirely absent, contrary to many previous statements. Rust formation is classified into 3 types according as it takes place (a) under, (b) through, or (c) over the surface. Type c is the "abnormal" type described by Wolff (cf. B., 1929, 988). Type b may be measured by electrical methods recording permeability, e.g., by means of Jäger's penetrator, but this does not show concordance with visual inspection of type a, nor can ultra-violet fluorescence methods be success-

fully applied in this case. Rust formation cannot be correlated with oil content and swelling of films. The results are discussed at length, and although it was not possible to define rust-preventing characteristics, the presence of calcium sulphate etc. is considered harmful, whilst high iron oxide content appears at least not unfavourable to rust prevention, especially when vehicles affording better adhesion than linseed oil are used.

S. S. WOOLF.

Evaporation balance [for paint, varnish, and lacquer solvents]. L. P. HART (Amer. Paint and Varnish Manufs.' Assoc., Feb., 1930, Circ. 360, 173—179).—A counterpoised-beam balance for determination of evaporation rates of such solvents is described, and a description also given of three further types of apparatus under development in this connexion.

S. S. WOOLF.

Composition of turpentine and of turpentine and rosin oils from the Vakhtan factory. B. ARBUZOV (J. Appl. Chem., Russia, 1929, 2, 585—594).—The composition of the fractions is recorded. The turpentine contains a substance (7.4%) which is not present in turpentine from *Pinus sylvestris*.

CHEMICAL ABSTRACTS.

Menthenol from turpentine from the Vakhtan factory. B. ARBUZOV (J. Appl. Chem., Russia, 1929, 2, 595—598).—The menthenol, $C_{10}H_{18}O$, had d^{16}_D 0.9378, n_D^{16} +16° to +50°, b.p. 213—214°. The component, $C_{10}H_{18}O$, of high optical activity had b.p. 98—99°/10 mm., n_D^{43} +43.20°.

CHEMICAL ABSTRACTS.

Sp. gr. and Baumé gravity tables for turpentine. W. C. SMITH and F. P. VEITCH (U.S. Dept. Agric., Feb., 1930, Circ. 110, 9 pp.).—Tables are given from which specific gravities and degrees Baumé of turpentine at 60° F. compared with water at 60° F. may be read off from observed hydrometer readings at known temperatures.

S. I. LEVY.

Refining of pine pitch with soda. F. SOLODKI (J. Appl. Chem., Russia, 1929, 2, 599—619).—Increase in concentration of the soda solution from 2.5 to 5% increases the yield by only 3%, but poor separation of the soaps results from the use of the more dilute solutions.

CHEMICAL ABSTRACTS.

Tests for resins of jalap, podophyllum, and scammony. DOTT.—See XX.

PATENTS.

Preparation of white lithopone from incompletely purified zincic solutions. M. BIDAINE (B.P. 325,641, 27.12.28).—Substances that are blue or greenish-blue or that acquire such colour by calcination, e.g., chromium sulphate, zinc or cobalt aluminate, are precipitated on or at the same time as lithopone, so that the yellow or reddish-yellow tint imparted to the latter by the impurities, e.g., nickel and cobalt, left in the solutions containing zinc is complemented.

S. S. WOOLF.

Preparation of lacquers and lacquering. BAKELITE GES.M.B.H. (B.P. 295,335, 9.8.28. Ger., 9.8.27).—Condensation products of phenol and aliphatic aldehydes in the intermediate or "B" stage are dissolved under specified conditions of temperature and pressure in cyclic ketones or alcohols, hydrogenated naphthalenes

(except tetrahydronaphthalene) or phenols, etc., or suitable mixtures of these, with or without addition of oils, e.g., linseed, poppyseed, and tung oil, and non-solvent liquids, e.g., bromobenzene, benzene hydrocarbons, tetrahydronaphthalene, etc. Greater quantities of solvent than are required for dissolution are used and the excess is subsequently removed by distillation. Catalysts, e.g., nitrophenols, and plasticisers may be added.

S. S. WOOLF.

Lacquers, coating compositions, etc. BRIT. CELANESE, LTD. (B.P. 307,289, 4.3.29. U.S., 3.3.28).—A cellulose acetate lacquer containing a furfuraldehyde-acetone resin (produced by alkaline condensation) gives adherent, clear films. Other resins, plasticisers, pigments, etc. may be added; the usual solvents (acetone, alone or with benzene, toluene, alcohol, etc.) are employed.

C. HOLLINS.

Lacquers, coating compositions, etc., and articles coated therewith. BRIT. CELANESE, LTD. (B.P. 307,290—1, 4.3.29. U.S., 3.3.28).—Coating compositions containing (i) a derivative (preferably organic) of cellulose, e.g., cellulose acetate, (ii) a synthetic resin obtained by the condensation of furfuraldehyde with (A) an aromatic amine, e.g., aniline, or (B) a phenol (in the presence of an alkaline catalyst), and (iii) a volatile solvent are claimed. In addition, one or more artificial resins (e.g., a phenol-formaldehyde resin, with an acid catalyst), natural gums or resins, semi-synthetic resins, plasticisers or softeners, solvents of medium or high b.p., pigments, and dyes may be incorporated.

S. S. WOOLF.

Coating material for the handles of utensils. C. SCHMIDT (G.P. 461,009, 20.7.26, and B.P. 326,255, 12.12.28).—The product obtained by oxidising with permanganate the hardened condensation product of formaldehyde with phenol in the presence of excess ammonia is mixed with the product obtained by digesting ivory-nut turnings with concentrated sulphuric acid and the mixture is heated until it hardens. The material so obtained adheres firmly to metal, is resistant to acids and alkalis, and is a non-conductor of heat and electricity.

A. R. POWELL.

Increasing the durability of coatings of paints, varnishes, and the like. CHEM. FABR. DR. J. WIERNIK & Co. A.-G., and BAKELITE GES.M.B.H., Assees. of J. SCHEIBER (B.P. 299,024, 19.10.28. Ger., 19.10.27).—The durability of paint coatings etc. that have completed their normal drying is improved by treatment with solutions of deoxidants, e.g., multivalent phenolic substances, α -naphthol, aromatic amines, in volatile solvent adapted to soften but not to dissolve the coating. Diminution in gloss is overcome by adding up to 10% of raw or treated tung oil to the deoxidant solution.

S. S. WOOLF.

Removal of varnish and enamel coatings. E. C. FRIES, Assr. to STUDEBAKER CORP. (U.S.P. 1,742,347, 7.1.30. Appl., 23.2.26).—Varnish and enamel removers containing a chlorinated acetic acid, dichlorobenzene, and, if desired, alcohol are claimed.

S. S. WOOLF.

Manufacture of cellulose ester compositions. E. C. DE STUBNER (B.P. 297,679, 22.9.28. U.S., 24.9.27).—A resinous ingredient for incorporation with cellulose

ester compositions (such as lacquers etc.) is prepared by esterifying the hydroxyl group or groups of a natural fossil or semi-fossil resin, the other ester ingredient (*e.g.*, butyl acetate) being simultaneously prepared by the esterification process. F. R. ENNOS.

Manufacture of crayons and sealing wax. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 325,014, 8.9.28).—A cellulose ether or a cellulose ester of an acid of high mol. wt., *e.g.*, lauric acid, is dissolved at a raised temperature in a fatty acid, a fat, a hydrocarbon which is solid at ordinary temperatures, or in a mixture of any of these, dyes or pigments are incorporated, and the solution is cast in moulds. S. S. WOOLF.

Manufacture of artificial resins and products containing them. BRIT. CELANESE, LTD. (B.P. 299,067, 20.10.28. U.S., 21.10.27. Addn. to B.P. 299,066; B., 1930, 338).—The ketone-phenol resins of the prior patent are condensed with formaldehyde or other aldehyde to give a soluble, non-reactive resin, m.p. about 100°, suitable for cellulose acetate lacquers. C. HOLLINS.

Manufacture of synthetic resins. BRIT. CELANESE, LTD. (B.P. 296,674, 3.9.28. U.S., 3.9.27).—A phenol is condensed with an aldehyde in molecular proportions not greatly exceeding 1 : 1, in the presence, at least in the final stages of the reaction, of soluble dihydrogen phosphates, *e.g.*, of ammonium, alkali or alkaline-earth metals. The reaction may be begun in the presence of phosphoric acid and sufficient alkaline material added later, thus modifying the rate of reaction. Pale fusible resins, soluble in acetone etc. and free from appreciable darkening on exposure, are obtained. S. S. WOOLF.

Manufacture of condensation products from phenols and formaldehyde. R. HESSEN (B.P. 324,913, 5.11.28).—Molecular proportions of a phenol and formaldehyde are condensed together in the presence of a basic catalyst, the aqueous layer is removed, subsequent additions (2—4) of formaldehyde and, if desired, catalyst are made, the condensation is continued, and the aqueous layer removed, the process continuing until no free phenol remains in the resinous layer. S. S. WOOLF.

Manufacture of condensation products of cresol and formaldehyde. P. CHESTAKOFF [SHESTAKOV] (B.P. 303,022, 24.12.28. Fr., 22.12.27).—Cresol is condensed with formaldehyde at 80—100° in the presence of a catalyst of low activity, *e.g.*, oxalic acid, succinic acid, zinc or lead acetate. The dehydrated intermediate product ("resol") is treated with 0.5—3% of an organic sulphonic acid of high mol. wt. and dispersive power, readily soluble in the resol, *e.g.*, the product of sulphonation of petroleum oils etc. The treated resol is cast in moulds and the temperature is gradually raised for 5—10 hrs. to 110—130°, until a hard, insoluble, infusible resin is obtained. S. S. WOOLF.

Artificial resins and their manufacture. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 296,787, 7.9.28. Ger., 8.9.27).—Polyhydric alcohols, *e.g.*, glycerin, are condensed with polybasic acids, *e.g.*, phthalic acid, in an atmosphere practically free from oxygen, *e.g.*, *in vacuo*, or under a

stream of inert gas, *e.g.*, carbon dioxide, hydrogen. The artificial resins so obtained are practically colourless. S. S. WOOLF.

[Grinding apparatus for] manufacture of paints. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. C. B. WEBSTER (B.P. 303,380, 31.12.28. U.S., 30.12.27).

Artificial resin from coal (B.P. 325,816). **Utilisation of waste liquors** (B.P. 326,112).—See II. **Products of acrylic acid** (B.P. 304,681).—See III. **Ice colours and pigments** (B.P. 300,504 and 300,557). **Azo dyes for lakes** (B.P. 325,229). **Azo dyes in drying oils** (B.P. 301,726).—See IV. **Artificial silk** (B.P. 303,867).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation of *Hevea* [rubber] latex. N. BEUMÉE-NIEUWLAND (Arch. Rubbercultuur, 1929, 13, [10]; Med. Proefstat. Rubber, 1929, [44], 555—567).—Latex may be regarded as a system of lyophilic colloidal proteins protecting the lyophobic rubber globules; it shows certain phenomena characteristic of lyophobic colloids and others typical of the lyophilic group. The phenomenon of the "second liquid zone" in its coagulation behaviour may arise from a change of molecular proteins into acid proteins, and this is confirmed by the fact that protein-free latex exhibits no "irregular series." The p_H at which coagulation of latex occurs also coincides with the p_H of protein precipitation. It is possible that lipins play an important rôle in latex, and that the rubber globules with a covering layer of lipins and a viscous interior are suspended in an emulsion of protein. D. F. TWISS.

Plasticity determinations in crude rubber. VII. **Relation between structure and plasticity of rubber prepared in different ways.** O. DE VRIES and N. BEUMÉE-NIEUWLAND (Arch. Rubbercultuur, 1929, 13, [6]; Med. Proefstat. Rubber, 1929, [43], 283—370).—The soluble serum substances mechanically retained in rubber have a marked influence on the plasticity, a high proportion leading to hardening of the rubber on keeping. On the other hand, the substances, *e.g.*, proteins, precipitated with the rubber during coagulation, which may amount to 2% or more, have little influence on the plasticity. Coagulation of latex with papain, which decomposes part of the protein substances, also gives rubber of fairly normal plasticity. Centrifuging, or especially re-centrifuging of re-diluted cream, is the only method discovered so far to yield a plastic rubber without tendency to deterioration and tackiness on keeping. Treatment of latex with rubber solvents, such as benzene or toluene, which cause fusion of the globules to aggregates has little influence on plasticity of the dried rubber. D. F. TWISS.

Tensile strength of Java plantation rubber. O. DE VRIES and R. RIEBL (India-Rubber J., 1930, 79, 419—420).—The average figures for tensile strength obtained in the routine testing of crêpe and sheet rubber at the Buitenzorg testing station from 1923 to 1929 inclusive give no indication whatever of any deterioration. Examination of 983 additional samples drawn from as wide a range of estates as possible, using a 92½ : 7½ rubber-sulphur mixture, confirms that there is no

decrease in tensile strength. Complaints may have arisen from faulty technique or equipment in testing or from failure of European manufacturers to discriminate between first-grade plantation rubber and rubber from small native or Chinese estates. D. F. TWISS.

Difference between ultramarine and Thénard's blue in rubber mixtures. R. DITMAR and K. H. PREUSSE (*Gummi-Ztg.*, 1930, **44**, 1355—1356).—Although ultramarine imparts a more attractive colour than the more expensive Thénard's blue in rubber, unlike the latter it is not resistant to rigorous vulcanisation with sulphur chloride and is also less stable in hot vulcanisation in the presence of certain organic accelerators.

D. F. TWISS.

PATENTS.

Centrifugal treatment of rubber latex and the like. AKTIEB. SEPARATOR (B.P. 304,686, 10.1.29. Swed., 24.1.28).—Latex, especially ammonia-preserved latex, intended for centrifugal treatment is stored in a vertical tank of uniform cross-section, the surface being protected from contact with the air by a float. Sedimented impurities are prevented from reaching the centrifuge by drawing the liquid from immediately under the float through a flexible tube attached thereto.

D. F. TWISS.

Producing a pure rubber from rubber latex. AKTIEB. SEPARATOR (B.P. 301,085, 23.11.28. Holl., 24.11.27).—Latex, treated with a preservative such as ammonia, is subjected to centrifugal force so that impurities and precipitates are removed before coagulation; the purified latex has substantially the same sp. gr. and rubber content as the original latex.

D. F. TWISS.

Electrical separation of rubber from rubber latex. SIEMENS & HALSKE A.-G. (B.P. 307,747 and 325,401, 4.3.29. Ger., [A] 12.3.28, [B] 10.11.28).—(A) Electrically charged particles (*e.g.*, of rubber with or without sulphur, pigments, etc.) are separated from fluid suspension by means of a "balanced" alternating current, *i.e.*, a current representable by a sine curve for which the arithmetic mean is zero. The most suitable frequency is up to 150, and aluminium or an aluminium alloy is the best material for the electrodes, although tantalum and its alloys are also applicable. The electrodes may be covered with porous conducting or semi-conducting material, or with their metallic oxides, or may be separated from the suspension by porous diaphragms with water, an aqueous solution, or a dielectric fluid, *e.g.*, oil, alcohol, or glycerin, in the electrode space. (B) The yield of rubber obtained per watt by the preceding process is the greater the further the curve for the alternating current departs from a sinusoidal form. The desired type of current may be obtained by inserting choke coils and condensers in the circuit of an alternating current of unsatisfactory characteristics, by superposing two or more alternating currents which are out of phase with one another, or by generating from continuous current by means of a rotating commutator.

D. F. TWISS.

Manufacture of rubber compounds. RUBBER LATEX RES. CORP. (B.P. 302,151, 13.11.28. U.S., 10.12.27).—Rubber compositions, in granular or other

state of sub-division suitable for moulding purposes, are produced by treating granular fillers or fibrous materials, *e.g.*, asbestos, with a small proportion of appropriately diluted latex containing some proteid protective colloid, such as hæmoglobin, preferably together with zinc oxide. The treated material is then dried and reduced to a suitable form for moulding. The rubber content of such material may be as little as 6—8% and is satisfactorily resistant to oxidation.

D. F. TWISS.

Manufacture of artificial rubber and rubber-like masses. I. G. FARBENIND. A.-G. (B.P. 294,963, 1.8.28. Ger., 2.8.27).—The polymerisation of a butadiene or mixture of butadienes is expedited and the yield of product rendered almost quantitative by forming an emulsion of the hydrocarbon in the presence of water (or an aqueous solution of an emulsoid colloid, *e.g.*, albumin, dextrin, or glue) and of a hydrotropic emulsifying substance (*e.g.*, sodium tetrahydronaphthalenesulphonate or sodium protalbinat) and polymerising the emulsion thus obtained.

D. F. TWISS.

Manufacture of latex-like emulsions and rubber-like masses. I. G. FARBENIND. A.-G. (B.P. 301,515, 1.12.28. Ger., 2.12.27).—The polymerisation of butadiene, homologues thereof, or analogues containing the chain $\cdot\text{C}:\text{C}:\text{C}:\text{C}\cdot$ is greatly accelerated by the presence of a water-soluble salt of a bile acid, *e.g.*, sodium deoxycholate, or a derivative.

D. F. TWISS.

Manufacture of rubber articles. DUNLOP RUBBER CO., LTD., E. A. MURPHY, R. G. JAMES, and D. F. TWISS (B.P. 324,988, 6.10.28).—In the manufacture of articles from aqueous dispersions of rubber by dipping, spreading, electrophoresis, etc., a wrinkled or corrugated surface is imparted by bringing the uncoagulated layers of the dispersion into contact with single liquids, *e.g.*, carbon tetrachloride, or mixtures of liquids, *e.g.*, acetic acid and benzene, which effect coagulation and swelling concurrently, or alternatively into successive contact with two liquids, one of which, *e.g.*, acetic acid, produces coagulation and the other, *e.g.*, a mixture of benzene and alcohol, causes swelling. The subsequent "setting" of the wrinkled layer may be effected by merely drying by the action of heat, or by immersion in a dehydrating and setting bath; alternatively, wrinkling and setting can be effected concurrently by means of a fluid mixture of dehydrating and swelling agent such as a mixture of aqueous ammonium acetate, alcohol, and benzene.

D. F. TWISS.

Manufacture of colouring materials for colouring materials of the nature of rubber. I. G. FARBENIND. A.-G. (B.P. 325,967, 30.1.29. Addn. to B.P. 242,274; B., 1927, 186).—Materials for colouring rubber are produced by introducing into factice or a mixture thereof with glycerin, rubber-softening agents, anti-agers, vulcanisation accelerators, etc., large quantities of insoluble, dry pigment powders, *e.g.*, cadmium-yellow, and working the mixture until the pigment is in a state of colloidal or almost colloidal dispersion. The resulting coloured mixtures can be added to rubber without appreciable detriment to its properties.

D. F. TWISS.

Preservation of rubber and the like. B. F. GOODRICH CO., ASSEES. OF W. L. SEMON (B.P. 312,630, 2.11.28.

U.S., 29.5.28).—For preservation purposes rubber is treated, before or after vulcanisation, with a compound of the class represented by the formula $\text{NHR} \cdot \text{R}'' \cdot \text{NHR}'$, where R and R' represent phenyl groups and R'' is a *p*-phenylene group. At least one of the groups R, R', and R'' contains neutral or basic substituents. Acidic substituent groups impair the age-resisting qualities. Phenyl-*p*-diphenylamino-*p*-phenylenediamine, di- β -naphthyl-*p*-phenylenediamine compounds of the structure $\text{NHR} \cdot \text{C}_6\text{H}_4 \cdot \text{NH} \cdots \text{NH} \cdot \text{C}_6\text{H}_4 \cdot \text{NHR}'$ containing up to 6 amino-groups connected by phenylene groups, and their alkyl, aryl, amino-, hydroxy-, nitro-, and other substitution products are included. D. F. TWISS.

Manufacture of agents for use in vulcanising rubber. I. G. FARBENIND. A.-G. (B.P. 311,372, 20.4.29. Ger., 10.5.28).—Selenium or a selenium compound is incorporated in the colloidal state into natural or synthetic rubber or rubber latex. *E.g.*, dissolved selenium compounds may be added to rubber latex and the selenium precipitated therein in a highly dispersed condition by the addition of an acid such as acetic, formic, or sulphurous acid, or formaldehyde; coagulation of the rubber generally occurs concurrently. The resulting mixture can be used, directly or after washing and drying, as a vulcanising agent. D. F. TWISS.

Accelerator of vulcanisation. GOODYEAR TIRE & RUBBER Co., Assees. of J. TEPPEMA (B.P. 302,143, 27.8.28. U.S., 10.12.27).—Vulcanisation accelerators are made by condensing sodium salts of thiols, such as 2-thiolbenzthiazole or dithiocarbamic esters, with phenyl 3-nitro-6-bromothiophenyl ketone, $\text{PhCO} \cdot \text{C}_6\text{H}_3(\text{NO}_2) \cdot \text{SBr}$ ["4-nitro-2-benzoylphenyl sulphur bromide"]. C. HOLLINS.

Vulcanisation of rubber. I. G. FARBENIND. A.-G. (B.P. 300,208, 7.11.28. Ger., 8.11.27).—The bases themselves or the dithiocarbamates, thiuram mono- and disulphides, carbamides, guanidines, thiocarbamides, and aldehyde-ammonias, etc. derived from secondary, hydrogenated, cyclic bases or from secondary amines carrying a hydrogenated aromatic radical, are used as vulcanisation accelerators. Examples are: decahydroquinoline, zinc cyclohexylethyldithiocarbamate, and thiuram disulphide from decahydroquinoline. C. HOLLINS.

Plastic composition and its manufacture. F. WECKERLE (B.P. 325,312, 27.11.28).—Latex or rubber is mixed with a vulcanising agent (*e.g.*, up to 30% of sulphur), quinol, and a glucoside, *e.g.*, α -esculin, or a phenol-formaldehyde condensation product (to impart age-resistance), and fillers selected according to the purpose for which the composition is intended. The mixture is kneaded, with the addition of benzene if desired, and subsequently worked hot or cold, *e.g.*, on a calender, and pressed or turned into the desired shape. The mixture may also be dissolved, *e.g.*, in cyclohexanol, and applied to the desired surface by spraying with hot air. The composition is resistant to acid, heat, and swelling, and possesses high dielectric capacity. D. F. TWISS.

Plastics. RUBBER LATEX RES. CORP. (B.P. 301,077, 13.11.28. U.S., 25.11.27).—Aqueous liquids, particularly latex, are thickened by the addition of haemoglobin solution, with a small amount of zinc oxide. The pro-

ducts, which have batter-like consistency, form good binding material for composite articles containing, *e.g.*, fibres, rubber scrap, granular abrasives, etc. which may be added to the latex either before or after the thickening process. The additional introduction of formaldehyde gives increased tensile strength to the final products. D. F. TWISS.

Uniting rubber to metal. GOODYEAR TIRE & RUBBER Co., Assees. of S. S. KURTZ (B.P. 307,056, 12.2.29. U.S., 2.3.28).—Rubber is attached to metal, such as iron, by first applying to the latter a copper or cobalt salt, *e.g.*, an oleate, stearate, acetate, or chloride. The salt may be applied in solution in benzene or in a mixture of benzene and alcohol. The rubber layer is then pressed against the surface and vulcanised by heat. D. F. TWISS.

Uniting rubber and other substances. GOODYEAR TIRE & RUBBER Co., Assees. of L. B. SEBRELL (B.P. 310,461, 29.1.29. U.S., 26.4.28).—A suitable polymerising agent such as stannic chloride is allowed to act on a solution of rubber, containing, if desired, also a rubber softener, and the resulting solution is applied to the surfaces to be secured together, *e.g.*, cardboard, wood, textile material, iron, or steel. The treated surfaces are brought into contact and heated, *e.g.*, at 80–120° for 10–30 min., when strong union is effected. D. F. TWISS.

XV.—LEATHER; GLUE.

Sheep skin defects. I. H. BLANK and G. D. McLAUGHLIN (J. Amer. Leather Chem. Assoc., 1929, 24, 544–567).—Low tensile strength of sheep skins as compared with that of calf skin is due to the small amount of corium and the short and almost parallel fibre bundles. Wilson's conclusion that there are numerous sweat glands and fat cells in the thermostat layer which leave empty spaces in the finished leather is confirmed. Pinholes are due to the grouped wool fibres in fine-woolled skins. The depilant removes the partitions between the individual wool follicles, leaving a large hole. "Blind rib" is a ribbed appearance evident only when the skin is viewed by transmitted light. "Lap rib" consists of raised ridges on the surface of the skin, usually running in parallel ridges from the backbone towards the flanks. They are characterised either by an abnormal fatty deposit or an excessive fibrous structure at the upper edge of the corium. A similar deposition of fat, which, however, is not present in ridges, appears in skins showing loose grain. It weakens the skin at the junction of the corium and thermostat layer, and the mechanical working of the skin brings up the loose grain. Cockle is a cellular and vascular congestion. No abnormal amount of fat was observed in the cockled area, which appears as dark raised spots, chiefly on the shoulders and flanks. They become hard and brittle after tanning and will not take the dye. From 2 to 200 million bacteria per g. of pelt were observed on fresh sheepskins, of which 95% were on the wool, and up to 260 million on salted skins. Bacterial growth during soaking is reduced by a low temperature, the use of sea-water, and changing the soak water at frequent intervals during soaking. Drumming the skins in water prior to changing the soak water reduced the number of organisms per c.c. Fresh

skins should be put into soak water immediately after flaying, to minimise bacterial growth.

D. WOODROFFE.

Tannin content of *Acacia arabica* pods. ANON. (Bull. Imp. Inst., 1930, 28, 1—6).—The composition of 11 samples of pod-cases from *A. arabica* is described. The tannin content varied from 30.4 to 41.7% except in one Northern Nigerian sample, which contained 19%. "Goniake" pods from French West Africa (2 samples) contained 30.6 and 41.7% of tannin; Sant pods from the Sudan (3 samples) 30.4% (desert pods), 35.5%, and 39.2%; Northern Nigeria pods 19.0%, 33.0%, 34.7%, and 38.3%; Neb-neb pods from French West Africa 31.8%; *A. nilotica* pods from the Sudan 38.9%. With the exception of the 19% sample, all were superior in tannin content to those of the same variety grown in India (18.8—27.6% of tannin).

E. H. SHARPLES.

Detection and determination of chestnut-wood extract in a mixture of other tanning extracts. Report of a Committee of the American Leather Chemists' Association. F. F. MARSHALL (J. Amer. Leather Chem. Assoc., 1929, 24, 567—570).—The following test was made on several tannin extracts: 50 c.c. of tannin solution of analytical strength were mixed with 5 c.c. of 50% iodic acid, and after 5 min. the mixture was shaken with carbon tetrachloride to extract the iodine, the iodine solution washed free from iodic acid, mixed with 20 c.c. of 50% potassium iodide solution, and titrated with 0.02*N*-sodium thiosulphate. The result was calculated in terms of the percentage of iodine liberated. The following values were obtained: pyrocatechol tans <1%, chestnut 21.79%, valonia 21.5%, myrobalans 21.06%, European chestnut 18.77%. The test can be completed in 30 min.

D. WOODROFFE.

PATENTS.

Treatment of hides. C. J. M. M. LE PETIT (B.P. 300,615, 15.11.28. Fr., 16.11.27).—A plasmolysing salt (e.g., sodium sulphate or bicarbonate, magnesium sulphate), which may be anhydrous, or a mixture of anhydrous sodium sulphate, sodium bicarbonate, and boric acid, with or without a dehydrating agent, is added to the mould culture obtained as described in B.P. 250,907 (B., 1926, 1022).

D. WOODROFFE.

Leather manufacture. C. A. VENINO and A. AZZONI (B.P. 320,053, 26.6.28).—Dry leather is drummed with tannin for some time and then a solution of skin scraps or other gelatin-forming material is added. After this has been absorbed, the leather is finished as usual.

D. WOODROFFE.

Leather colour. W. RITZENTHALER (Swiss P. 123,512, 8.2.27).—The colour comprises a mixture of a pigment, a dissolved leather dye, castor oil, varnish, and a diluent (acetone and amyl acetate).

A. R. POWELL.

Manufacture of a casein adhesive. E. STERN (G.P. 460,141, 16.10.24. Addn. to G.P. 451,308; B., 1930, 71).—A dry mixture of casein and starch prepared as described in the prior patent is treated with sodium carbonate, sodium hydrogen phosphate, or ammonium chloride.

A. R. POWELL.

Manufacture [casting] of [glue-gelatin] impression transfer material. H. WADE. From OXFORD VARNISH CORP. (B.P. 325,615, 5.12.28).

XVI.—AGRICULTURE.

Clay formation and the weathering of feldspars.

O. TAMM (Medd. Stat. Skogsförsöksanst., 1929, 25, 1—28; Proc. Internat. Soc. Soil Sci., 1929, 4, 347—348).—Feldspars were pulverised in the presence of water in a mill producing ultimate particles of 2—0.2 μ in diam. In the case of microcline and oligoclase sufficient decomposition occurs to produce an alkaline reaction reaching p_H 10.7 and 11.1, respectively, for the two minerals. The decomposition products absorb water in amounts which increase with the fineness of grinding. This water is not removed by drying at 105—110° nor *in vacuo* over sulphuric acid at ordinary temperatures. The decomposed particles of microcline exhibit base-exchanging properties within the range p_H 6—10. Under acid conditions (p_H 6—3) there is a steady decomposition of the particles, and soluble alkalis and aluminium appear. Feldspar particles of less than 0.5 μ in diam. react with hydrogen-ions to an extent which increases with decreasing diameter of the particles. Particles of >2 μ in diam. are not appreciably reactive. The weathering of minerals in soil involves processes and materials similar to the above.

A. G. POLLARD.

Annual variations in the reaction of forest soils.

T. WŁOCZEWSKI (Lwów Nakł. Polskiego towarz. Leśn. Tłoczono pierwszej związkowej drukarni we Lwowie, 1928, Pt. 4; Proc. Internat. Soc. Soil Sci., 1929, 4, 351—352).—The humus layers showed greater variation in p_H value than the mineral strata, and, in general, were more acid. Humus and mineral soils showed maximum values in autumn and minimum in spring, although divergences from this rule were more common in mineral soils. Considerable differences in the p_H values of fresh and air-dried samples were observed. In humus soils seasonal variations were less definitely marked in the air-dried condition.

A. G. POLLARD.

Decomposition of forest litter. M. HELBIG and E. JUNG (Allgem. Forst- u. Jagdztg., 1929, 105, 336—344, 382—392; Bied. Zentr., 1930, 59, 109—110).—The micro-organic decomposition of forest litter is examined by means of the carbon dioxide produced. The rate of decomposition varies with the species of tree concerned. Temperature largely affects the rate of decomposition in the early stages, but its influence decreases later. Rates of decomposition of leaves of different species largely depend on the physical properties of the leaves and their mode of accumulation on the forest floor.

A. G. POLLARD.

Phosphate content of forest humus soils. A. NĚMEČ (Forstwiss. Zentr., 1929, 51, 721—732; Bied. Zentr., 1930, 59, 102—103).—In the forest area examined the soluble phosphate content was greatest in the surface covering (litter) of the soil, less in the humus layer, and least in the underlying mineral soil. Hydrochloric and hydrofluoric acid extracts give no indication of the assimilability of soil phosphates. The humus layer is the immediate source of phosphate for plants. Removal of the forest litter has led to serious reduction of the soil phosphate contents.

A. G. POLLARD.

Influence of the lime condition of soils on the solubility of phosphates. A. H. ERDENBRECHER (Superphosphat, 1929, 5, 213—214; Bied. Zentr., 1930, 59,

106—107).—The mobilisation of citric-soluble phosphates in soil results from favourable lime conditions. Liming favours the leaching of potash from soil.

A. G. POLLARD.

Neubauer tests for phosphoric acid [in soils] in the Brunswick area and confirmatory tests of the method. A. GEHRING (Superphosphat, 1929, 5, 207—209; Bied. Zentr., 1930, 59, 99—100).—Records of the root-soluble phosphate content (Neubauer) of numerous soils are given. The limiting value (4 mg./100 g.) is considered too low for practical purposes. A. G. POLLARD.

Determination of effective phosphoric acid [in soils] by Neubauer's method. K. KONISHI and G. MATSUKI (Soils and Fertilisers, Japan, 1929, 3, 10—29).—With rice, the addition of nitrogen, potash, and lime is necessary. Little absorption of phosphate occurs in alluvial soil.

CHEMICAL ABSTRACTS.

Determination of absorbable phosphate in soils by Neubauer's method. S. KASUGA (Soils and Fertilisers, Japan, 1929, 3, 31—50).—Young rice plants are used; the phosphate absorbed is increased by a suitable quantity of magnesium and sodium sulphates; hindrance of growth due to excess of one component salt is mitigated by use of calcium phosphate.

CHEMICAL ABSTRACTS.

Soil moisture determinations by the alcohol method. A. SMITH and F. W. FLINT (Soil Sci., 1930, 29, 101—107).—The alcohol method of Bouyoucos (B., 1927, 454, 887) is compared with the ordinary oven-drying method for determining soil moisture contents. The former is unreliable on fine-textured and alkali soils.

A. G. POLLARD.

Index of friability of soils. O. CHRISTENSEN (Soil Sci., 1930, 29, 119—135).—A compression apparatus is described for determining stress-strain curves for small cylindrical soil samples. Reproducible results can be obtained. Results are expressed mathematically in terms of a "friability index" and characteristic constants for individual soils. Relationships between the friability index and the soil composition and moisture content are discussed.

A. G. POLLARD.

Hydrogen-ion concentration of Egyptian soils. R. R. LE G. WORSLEY (Tech. Sci. Service Bull. No. 83, Govt. Press, Cairo, 1929, 33 pp.; Proc. Internat. Soc. Soil Sci., 1929, 4, 353).—Colorimetric measurements of the p_H values of alkali soils are unsatisfactory owing to the turbidity of the extracts. A suitable electrical method is described. Among the ameliorants examined, sulphuric acid was the most effective. Addition of neutral salts to alkali soils decreases their p_H as a result of base exchange. Leaching with water removes much calcium and increases the p_H values and impermeability, probably by the formation of sodium carbonate. A method for calculating the potential fertility of soils after the removal of soluble alkali is described.

A. G. POLLARD.

Basicity of Texas soils. G. S. FRAPS and E. C. CARLYLE (Texas Agric. Exp. Sta. Bull., 1929, No. 400, 20 pp.).—A discussion of the basicity and sulphur requirements of the soils.

CHEMICAL ABSTRACTS.

Nitrogen balance in cultivated semi-arid Western Kansas soils. P. L. GAINEX, M. C. SEWELL, and W. L.

LATSHAW (J. Amer. Soc. Agron., 1929, 21, 1130—1153).—Cropping in relation to nitrogen losses is considered.

CHEMICAL ABSTRACTS.

Significance of nitrogen in soil organic matter relationships. F. J. SIEVERS (J. Amer. Soc. Agron., 1930, 22, 10—13).—From analyses of New England soils it is deduced that the N : C ratio of soils is fairly constant, that variations in soil treatment have no consistent influence on the soil organic matter or the nitrogen or carbon content, that liming scarcely affects the soil organic matter content, and that there can be no apparent increase in soil organic matter without a proportionate increase in nitrogen.

CHEMICAL ABSTRACTS.

Neutralising values and rates of reaction with acid soils of different grades and kinds of liming materials. W. H. PIERRE (Soil Sci., 1930, 29, 137—158).—The interaction of ground limestone, oyster shell, and basic slag with acid soils is examined. The rate of reaction increases with the degree of soil acidity and with the fineness of grinding of the liming material. Coarser materials (20-mesh) do not react completely even after 2 years.

A. G. POLLARD.

Exchangeable calcium and potassium in soils as affected by cropping and fertilisation. B. D. WILSON (Soil Sci., 1930, 29, 91—100).—Limed and fertilised soils were examined after 15 years' cropping. No relationship existed between the amount of exchangeable calcium (by electrodialysis) in the soils and the quantity of lime applied. The p_H values of untreated soils and their exchangeable calcium contents were related, but the relationship failed on limed soil. Application of fertilisers had little or no effect on the exchangeable calcium or potassium content of soils. After continuous cropping, the proportion of exchangeable ions other than calcium was very small. No relationship exists between the proportion of colloidal matter in soil and its exchangeable calcium content. Cropping reduced the exchangeable calcium and potassium of untreated soils. It is suggested that the exchangeable calcium content of limed soil depends on the specific adsorptive power of the soil and not on the amount of lime applied, and that therefore the hydrogen-ion concentration of a soil is no criterion of its exchangeable calcium content.

A. G. POLLARD.

Fixation of the potash of a green manure by liming materials. W. H. MACINTYRE and K. B. SANDERS (Soil Sci., 1930, 29, 109—117).—Lysimeter experiments show that the addition of lime, limestone, or dolomite decreased the amount of potash leached from soils with which ground red clover hay had been incorporated. The proportion of potash leached in different seasons was not proportional to the rainfall.

A. G. POLLARD.

Biological effects of certain nitrogen fertilisers. P. E. BROWN and F. H. MENDELL (Proc. Iowa Acad. Sci., 1928, 35, 87—95).—The results of nitrification studies depend on the methods used. Sodium nitrate, but not other nitrogen fertilisers, stimulated nitrification in the Carrington loam employed when applied at the rate of 200 lb. per acre. Smaller quantities caused no increases.

CHEMICAL ABSTRACTS.

Reaction of the soil and agricultural crops. A. LØDDEØL (Meld. Norges Landsbruks., 1928, 8, No. 6; Proc. Internat. Soc. Soil Sci., 1929, 4, 348—351).—The sampling of soils is discussed and the probable error involved in surveying large areas for p_H values and lime requirements is examined. A. G. POLLARD.

Hydrogen-ion concentration of the sap as a factor in plant metabolism. W. F. LOEWING (Proc. Iowa Acad. Sci., 1928, 35, 135—139).—Increased absorption of certain mineral nutrients by plants on unproductive muck soils involves diminished intake of others. Shifts in the internal nutrient balance are correlated with marked changes in the hydrogen-ion concentration of the sap. Profound alteration in the soil solution is believed to follow the use of lime or potash; ionic replacement in the colloidal fraction of the soils and preferential absorption of certain ions by plants following addition of mineral fertilisers may render the soil solution toxic. Differences in response to lime and potash applications on muck and mineral soils are differences chiefly in degree.

CHEMICAL ABSTRACTS.

Differences and relationships between varieties of wheat and barley in their resistance to the toxic action of potassium chlorate. M. YAMASAKI (J. Imp. Agric. Exp. Sta., Tokyo, 1929, 1, [2]; Bied. Zentr., 1930, 59, 121—122).—Seedlings of many varieties of wheat and barley were treated with potassium chlorate solutions of varying concentration. Resistance to potassium chlorate and cold-resistance were generally parallel. Early varieties were more resistant than late ones. High growth temperature and a deficiency of nutrients tended to weaken resistance to potassium chlorate. Resistance decreased with the age of the plants. High cell-sap concentration is associated with low resistance, and *vice versa*. Although the amount of water absorbed from chlorate solutions by resistant varieties was greater than that of less resistant varieties, their total intake of chlorate was less. Differences in resistance are not due to different concentrations of the cell sap, but depend on the semipermeability of the root-cell membranes. A. G. POLLARD.

Consumption of nutrients and the process of their absorption by various vegetables. H. LIESEGANG (Gartenbauwiss., 1929, 2, 418—455; Bied. Zentr., 1930, 59, 118—119).—Economic applications of fertilisers for a number of vegetables may be calculated from the known mineral contents of the plants, using the factors 0.67 for nitrogen, 1.25 for potash, and 1.50 for phosphate. Curves showing the course of the intake of nutrients are given and discussed. A. G. POLLARD.

Changes in the carbohydrate constituents and feeding value of mangolds from October to March. S. D. F. HARWOOD and H. MARTIN (J. South Eastern Agric. Coll. Wye, 1928, No. 25, 200—202).—The content of dry matter and of sucrose was highest in December and lowest in March; reducing sugars increased from December (0.173%) to March (1.665%).

CHEMICAL ABSTRACTS.

Neubauer values of some [Czechoslovakian] soils. J. SUK (Z. Zuckerind. Czechoslov., 1930, 54, 349—351).

Action of sulphur dioxide on natural phosphates. CALCAGNI.—See VII. **Agricultural value of sugar-factory mud.** BROADBENT. **Disposal of sugar-factory waste waters.** MONTGOMERY.—See XVII. **Sardine waste products.** YOSHIMURA and others.—See XIX.

See also A., Apr., 502, **Counting soil *Actinomyces*** (RAO and SUBRAHMANYAN). 507, **Assimilation of nitrate by asparagus in absence of light** (NIGHTINGALE and SCHERMERHORN). 508, **Relation of potassium to properties and functions of the leaf** (JAMES).

PATENTS.

Production of material containing a high percentage of calcium nitrate and capable of being readily strewn. F. UHDE (B.P. 308,717, 5.3.29. Ger., 27.3.28).—Potassium, sodium, or magnesium nitrate is added to a solution of calcium nitrate, in such quantity that the product contains 5—20% thereof. The added nitrate may be formed *in situ* from the corresponding sulphate, and is preferably introduced into the hot calcium nitrate solution seeded with the final product. F. G. CLARKE.

Manufacture of manures [phosphatic fertilisers]. E. F. EHRHARDT. From H. WIGGLESWORTH, U. ORLANDI, and G. LEVI (B.P. 326,814, 14.11.28).—Phosphatic material is treated in three stages with a countercurrent of a mineral acid, the mother-liquors from a previous operation being added during the intermediate stage, in the presence of ammonium or an alkali sulphate in quantity sufficient to yield an easily filterable, complex ammonium or alkali calcium sulphate, and the phosphoric acid solution obtained is treated with sufficient ammonia in the presence of a sodium salt to yield ammonium sodium hydrogen phosphate, which is recovered from the solution. Mixed fertilisers containing potassium and nitrogenous salts in addition to the main product may be obtained, *e.g.*, by adding sodium nitrate or excess potassium or ammonium sulphate to the mineral acid used for treating the phosphatic material. The precipitated complex sulphates are treated with ammonia and carbon dioxide to recover the alkali sulphates together with ammonium sulphate. L. A. COLES.

Manufacture of mixed fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,529, 16.11.28).—To a hot liquid mixture containing ammonium nitrate, a second fertiliser, and 10% or less of water is added 6% or less of one or more substances which, in dissolving in the liquid, increases its surface tension; the mixture is then dispersed into solid globules. The added substance is preferably also a fertiliser, *e.g.*, urea or calcium nitrate. F. G. CLARKE.

Instruments for testing or comparing the physical properties [penetrability] of soil. (SR) E. O. WILLIAMS (B.P. 326,954, 14.2.29).—A tube is provided with a cylindrical foot for applying to the soil and with a collar for holding weights or for receiving impulses by sliding the weights down the upper section of the tube. The tube is made in two sections which can be connected at the collar, and the foot is preferably of 2½ in. diam., so that the application of a 28 lb. weight corresponds to a load of ½ ton/ft.² L. A. COLES.

Granular chemicals (G.P. 453,366).—See VII.
Fungicides (B.P. 326,460).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Pan boiling [control of cane sugar]. S. J. SAINT (Trop. Agric., 1930, 7, 3—8).—A continuation of earlier work on the subject (cf. B., 1929, 655). Suggestions are made for the more efficient boiling of final low-purity strikes, using the Zeiss industrial refractometer. By raising the true coefficient of supersaturation of the mother-liquor of the low-purity strike to 1.35 before striking into the crystalliser, a considerably greater extraction of sucrose from the final molasses could be obtained. This can be done with safety with the aid of the refractometer, whereas ordinarily a pan-boiler without such a means of control is cautious of raising the concentration in case false grain may form. It was also shown that the so-called vacuum method of graining, which yields a uniform grain, can be as readily controlled with the aid of the refractometer as the "waiting" method. J. P. OGILVIE.

Pressure evaporation in cane-sugar factories. P. HONIG and J. F. BOGTSTRA (Archief, III Meded., 1929, No. 14, 689—834).—Working in two cane-sugar factories in Java with a standard triple-effect evaporator without vacuum, the juice temperatures being, respectively, 114°, 108°, and 102°, continuous tests showed that, provided the tubes are kept clean, no fall in the purity nor of the glucose ratio can be detected during concentration to 50° Brix. In the case of juice clarified by the sulphitation process, however, the scale forming on the tubes was found greatly to lower the efficiency of the apparatus, much more so than in ordinary vacuum evaporation. In fact, pressure evaporation cannot be recommended for juice thus clarified, nor for defecation juice, though it appears to be practicable with carbonatation juice. J. P. OGILVIE.

Removal of incrustation from [sugar-factory] evaporator and heater tubes. H. B. SPRINGER (Trop. Agric., 1929, 6, 235).—Incrustation in evaporator and heater tubes (consisting largely of calcium silicate and sulphate with some phosphate) was softened by boiling with a 2% solution of ammonium hydrogen fluoride during 3 hrs. under 1 in. vacuum, the used solution being made up to its original concentration and used the following week. In this way scraping of the scale was greatly facilitated, and comparatively large quantities were removed from each of the last calandrias. Though the cost of cleaning was increased by £4 per week by the use of this reagent, this extra cost was more than counterbalanced by an increased evaporative capacity of about 25%, making possible an increase in maceration. Less steam had to be used in single effect, and the various power units operated against a lower back pressure. J. P. OGILVIE.

Sucrose losses in crystallisers during cooling. F. H. KING and E. B. JAMORA (Sugar News, 1930, 11, 19—27).—Observations made of the dextrose content per 100° Brix during the cooling of massecuites from about 74° to about 50° show that sucrose was lost to the extent of 2.93% as the average of tests carried

out in 12 crystallisers during varying periods of time from 24 to 192 hrs. This for a factory grinding 1000 tons per day would mean a loss of about 0.833 ton per day. The sugar decomposition was greatest when the temperature was initially highest, and it is also controlled to some extent by the purity of the product. Acids were rapidly formed during the period when the massecuites was maintained at its highest temperature. The number of micro-organisms remained practically constant during the cooling.

J. P. OGILVIE.

Use of high-power machines for centrifuging raw [beet] sugars. V. STANEK and K. SANDERA (Z. Zuckerind. Czechoslov., 1930, 54, 305—310).—Bergé's process (Austr.P. 115,646; cf. B.P. 290,265; B., 1929, 618) relating to the use of high-power centrifugal machines for affining raw sugars was examined. Using a machine having diam. 32 cm. and speed 2500 r.p.m., it was found possible to free raw beet sugar from the greater part of its adhering syrup, thus opening up the possibility of eliminating an important proportion of its molasses from refining operations, and of thus shortening after-product working without the addition of syrups in affining, and consequently without loss of sugar by dissolution of the crystals in the wash-liquors. J. P. OGILVIE.

Maceration of filter-cake in defecation factories. C. J. E. STOK (Archief, 1929, 37, 1102—1107; Internat. Sugar J., 1930, 32, 139).—In factories grinding the new cane POJ 2878, which contains a relatively high proportion of wax, the filtration of the mud was found to be difficult, the percentage of sucrose in the cake obtained being unreasonably high. Therefore, after steaming out, the cake was dropped into a mixer, where it was diluted to a thin paste with condensed water from the 3rd and 4th bodies of the evaporator, being then filtered through two Kroog presses. In this way it was possible to reduce the sucrose content of the cake from about 7% to 1.19%. J. P. OGILVIE.

Changes in the composition of [sugar-factory] filter-cloth with use. P. HONIG (Vers. Vereen. van Adviseurs [Java], 1929, No. 4, 158—162; Internat. Sugar J., 1930, 32, 138).—Analysis of a filter-cloth which had been in use in a defecation sugar factory gave wax 4.91% and ash 38.37%. This ash contained 37.60% ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$), 26.06% CaO , and 19.58% P_2O_5 . Washing with hydrochloric acid up to 8% failed to remove the accumulated impurities sufficiently and deteriorated the fabric. J. P. OGILVIE.

Use of fats for subduing froth in carbonatation. V. STANEK and J. VONDRÁK (Z. Zuckerind. Czechoslov., 1930, 54, 353—360).—Emulsified rape oil gave less satisfactory results than the straight oil in combating the froth arising during carbonatation in the beet-sugar factory. Experiments showed tallow, coconut oil, and lanoline also to be advantageous. J. P. OGILVIE.

Classification of sugars. P. HONIG (Archief, 1929, 37, 949—957).—A comparison of the figures for the polarisation, reducing sugars, ash (by incineration), conductivity, p_H , and colour for beet white granulated made in Holland with Java plantation white sugars leads

to the conclusion that the purity is almost the same, excepting that in the latter the reducing sugar and colour values are greater. Especially is the quantity of colour around the crystal a little greater in the plantation white than in the Dutch beet white, from which it follows that the Java product must be better washed in the centrifugals to be equivalent to European white sugars. If the technique of washing plantation white sugars were improved by using a better quality of covering liquor, by boiling a more uniform grain, and by more careful washing in the centrifugals with water and steam, a sugar could be made corresponding in composition and in appearance to the best European semi-refined grades.

J. P. OGILVIE.

Maple syrup colour standards. R. T. BALCH (Ind. Eng. Chem., 1930, 22, 255—257).—Bryan's colour standards, prepared from sugar caramelised under prescribed conditions and diluted with glycerin (B., 1911, 299), are liable to differ considerably. The absorption spectrum of maple syrup is very similar to that of caramel solutions. By use of a spectrophotometer or a Pfund colour grader accurate standards can be prepared from any caramel solution, once the percentage transmissions for any given wave-length (e.g., 560 m μ in a 1-cm. cell) or the Pfund grader readings have been established for the Bryan standards. This has been done by the author, who also suggests slight modifications in the relative dilutions of the Bryan standards.

J. H. LANE.

Measurement of colour in the sugar industry. P. HONIG and J. F. BOGTSTRA (Archief, III Meded., 1928, No. 1, 1—35).—Factors affecting the colour of sugar solutions are the p_H , the concentration (a liquid may be yellow when dilute, but reddish when in the state of syrup), and the degree of dispersion of particles that may be present in it. Lundén's amethyst shade (cf. B., 1927, 395) is the colour showing a strong absorption in the yellow part of the spectrum, and juices showing it must be regarded as inferior. It is very difficult to eliminate, and it crystallises out with the sugars. Attempts were made to obtain measurements of the colours of Java white sugars (using inferior samples), but the Hess-Ives photometer failed to do so, only the extinction in the left part of the spectrum being measured. So-called "colour curves," obtained by plotting the extinction coefficient against the wave-length at p_H 2, 7, 10, and 12 for raw, clarified, and evaporated juices at different periods through the season, demonstrated the varying colour quality, especially in respect of the amethyst shade.

J. P. OGILVIE.

Determination of sugar in the beet. G. R. CLARKE, L. F. NEWMAN, and A. W. LING (J. Min. Agric., 1930, 36, 1061—1068).—In preparing the beet pulp for analysis, the use of the "Sans Pareille" press is too tedious, whilst a circular saw removes only a flat section of definite thickness. On the other hand, the conical rasp gives an excellent pulp and the proportion removed is a true fraction of the entire root. A comparative examination of methods of determining sucrose showed that, compared with the Krüger process, the Sachs-le Docte method appears to be the more reliable. It is also more convenient to the routine analyst, owing to its

simple manipulative details and the rapidity with which a large number of samples can be examined.

J. P. OGILVIE.

Constant-weight and constant-volume methods [of determining sucrose in the beet]. E. SAILLARD (Sucr. Belge, 1930, 49, 184—188).—When the constant-weight method was established, the typical beet was assumed to have an average mark content of 4.75%, and to contain juice of sp. gr. 1.075, but now the average beet contains more marc, and a correction is here necessary. In the last campaign, in fact, roots having 6.2% of marc and 22.5% of sugar were largely encountered. The Kaiser-Lewenberg method is not to be recommended.

J. P. OGILVIE.

Determination of the amount of crystal in masse-cuites, using the electrical conductivity method. K. SANDERA (Z. Zuckerind. Czechoslov., 1930, 54, 361—364).—In place of calculations based on formulæ employing the dry substance or polarisation of the masse-cuite, electrical conductivity measurements afford a more reliable method for the calculation of the amount of crystal to be expected, and are besides considerably more expeditious.

J. P. OGILVIE.

Defecation control by the hydrogen-ion method. J. H. PARDO (Facts about Sugar, 1930, 25, 220).—Prior to the introduction of p_H control in a cane-sugar factory in Peru, the juice was limed to a "slight alkalinity" using test-paper, which point was found to be about p_H 7.2. On putting the colorimetric method of hydrogen-ion control into use, it was found that the juice clarified best when limed to p_H 8.0—8.2, but, as this high alkalinity considerably delayed the time of settling, the liming was reduced to p_H 7.8. Whereas with the old method of control there had been a fall in the purity between clarified juice and syrup, after instituting hydrogen-ion control this was changed into an increase, there being also a reduction of inversion losses and an increase in the rate of filtration. Generally, phenol-red was used.

J. P. OGILVIE.

Determination of sugar in molasses from Uba cane. E. HADDON (S. Afr. Sugar J., 1929, 13, 833).—In molasses from Uba cane juice, optically active substances are present, probably dextrins hydrolysed from the starch. Previous to polarising, treatment for their elimination is necessary, and the author advises boiling with baryta, addition of basic lead acetate, filtration, addition of ammonia, and refiltration.

J. P. OGILVIE.

Determination of the true sugar content of carbonatation cakes [in beet-sugar factories]. O. SPENGLER (Z. Ver. deut. Zucker-Ind., 1930, 80, 69—80).—In 3 out of 20 German beet-sugar factories visited by the author last campaign the carbonatation cakes contained considerably more sugar (30—100% more) than was indicated by the usual ammonium nitrate and acetic acid methods. As a provisional method for determining the true sugar content in such cases it is proposed to heat 53 g. of the cake in a stoppered nickel beaker or wide-necked glass flask, in a water-bath at 85—90° for 1 hr., the stopper being tightened after the first minute's heating, and then to add 177 c.c. of

ammonium nitrate solution, stir until the cake is completely disintegrated, cool, and polarise as usual.

J. H. LANE.

Crystallisation of "uncrystallisable" syrups. K. SANDERA (Z. Zuckerind. Czechoslov., 1930, 54, 333—338).—Experiments are described in which it was found that if "exhausted" beet-factory molasses is placed between two glass plates in a layer 0.05—2.0 mm. thick it undergoes at ordinary temperatures further crystallisation around the edges. Beet-factory molasses, therefore, is not a stable system, as has been stated. By the improvement of after-product work it should be possible to raise the yield of sugar in the factory.

J. P. OGILVIE.

Electrodialysis of exhausted molasses. N. KAMEYAMA and J. KATO (J. Soc. Chem. Ind., Japan, 1928, 31, 201—202 B).—Diluted molasses was dialysed in the centre chamber of a three-chambered dialyser having platinum electrodes immersed in water in the outer chambers. The diaphragms were of parchment paper on which copper ferrocyanide had been precipitated. The voltage employed was 70—100 volts, and the current was 1 amp. per 100 g. of molasses. In 4 hrs. the ash content of the molasses was reduced from 10.06% to 1.61—1.86%, the alkali salts being removed more completely than those of the alkaline earths; the sucrose content fell from 34.33% to 29.3%, and the invert sugar content increased from 16.57% to 17.16—17.6%. The molasses became acid, but inversion was restricted by maintaining temperatures below 40°. The chief loss of sugar seems to have been by transformation into acids, the amount of sugar passing into the outer chambers being small.

J. H. LANE.

Sugar losses in beet-sugar factories and the recovery of sugar contained in factory waste products. I. Sugar losses. H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1930, 80, 55—68).—The calculation of sugar losses should be based on the weight of washed roots as weighed before slicing. In many factories a deduction is made from this weight for adhering moisture and residual dirt on the roots and, in consequence, the sugar loss calculated on beets is 0.3—0.9% too low. The moisture and dirt adhering to the weighing machine, however, should be treated as part of the tare and deducted from the apparent weight of roots. Properly calculated, the total sugar losses in the factory usually amount to 1.25—1.5% of the beets. Of this total 0.5—0.6% represents sugar lost in waste products, mainly in exhausted slices, waste waters, and filter-press cake. The balance, 0.75—0.9%, represents the so-called undetermined loss. Part of it is due to causes which can be specified but not accurately assessed. Thus incorrect tare of the weighing machine, drying out of the fresh slices between sampling and analysis, and low results in the usual methods of determining sugar in filter-press cake may each account for 0.05—0.1%. Lowering of polarisation of the juice due to the action of lime may account for 0.1%, and destruction of sugar during heating and evaporation of the juice may add a further 0.02%. There still remains, however, a loss of about 0.4—0.5% of sugar (calc. on beets) for which no explanation is forthcoming.

J. H. LANE.

Composition and agricultural value of [sugar] factory mud (press cake). F. W. BROADBENT (Internat. Sugar J., 1930, 32, 216).—Calculated on the solid matter, factory mud contains: CaO 11.33, N 1.85, P₂O₅ 8.89, and K₂O 0.37%. In Hawaii the mud mixed with water added to give about 14% of solids is being distributed over the fields, where it is used at the rate of 0.0403 ton of wet mud per ton of cane.

J. P. OGILVIE.

Waste waters from beet-sugar factories and suggestions for their disposal. W. MONTGOMERY (Internat. Sugar J., 1929, 31, 545—549, 602—608).—Analyses are given of fluming water, pulp waste water, silo drainage waste water, lime-cake drainage waste water, and final waste water. A method of treatment comprises the separation of the waste waters at the mill, the fluming water being submitted to fine screening and its grit removed, cooled by means of spray towers, and stored in reservoirs for re-use. Finally it is proposed to dispose of these wastes for the irrigation of land, utilising the check method of application with sub-surface pipe or surface ditch.

J. P. OGILVIE.

Determination of the solubility of dextrin. PARLOW (Z. Spiritusind., 1930, 53, 52).—10 G. of dextrin are stirred for ½ hr. in a beaker with 500 c.c. of water at 20°, and 25 c.c. of the clear filtrate are carefully evaporated to dryness in a weighing glass. The bottom of the weighing glass is covered with a layer of sea sand which has been previously washed and dried at the temperature of evaporation after extraction with acid. The sand ensures more rapid and more regular evaporation, and by lessening the tension when the dextrin solution becomes solid prevents the cracking of the thin-walled weighing glass during cooling.

C. RANKEN

Influence of the dry summer of 1929 on the tenacity of potato flour. SCHULZ and PARLOW (Z. Spiritusind., 1930, 53, 85).—Not only is the tenacity of starch flour influenced by the soil, method of drying the starch, and the size of the starch grains, but the climatic conditions also have their effect. The dry summer of 1929 has yielded potato flours with very low tenacity values. Using Wolf's standard starch for comparison, the average values for the tenacity of the potato starches of 1929 approximated to 120%, compared with 150% for the previous year.

C. RANKEN.

Alcohol and yeast manufacture. STICH.—See XVIII.

See also A., Apr., 499, **Refractometric measurement of invertase action** (GORBACH).

PATENTS.

Manufacture of concentrated grape juice [to obtain dextrose]. SOC. DES ÉTABL. BARBET (Addn. F.P. 32,711, 8.7.26, to F.P. 615,942; B., 1927, 856).—The solution is concentrated in a high vacuum produced by a two-stage pump until $d_{1.4}$ is exceeded; on cooling, dextrose separates in crystals.

A. R. POWELL.

Wheat [malt] syrup and its manufacture. F. H. ROGERS. FROM INTERNAT. WHEAT MALT SYRUP Co. (B.P. 326,264, 17.12.28).—Ground wheat malt is mashed

with water at approx. 42.5° , and 1 hr. later is heated by successive increments alternated with rest intervals to between 68° and 80° , the final temperature depending on whether a high or low content of sugars is required. The liquid is then separated from the solid portion and evaporated to the desired consistency. C. RANKEN.

XVIII.—FERMENTATION INDUSTRIES.

Metafiltration and the brewing industry. J. A. PICKARD (J. Inst. Brew., 1930, 36, 179—188).—The filter is built up of strips of metal piled one upon the other. On one side the strips carry bevelled edges and a central flat part down the middle of which is a slightly raised rib. When the strips are held in frames, the bevelled edges constitute V-shaped grooves through which the liquid flows from the outside of the strips, whilst regularly spaced perforations in the strips come into register and provide the outlet channel for the drainage of the filtered liquid. Filtration is improved by a filter-bed of kieselguhr deposited in the V-shaped grooves, and when beer is being filtered it is an advantage to add to the stream of beer a further small quantity of kieselguhr to counteract the impervious nature of the deposit and prevent the rapid giving out of the filter. The filter can be used at any pressure and at any temperature, and can be cleaned and sterilised in a few minutes. C. RANKEN.

Action of high pressure of carbon dioxide on yeast fermentation. F. WINDISCH (Woch. Brau., 1930, 47, 82—83).—It has been shown by Lieske and Hofmann (A., 1929, 1199) that whereas above 100 atm. pressure of an inert gas is required to check fermentation, about 40 atm. pressure of carbon dioxide brings the process to a standstill. The author gives a comparison of parallel fermentations under 1.0, 1.4, and 2.0 atm. pressure of carbon dioxide, which show a very slight falling off in the rate of fermentation with increasing pressure. Since he finds that the biological condition of the yeast is improved by pressure, he considers that the inhibiting effect is due to the effect of the dissolved carbon dioxide on the reaction of the medium rather than to any poisonous effect it exerts on the yeast. It is suggested that as no means are available for determining the p_H in pressure vessels, the question might be answered by comparing the behaviour of buffered and unbuffered fermentations under pressure. F. E. DAY.

Electrodialysis of wort and beer. W. WINDISCH, P. KOLBACH, and E. BORGES (Woch. Brau., 1930, 47, 67—71, 77—81).—Wort and beer were submitted to electrodialysis under a *P.D.* of 100—120 volts for about 6 hrs. The current passing increased from about 0.5 amp. to about 1 amp. during the first 15 min., then decreased to about 0.1 amp. Cooling had to be employed. The nature of the membrane has a strong influence on the rate of dialysis, and the most rapid results were obtained by using leather as anode diaphragm and certain Zsigmondy ultrafilters, especially "cella-filter," as cathode diaphragm. With this combination only 2% of the ash of wort remained after 2 hrs. The effect of using various membranes on the electrodialysis of different classes of constituents was studied.

A wort and the beer from it were electrodialysed for 6 hrs., and the buffer titrations compared with those of the same materials before and after treatment with baryta, and with those of the latter after electrodialysis. Determinations of inorganic and phytin phosphorus and of total and formol nitrogen were also made. By using suitable membrane combinations phosphates could be almost completely removed, without great alteration of the nitrogenous constituents, and from the buffering of the resulting liquids the isoelectric zone of the proteins and their decomposition products could be determined. F. E. DAY.

Clarification [of wine] with potassium ferrocyanide. O. REICHARD (Z. Unters. Lebensm., 1929, 58, 488—505).—Clarification resolves itself into three operations: firstly, a test to determine whether iron compounds are the cause of the turbidity; secondly, measurement of the amount of potassium ferrocyanide needed; and thirdly, a test for the completeness of the process. (i) To determine the nature of the iron compounds, 20 c.c. of wine are centrifuged, whereon a yellow deposit indicates phosphate and a black one tannate; a mixture of the two may also be present. The sediment is taken up with water slightly acidified with sulphuric acid and tested for iron with potassium ferrocyanide. Microscopical examination of the deposit gives confirmatory evidence. The iron content may be insufficient to cause a deposit in young wine, but will do so after storage; in such cases determination of total iron by colorimetric methods is necessary. The iron content capable of causing turbidity differs from one wine to another, and may vary between 5 and 15 mg./litre, according to the relative amounts of malic and tartaric acids present. Oxidation by keeping and stirring in open vessels or the addition of 3 drops of hydrogen peroxide to 100 c.c. of wine, stored in the dark, will determine whether turbidity is likely to develop. (ii) Exact determination of the amount of potassium ferrocyanide to be added is most essential, but since an excess is to be guarded against it is customary to add sufficient to remove all but 2 mg./litre of iron. The requisite amount is determined by titration in which to 5 samples of wine (each 10 c.c.) are added 0.1, 0.3, 0.5, 0.7, 0.9 c.c. of potassium ferrocyanide solution (0.50 g. in 100 c.c.), respectively, 1 c.c. of gelatin solution (0.2 g. in 100 c.c. of aqueous alcohol containing 1.0 g. of tartaric acid), and 1 c.c. of tannin solution (0.2 g. in 100 c.c. of aqueous alcohol). The liquids, after filtration, are divided and tested for iron and potassium ferrocyanide. The test is then repeated, using smaller volumes of ferrocyanide. The number of g. per litre required is called the "clarification number" ("Schönungszahl"), but actually 0.04—0.05 g./litre less than this figure is used in the clarification. Owing to the variations in iron content even amongst different batches of the same wine, every barrel must be tested separately, the samples being taken with every precaution. All wines which have been treated must subsequently be filtered, whether a deposit be visible or not. (iii) The final proving consists of a test for excess potassium ferrocyanide.

H. J. DOWDEN.

Description of gin. W. LANGE (Chem.-Ztg., 1930, 54, 230—231).—Gin prepared exclusively from juniper berries comes under the protection of the brandy monopoly law. Hence the name "Wacholder" (juniper) applied to a mixture of juniper essence and diluted spirit is a false description and should be changed.

J. H. BIRKINSHAW.

Description of gin. A. JUCKENACK (Chem.-Ztg., 1930, 54, 231—232; cf. preceding abstract).—It is not by any means clear whether the brandy monopoly law is intended to apply to gin. J. H. BIRKINSHAW.

Alcoholometry. F. BORDAS and F. TOUPLAIN (Ann. Falsif., 1930, 23, 84—102).—The International Conference (June, 1929) having fixed 20° as the temperature for alcoholometric measurement, the authors have established formulæ for inter-conversion of results at this temperature and at 15°, the legal tables in France being based on d_{4}^{15} of absolute alcohol *in vacuo*. Attention is incidentally drawn to the fact, not generally recognised in commercial transactions, that the alcoholometric calculations are based on weights *in vacuo*.

E. B. HUGHES.

Rectification of raw spirit and the manufacture of absolute alcohol. W. SCHLAGE (Z. Spiritusind., 1930, 53, 49—51).—Raw spirit is rectified by periodic and continuous distillation methods and yields a spirit containing 94.4—94.8% by wt. of alcohol. The yield of "fine" or "prima" spirit is higher with the continuous process. A purer alcohol is obtained by filtering the raw spirit through charcoal prior to the distillation. For the manufacture of absolute alcohol dehydration of the rectified alcohol by lime is still in use, but is steadily being replaced by methods in which pure benzene, fine benzene (b.p. 80—110°), or, better, a benzene-benzene mixture (b.p. 94—104°) is used as the dehydrating agent. Most of the absolute alcohol is manufactured by the continuous distillation process either at ordinary or under 10 atm. pressure. The manufacture of 1 hectolitre of absolute alcohol by the periodic system requires 475—500 kg. of steam, whereas 155—185 kg. are sufficient in the continuous system.

C. RANKEN.

Economics of alcohol and yeast factories. E. G. STICH (Chem.-Ztg., 1930, 54, 217—220, 238).—In the manufacture of alcohol and yeast from molasses the chief factors for economic working are extremely fine division of the air supplied and strict regulation of the air and mineral nutrients in accordance with the rate of multiplication of the yeast. Fermentation in stages, employing a series of small vats, leads to a considerable saving in total vat capacity, and therefore in installation costs. Comparative cost figures for the old and new processes are given.

J. H. BIRKINSHAW.

Observations on the Stich fine-aeration process. P. LINDNER (Chem.-Ztg., 1930, 54, 238—240).—The easy assimilation of alcohol by yeast in presence of air leads to a comparison of the human respiratory system with the Stich fine-aeration process. Attention is drawn to the glycogen and fat formation in yeast. When all the oxygen of the cell is used up, no further growth is possible and synthesis of glycogen may occur. Aeration in a dilute alcohol solution leads to formation of fat: the fatty cells are incapable of further reproduc-

tion. The best temperature for fermentation (about 37°) differs from the optimum for growth, which is about 25°.

J. H. BIRKINSHAW.

Importance of hydrogen-ion concentration in potato-spirit distilleries. B. DREWS (Z. Spiritusind., 1930, 53, 91—93).—The p_H value of the liquid expressed from raw or steamed potatoes is approx. 5.9, whilst that of the sweet wort prepared from potatoes and malt varies between 5.1 and 5.3 and falls within the zone of optimum action of the diastase. Lactic acid is preferable to sulphuric acid for the acidification of the mash, as the former acid is less dissociated and during its bacterial formation is accompanied by a simultaneous increase in the content of buffer substances. A high buffer content prevents too great a decrease of the p_H value of the mash below 4.5, which is the optimum value for alcoholic fermentation and for the after-saccharification necessary for the attainment of a completely fermented mash. To retain the mash at approximately this value when sulphuric acid acidification is used, a greater proportion of green malt must be added and a more concentrated mash used.

C. RANKEN.

Beer-slop waste from corn-mash distillation [in butyl alcohol manufacture]. W. D. HATFIELD (Ind. Eng. Chem., 1930, 22, 276—277).—In a butyl alcohol factory working 20,000 bushels of maize per day, over 1.25 million gals. of distillation residue (beer slop) are obtained daily. This slop, which has p_H 4.6—5.0, contains 11,000 pts. per million of total solids, of which about 4000 pts. are in suspension. Its total nitrogen content is 500 p.p.m., and its oxygen absorption about 5700 p.p.m. On settling in cylinders, the suspended matter occupies 40, 30, and 22% of the original liquid volume after 1, 2, and 11 hrs., respectively; it is so light that it would cause trouble in settling tanks. After dilution of the slop with sewage the solids are easily separated. The sludge is readily digested with sewage solids and produces the same quality and quantity of gas as the organic matter of sewage. Its high oxygen requirements would greatly increase the amount of sprinkling-filter or activated-sludge treatment necessary. The cost of treating such a strong waste makes its recovery as a by-product in the industry itself advisable and necessary.

J. H. LANE.

Utilisation of distillery by-products. E. HUMBOLDT (Facts about Sugar, 1930, 25, 18—19).—In utilising the carbon dioxide from fermentation tanks, the impurities present can be separated by absorption by activated carbon, glycerin, or neutral paraffin oil, preferably the last-named as it retains the last traces of fusel oil and can easily be regenerated by heating. Yeast is not separated in modern distillery practice. The whole of the slop is concentrated to d 1.33—1.38 in a triple-effect evaporator, the syrup obtained being sprayed into a brisk fire in a furnace having a good draught, a product containing 35—45% K_2O being thus obtained. Or the slops may be destructively distilled for the recovery of the usual products, using a well-designed equipment requiring little auxiliary heat.

J. P. OGILVIE.

Titration of acids in wine. HIRSCH and RICHTER.
Determination of alcohol in foods. TÄUFEL and DÜN-

WALD.—See XIX. Alcohol determination. MEYER.—See XX.

See also A., Apr., 499, Refractometric measurement of invertase action (GORBACH). Effect of ethylene on activity of diastase and invertase (ENGLIS and ZANNIS). 500, Natural activators and inhibitors of proteolytic enzymes (GRASSMANN and others). 501, Higher alcohols of fermentation (SWENARTON). 502, Growth and acid production of *Aspergillus niger* under water (AMELUNG).

PATENTS.

Production of malt enzymes, diastases, or the like as by-products in brewing. E. JALOWETZ and M. HAMBURG (B.P. 303,110, 17.12.28. Austr., 28.12.27).—Malt is mashed with water under 35°, and after separation of the solution of enzymes and albuminous matter, the residue is used to produce beer wort by any of the known brewing methods. The enzymic solution is not added to the beer wort, but is concentrated as desired. C. RANKEN.

Enriching the flavour and improving the aroma of beer and the like. T. BREHENY (B.P. 326,587, 31.12.28).—Vapours from the contents of brewing and agitation vats are collected in the intermediate of three chambers of a condenser which consists of three concentrically arranged casings. Prior to incorporation in the finished beer, the condensed liquid is delivered to a chamber where it is cooled and divided into fine streams which absorb the cooled, non-condensable aroma gases. C. RANKEN.

Wheat syrup (B.P. 326,264).—See XVII.

XIX.—FOODS.

Action of aldehydes on white bread. L. KARÁCSONYI (Z. Unters. Lebensm., 1929, 58, 517—524).—The action of aldehydes in preventing bread from becoming stale has been further investigated (cf. Katz, B., 1916, 901) in order to discover which aldehydes produce the most pronounced effect, and also to determine which constituent of the bread is affected. The influence of the aldehydes was followed by means of viscosity determinations, 10 g. of bread crumb being pressed through a fine sieve and brought into suspension in water, the total liquid weighing 100 g. Measurements on 48-hr. old, treated and untreated bread when compared with those on new bread showed that acetaldehyde and propaldehyde have a far greater influence than any of the others tested. Propaldehyde was then used with starch and with a fat-free albumin preparation made from the bread, and it was found that whereas the aldehyde, after acting for 48 hrs., could be easily removed from the starch, it was firmly attached to the albumin. When treated with certain aldehydes, especially acetaldehyde and crotonaldehyde, bread exhibits a change of colour which is not influenced by light, and which persists when the bread is dried at 60° and finely ground. Starch alone gave no colour effect, and that the albumin constituent is the part affected was confirmed by tests on fat-free gluten. Crotonaldehyde and acetaldehyde affect the physical character of the bread, rendering it tough and springy; this fact may

explain the differences between the author's findings by the viscosity method and those of Katz (*loc. cit.*) by the sedimentation method. The retardation of staling by aldehydes is only effective so long as the concentration of aldehyde is maintained, for when the aldehyde was removed at the end of 48 hrs., in a further 48 hrs. the viscosity was found to be the same as that of bread 4 days old. H. J. DOWDEN.

Determination of lecithin-phosphoric acid in ice cream and its intermediates. A. GRONOVER and P. LEDERLE (Z. Unters. Lebensm., 1929, 58, 448—453).—In the determination of lecithin-phosphoric acid, dehydration by heating with sand or pumice leads to low values. More satisfactory results are obtained if the sample of ice cream (30—50 g.) or the intermediate (5 g.) be partially dried to a soft paste by heating with pumice (15—25 g.) on a water-bath, and then completely desiccated by adding anhydrous sodium sulphate. The dried material is ground and extracted with alcohol according to Arragon's method (cf. A., 1906, ii, 592), or, alternatively, Besson's extraction apparatus may be used as recommended by Grossfeld (cf. B., 1928, 313). After removal of the alcohol, the residue is ashed together with 3 c.c. of 50% magnesium acetate solution and the same volume of 8% alcoholic potash. Phosphoric acid is determined in the nitric acid extract; the authors found that Lorenz's method (cf. A., 1901, ii, 278) is very suitable. Instead of ashing, the destruction of organic matter can be conveniently effected by nitric and sulphuric acids. To the alcoholic extract 5 c.c. of concentrated sulphuric acid are added, followed by small quantities of nitric acid, heating gently meanwhile, and when most of the organic matter has been removed heating more strongly until the solution is colourless. Phosphomolybdate is precipitated in the solution after dilution and addition of pure nitric acid. H. J. DOWDEN.

Constituents of cacao husk. C. GRIEBEL and P. CASAL (Z. Unters. Lebensm., 1929, 58, 478—484).—The determination of the mucous cell content of cacao products by microscopical examination has been previously described (cf. Griebel, B., 1927, 568), and the constitution of these cells has now been investigated in an endeavour to devise a more reliable test. A filtered aqueous extract of the fat-free husk when treated with a large volume of alcohol-ether gave a precipitate which showed the reactions of galacturonic acid. The analysis calculated on the ash-free material was arabinose 14%, methylpentose 7%, galactose 32%, and galacturonic acid (expressed as lactone) 47%. Three methods of determination now present themselves: (i) precipitation of the mucous material from the aqueous extract and weighing; (ii) oxidation by means of 25% nitric acid of the residue after evaporating the aqueous extract, whereby mucic acid is formed; and (iii) distillation with 12% hydrochloric acid according to Lefèvre's method, whereby the carbon dioxide evolved is measured and used to calculate the galacturonic acid content. These methods were applied to the examination of the husks of 11 varieties of cacao. The first two methods were found to be inconclusive, duplicate experiments being not concordant. By the third method the "galacturonic acid number" (mg. of acid, expressed

as lactone, in 1 g. of fat-free, dry husk) was found to correspond closely with the number of scleridæ (cf. B., 1926, 605). The method is, however, not applicable to products which have been treated with alkali, since in products so treated part of the pectin material is rendered water-soluble and gives rise to large amounts of carbon dioxide.

H. J. DOWDEN.

Use of formaldehyde titration for the testing of foodstuffs [such as honey, cacao, etc.]. A. NIETHAMMER (Z. Unters. Lebensm., 1929, 58, 530—531).—It is shown that Tillmans' reaction (cf. B., 1927, 456) is most valuable in differentiating natural from artificial honey, the results being in agreement with those from Fiehe's test. The presence of pollen in honey has no appreciable influence on Tillmans' test, but it is observed that samples containing very large amounts of pollen require an abnormally large addition of alkali to restore the colour. Formaldehyde titration serves as a rough test for the presence of husk in cacao products, the alkali addition required to restore the colour being rather less for adulterated samples than for pure cacao.

H. J. DOWDEN.

Influence of neutral salts on the colorimetric determination of creatinine by Folin's method, and the determination of the degradation products of albumin by van Slyke's method in the presence of creatinine. K. and H. BECK (Z. Unters. Lebensm., 1929, 58, 409—425).—(i) To 15 c.c. of a 1.2% solution of picric acid were added 6 c.c. of 10% caustic soda solution and 10 c.c. of 0.1% solution of creatinine. After 5 min. the red mixture was diluted to 500 c.c. and its colour compared against 0.5*N*-potassium dichromate solution in a Duboseq colorimeter. Measured volumes of *M*-solutions of the sulphates, carbonates, chlorides, and phosphates of sodium, potassium, calcium, and barium were added to the mixture, and the variation in colour was measured in the colorimeter. The salts of the alkaline earths produce a greater reduction in colour than do those of the alkali metals, and in all cases the small initial amounts have relatively a much greater effect than the subsequent additions. Solutions of acid salts, e.g., sodium dihydrogen phosphate and potassium hydrogen sulphate, produced a colour inversion from red to yellow when a certain degree of acidity was reached. From the m.p. curves of mixtures of picric acid and creatinine it was deduced that over the range 0—20 mols. of creatinine in 100 mols. of picric acid only mixed crystals occurred; the molecular lowering of f.p. for picric acid is 14.2 and the molecular heat of fusion 5.02 kg.-cal. It is concluded that creatinine dissolves in the unimolecular state. Examination of the mixed melt by extraction with different solvents, fractional precipitation, etc. having lead to no definite conclusions, the red-coloured material was precipitated from aqueous solution by dilute sulphuric acid, freed from picric acid, and analysed. The resultant formula, $C_{10}H_{16}O_9N_6$, represents 1 mol. each of creatinine, picric acid, and water. The sodium derivative was then prepared in alcoholic solution by adding 200 c.c. of saturated creatinine solution to 100 c.c. of saturated picric acid solution mixed with 10 c.c. of 10% sodium ethoxide. After keeping in ice, the red precipitate was filtered off, dissolved in water, and the solution then acidified with sulphuric

acid, whereby the creatinine picrate was precipitated. The composition of the material obtained by these two methods agrees with the formula $C_4H_7ON_3 \cdot C_6H_2(NO_2)_3 \cdot OH \cdot H_2O$, and the change of colour from red to yellow is accounted for by keto-enol tautomerism (cf. Greenwald, B., 1925, 525). The gradual fading of colour and the action of neutral salts are both ascribed to alterations in the dissociation constant of the creatinine-picric acid complex and to displacement of the equilibrium, creatine \rightleftharpoons creatinine. (ii) In the examination of the hydrolysis products of meat extracts by van Slyke's method, it was found that creatinine is not completely precipitated by phosphomolybdic acid, and that when the filtrate residues are digested with lime decompositions occur in which the creatinine participates, rendering the results unsatisfactory. The nitrogen distribution in the hydrolysed extracts from beef, pork, and cod-fish are recorded, and are shown to vary with the degree of concentration.

H. J. DOWDEN.

Preservation of raspberry juice with hydrofluoric acid. A. HANAK (Z. Unters. Lebensm., 1929, 58, 453—478).—The expressed juice of raspberries, which is used largely for flavouring purposes, is frequently preserved by the addition of small quantities of hydrofluoric acid. Before the juice is used the acid is removed by precipitation with calcium carbonate. An extensive study has been made of this precipitation, especially as regards the influence on it of the mineral constituents of the juice, magnesium, iron, phosphoric acid, silica, etc. It was found that the hydrofluoric acid is never entirely removed. The best results were obtained when to 100 c.c. of juice were added 0.025 g. of hydrofluoric acid and as precipitant 3 times the equivalent of calcium carbonate was employed. After acting for 3 days the filtrate was found still to contain 22% of the original acid. The presence of free hydrofluoric acid in the treated juice manifests itself by the etching of glass vessels. Physiological considerations lead the author strongly to oppose this method of preservation.

H. J. DOWDEN.

Pectic juices in fruit jellies. F. MUTTELET (Ann. Falsif., 1930, 23, 79—81).—A method for differentiating between genuine, pure fruit jellies and jellies made with commercial pectin preparations is suggested, depending on the fact that the sorbitol content of the fruit jelly is considerably higher than that of the jelly made with pectin preparations. The sorbitol is determined by precipitation as dibenzylidenesorbitol, following the method of Vincent and Delachanal (A., 1898, 478).

E. B. HUGHES.

Use of indicators for the titration of the chief organic acids occurring in fruit and wine. P. HIRSCH and K. RICHTER (Z. Unters. Lebensm., 1929, 58, 433—448).—Formulae and equations have been developed from a consideration of the "molar combining power" (cf. A., 1924, ii, 795). The electro-metric results obtained by Auerbach and Smolezyk (cf. A., 1925, ii, 118; B., 1925, 153) have been used to calculate the values of this function for acetic, lactic, benzoic, tartaric, malic, succinic, and citric acids, and the results are represented graphically. By titration of the solutions to colour equality with buffer solutions of known p_H (e.g., 8.3, 5.02, and 3.2, using phenol-

phthalein, methyl-red, and dimethyl-yellow, respectively, as indicators) data were obtained from which the acid concentrations could be calculated. The influence of neutral salts on the dissociation of the acids was investigated by titration in the presence of saturated sodium chloride solution. Good results were obtained for a single acid in the presence of its salts and for mixtures of two acids, but the indicator method is not so accurate as the electrometric method. With mixtures of two acids the accuracy diminishes as the difference between the dissociation constants of the acids becomes less.

H. J. DOWDEN.

Determination of alcohol in foods by "salting out" with potassium carbonate. K. TÄUFEL and H. DÜNWALD (*Z. Unters. Lebensm.*, 1929, 58, 485—488).—The method of Nag and Lal (*cf. J.S.C.I.*, 1918, 290 τ) has been modified by Gadamer and Neuhoß (*cf. B.*, 1926, 214) by introducing a preliminary distillation, after which an excess of anhydrous potassium carbonate is added to the aqueous alcohol and the volume of the alcoholic layer is used in calculating the percentage of alcohol. The original formula has now been simplified and the alcohol content in g./100 c.c. is given by $(v \times 74 \cdot 28)/M$, where v is the volume of the supernatant so-called alcohol hydrate at 20°, and M the volume of the original liquid. The method has been used for the examination of materials of high alcohol content, such as brandy, and the results were found to agree to within 1% with those determined by pyknometric methods, even when the distillate was contaminated with acetic acid. The results with liquids containing less than 20% of alcohol were not satisfactory owing to the large volume of material required; thus with beer a sample of 100 c.c. must be taken in order to obtain an alcoholic layer of 5 c.c. The large amounts of potassium carbonate needed involve prolonged shaking; emulsions are formed and the rise in temperature causes loss of alcohol by evaporation.

H. J. DOWDEN.

Discoloration and corrosion of the inner surfaces of containers for canned foods. W. D. BOGATSKY, W. A. BIBER, and L. G. KISCHINEWSKAJA (*Z. Unters. Lebensm.*, 1929, 58, 506—517).—The black stains on the inner surfaces of tinned-iron containers have been investigated and were found to consist largely of metallic sulphide. By exposing containers for 1 hr. at 3 atm. pressure to the action of an emulsion of equal parts of 1% acetic acid and sunflower oil to which was added albumin, cystine, or allyl sulphide, it was shown that the discoloration is caused by the sulphur constituent of the can contents. Allyl sulphide in acetic acid also produced stains in the cold after 21 days, so that the high temperatures and pressures during processing are not responsible. A number of samples of tin-plate from different sources were compared as regards their liability to stain. The presence of lead or copper in the tin film does not appreciably affect the tendency to discolour, but the thickness and particularly the regularity of the deposit has considerable influence. The dark patches were found where the tin layer was thin and they were caused by mixtures of sulphides of tin and iron, the colour of the iron sulphide being visible through the thin layer of tin sulphide. Corrosion

of the inner surface of containers is caused by acid juices and involves dissolution of the tin coating. The resistance to corrosion of tin-plates of different origin was studied by determining the amount of tin passing into solution when they were exposed for 21 days to the action of 1% acetic acid. The amounts varied from 0.15 to 0.75 mg./cm.², but when an oil emulsion with 2% acetic acid was used the corrosion was much decreased and averaged 0.05 mg./cm.² Tin-plate which has been given a coating of tin sulphide appeared to be resistant to subsequent corrosion and staining by acetic acid or by sodium sulphide in acetic acid, respectively. Although the colour of the film of sulphide is diminished by the action of an emulsion of oil and acetic acid containing albumin or onions, no blackening occurred. The sulphide layer appears to be unaffected during the sterilisation of fish and vegetable preserves by heating at 100° for 1½ hrs.

H. J. DOWDEN.

Absorption of aluminium from aluminium utensils in the cooking of foods. SERGER and NEHRING (*Hansz. V. A. W. u. Erftwerk A.-G. f. Aluminium*, 1929, 1, [7]; *Bied. Zentr.*, 1930, 59, 125—127).—Numerous food preparations in aluminium and in enamelled vessels are compared. In no case was the aluminium absorbed by the foods sufficient to have any physiological significance. Repeated use of aluminium vessels does not influence the amount absorbed.

A. G. POLLARD.

Foil-wrapped food material. I. Zinc foil. L. T. FAIRHALL and L. C. WALKER (*Food Ind.*, 1929, 1, 642—645).—Ginger ale normally contained 0.5—2.3 pts. of zinc in 10⁶ pts., chocolate mints 16, chocolate 15.2—32.4, and acid drops (candy) 3.6 in 10⁶. Ginger ale readily dissolves zinc foil. In 2 weeks wrapped chocolates contained 4.6—65 pts. in 10⁶. The amount of zinc retained markedly increases with rise of temperature, but the effect on health of the zinc intake from such foods is negligible.

CHEMICAL ABSTRACTS.

Occurrence of antimony and tin in foil-wrapped cheeses. C. H. MANLEY (*Analyst*, 1930, 55, 191—192).—Samples of Gruyère cheeses wrapped in foil were found in every case to be discoloured, as was also the foil, and 15—160 pts. of tin per million were present. The foil consisted in one case of 96.8% Sn and 3.2% Sb, and the cheese in this wrapper contained 160 pts. of tin and 17 of antimony.

D. G. HEWER.

Sardine waste products. S. YOSHIMURA, K. NISHIDA, and A. YAMADA (*Bull. Kagoshima Agric. Coll.*, 1929, 7, 95—112).—Putrefaction results in decomposition of all the organic bases except cadaverine and putrescine. As a fertiliser the material is quickly effective.

CHEMICAL ABSTRACTS.

Bacteriostatic action of dyes. OWEN.—See IV. **Preservation of fats.** FIERO. **Re-esterification of neutral fats.** TÄUFEL and PREISS. **Saponification of fatty substances.** DE'CONNO and FINELLI. **Colorimetry of oils and fats.** PALLAUF. **Hardened fats in cacao butter.** GROSSFELD.—See XII. **Feeding value of mangolds.** HARWOOD and MARTIN.—See XVI.

See also A., Apr., 502, **Utilisation of micro-organisms for human food materials.** (TAKATA).

PATENTS.

Treatment of milk by ultra-violet light. J. O. and (MRS.) N. V. HICKMAN (B.P. 325,470, 12.9.28 and 12.2.29).—The irradiation of milk by ultra-violet light can be so regulated that the vitamin-D content is increased without having to protect against exposure to air, and with no effect on the taste, smell, or other characteristic properties, by exposing the milk, in a thin film and on one side of a milk cooler, at a distance of 11 in. from a 2000-c.p. quartz mercury-vapour lamp, with reflectors, for 8–30 sec. E. B. HUGHES.

[Manufacture of] cheese. T. VINK (Dutch P. 15,844, 12.8.25).—Addition of small quantities of alum to the curd used in the manufacture of cheese results in the production of a high-grade, homogeneous cheese. A. R. POWELL.

Extraction of caffeine from coffee beans. W. KLAPPROTH (B.P. 314,059, 4.6.29. Ger., 22.6.28).—Water (cold or at 65°) is circulated over raw coffee beans and the caffeine removed from the aqueous extract by means of a suitable solvent. This aqueous extract is repeatedly used for fresh charges of coffee, thus removing only caffeine. The extracted beans are then dried and roasted. E. B. HUGHES.

Production of coffee beans free from or poor in caffeine with an extract percentage corresponding to that of untreated coffee beans. G. and I. NEUSTADT (B.P. 314,308, 24.6.29. Ger., 22.6.28).—Coffee is extracted by water at 60–90°, the aqueous extract being decaffeinated and then returned to the wet coffee. In order to bring the coffee to its original content of extractive matter additional caffeine-free extract, similarly obtained from about one tenth to one fifth the quantity of beans used, is also added. After soaking at 60–90° for 3 hrs. the beans are drained, washed, and dried. E. B. HUGHES.

Soluble coffee. E. KLEIN (U.S.P. 1,742,261, 7.1.30. Appl., 1.7.27).—Raw coffee is soaked, allowed to germinate for 3–5 days to render the proteins soluble, dried, and roasted. The product after grinding may then be rendered soluble by treatment in the cold with dilute nitric acid (11% wt./vol.) and an alkaloid (quinine), and subsequent neutralisation by caustic soda and ultimately drying. E. B. HUGHES.

Preservation of green peas by canning. G. D'ONOFRIO (B.P. 325,622, 8.12.28).—Before canning, the peas are boiled in a dilute citric acid solution of a green dye (e.g., Guinea Green B) and a yellow dye (e.g., Naphthol Yellow S extra). The colour may be fixed with aluminium chloride. The peas are then washed and canned in a salt solution rendered acid with citric acid and autoclaved at 115°. Varnished cans must be used to prevent reduction of the dyes by the metal of the container. E. B. HUGHES.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Philippine camphor. A. P. WEST and H. TAGUIBAO (Philippine J. Sci., 1930, 41, 103–118).—Camphor in young Philippine trees is located almost entirely in the leaves (2–7% of crystal camphor in moisture- and oil-free leaves). About 1% (on the wt. of crystal camphor)

of camphor oil ($\alpha_D^{30} +26.01^\circ$) and a small quantity of volatile oil (0.22% on dry leaves) are also obtained on distillation. Leaves of some trees gave no crystal camphor, but only camphor oil (0.77–7.0% on dry leaves) having $d_{30}^{20} 0.8858$, $n_D^{20} 1.4652$, and $\alpha_D^{30} -19.2^\circ$. E. H. SHARPLES.

Solution of arsenious and mercuric iodide. M. G. ACTON, JUN. (Amer. J. Pharm., 1930, 102, 159–164).—In the U.S.P. X method for the assay of Donovan's solution, after reduction with formaldehyde the solution should be filtered and not decanted, to prevent loss of mercury. An examination of the keeping properties and stability of the solution showed that the solution deteriorates rapidly irrespective of the conditions of storage; during a period of 3 months oxidation of 60% of the arsenious iodide was noted. During oxidation there is an increase in acidity due to the production of arsenic acid. In a sample at least two years old free iodine was detected, and only about 5% of the original arsenious iodide content remained in the arsenious condition. E. H. SHARPLES.

Increasing the yield in alcoholic preparations [of drugs]. F. GRAF (Pharm. Ztg., 1930, 75, 275–276).—The problems occurring in the alcoholic extraction of drugs and the control of pressing and extracting processes by density determinations are discussed. E. H. SHARPLES.

Determination of ether and alcohol in Spiritus æthereus and Tinctura valerianæ ætheræ. F. WRATSCHKO (Pharm. Ztg., 1930, 75, 319).—An amplification of the earlier account of the author's light petroleum method (Pharm. Presse, 1928, 384). S. I. LEVY.

Alcohol determination and the alcohol value in Tinctura iodi, D.A.B.VI. W. MEYER (Pharm. Ztg., 1930, 75, 273–275).—A mixture of 10 g. of iodine tincture and 5 g. of a cold solution of sodium thiosulphate (1:2) is shaken until colourless and after about 15 sec. 5 g. of 15% potassium hydroxide are added. The liquid is then distilled according to the official method and the alcohol content is determined in the first 13 c.c. of distillate by the addition of potassium carbonate. The alcohol figure should not be below 10.1%. By this method no side-reactions occur, the distillate is not cloudy, acid, nor decomposed, and the residual liquid contains no free sulphur, sulphurous acid, hydriodic acid, iodoform, nor methylene iodide. The causes and prevention of the formation of these by-products in the usual methods for the determination of alcohol are discussed. E. H. SHARPLES.

Official tests for resins of jalap, podophyllum, and scammony. D. B. DOTT (Pharm. J., 1930, 124, 213–214).—The residue, after stirring 1 g. of powdered jalap resin with 30 c.c. of ether, decanting after 15 min., extracting with a further 20 c.c. of ether in successive quantities, and evaporating the extracts, should not exceed 0.05 g. The following test with jalap resin is suggested: 1 g. of the powder is boiled with 30 c.c. of carbon tetrachloride for 30 min., cooled, filtered, and washed with 15 c.c. of solvent. The extracts on evaporation should yield about 3% of the powder used. In the ammonia-solubility test for podophyllum resin it is

necessary to fix quantities and times. 0.5 G. of the powdered resin is stirred with 30 c.c. of the ammonia solution and, after 30 min., is filtered through a tared filter, using 30 c.c. of water for transferring the solid and washing. The dried residue should not (for *Peltatum* resin) exceed 0.55 g. [95.5%]. *Emodi* resins by this test gave 40–48% of insoluble matter. By the ether test, using 0.5 g. with 30 c.c. of ether, *Peltatum* gave 66–77% of extract, *Emodi* 55–59%. Notes on the coagulation test to distinguish the above two varieties of resin are given. In the examination of scammony resins, 85.4% was the lowest figure observed in the ether test using 30 or 40 c.c. of ether with 1 g. of resin as indicated in the jalap resin test. Similarly, the boiling carbon tetrachloride test gave 52.8–60.9% of extract for four samples.

E. H. SHARPLES.

Quinoline-hydrogen peroxide reagent: a distinguishing colour test for aloes. E. J. SCHORN (Pharm. J., 1930, 124, 212–213).—An anhydrous solution of hydrogen peroxide is prepared by extracting a 30% solution with quinoline and drying the extract. This reagent, diluted to 1% concentration, is standardised by dissolving in dilute sulphuric acid and determining the liberated iodine after the addition of potassium iodide. On warming a few mg. of finely-powdered aloes with a small amount of the reagent at 60° for 3–5 min. a colour is produced which is characteristic for each variety, e.g., *Aloe climensis* and *A. vulgaris* give a purple colour which becomes rose-pink on acidification with 1% sulphuric acid; *A. ferox* and *A. spicata* give a greenish colour changing to greenish-yellow on acidification; *A. Perryi* gives a brown colour and remains brown on acidification. All the species of aloes examined, when treated with dilute ferrous sulphate solution followed by addition of hydrogen peroxide, gave a dark colour which became rose-pink when acidified.

E. H. SHARPLES.

Assay of mixtures of cinchona alkaloids. J. A. GOODSON and T. A. HENRY (Pharm. J., 1930, 124, 351–352).—The application of the methoxyl determination in the assay of cinchona alkaloids is illustrated by the examination of the alkaloids from 3 kg. of a hybrid bark. The total alkaloids were fractionated by the Howard and Chick method and from the methoxyl determination of the two fractions, viz., crude quinine sulphate and crude cinchonidine tartrate, the relative percentage proportions of the corresponding alkaloids were calculated. Analysis of a number of Tanganyika barks are given, also notes on “cinchona febrifuge.”

E. H. SHARPLES.

Isomeride of yohimbine. R. LILLIG (Mercks Jahresber., 1929, 42, 20–26; Chem. Zentr., 1929, ii, 2465).—Commercial yohimbine contains an isomeride, α -yohimbine, m.p. 246° (decomp.), $[\alpha]_D^{25}$ –25.05° in alcohol (hydrochloride, m.p. 286° (decomp.), $[\alpha]_D^{20}$ +58.3° in water; nitrate, decomp. 276°; picrate, decomp. 213°). α -Yohimboic acid, $C_{20}H_{24}O_3N_2$, has m.p. 280°, $[\alpha]_D^{20}$ +56.9° in pyridine.

A. A. ELDRIDGE.

[Isomeride of yohimbine.] H. KREITMAIR (Mercks Jahresber., 1929, 42, 20–26; Chem. Zentr., 1929, ii, 2465).— α -Yohimbine (cf. preceding abstract) has

practically the same physiological action as yohimbine, but disturbs respiration much less markedly.

A. A. ELDRIDGE.

Testing of “nicotine-free,” “de-nicotinised,” and “harmless” commercial tobacco products. K. BAUMANN and J. KUHLMANN (Z. Unters. Lebensm., 1929, 58, 524–529).—Commercial tobacco products described as “nicotine-free” etc. have been found to contain almost as much nicotine as ordinary tobacco. The flavour of tobacco, cigars, and cigarettes designated as “strong,” “medium,” “mild,” etc. is closely associated with the nicotine content, the “stronger” the tobacco the higher is the nicotine content, but the classifications by different manufacturers are by no means uniform. An analysis has been made, by the method of Pfyl and Schmitt (cf. B., 1927, 955), of numerous commercial brands of tobacco described as “free from nicotine,” “de-nicotinised,” etc., and the results revealed that whilst none of the samples contained less than 0.75% of nicotine, about one third of them contained 0.76–1.00%, one third more 1.01–1.25%, and the remainder contained up to 1.75% of nicotine. It is emphasised that statutory definition of the nicotine limits for various grades of tobacco is urgently needed.

H. J. DOWDEN.

Determination of nicotine in tobacco and tobacco smoke. K. BRAUER (Pharm. Zentr., 1930, 71, 209–213).—Results of long experience of the methods described in the literature are given; those of Pfyl and Schmitt (B., 1927, 955), with some modifications, are found reliable. Attempts to prepare nicotine-free tobaccos are criticised, and it is concluded that whilst chemical methods may give acceptable products, the finest tobaccos cannot be de-nicotinised without affecting the aroma.

S. I. LEVY.

Essential oils. ANON. (Bull. Imp. Inst., 1930, 28, 8–27).—**Lavender oil from S. Africa.**—Steam-distillation of the flowers of *Lavandula vera* grown on an experimental plantation yielded 0.70% (on fresh wt.) of an almost colourless oil having d_{15}^{20} 0.8901; α_D –7.25°; n_D^{20} 1.463; acid value 0.7; esters (as linalyl acetate) 49%; solubility in 70% alcohol at 20°, insol. in 10 vols., in 80% alcohol at 20°, 1 in 0.9 vol. The oil compares favourably with the French lavender oil. **Lavender oil from Cyprus.**—Two samples of oil, one from *L. vera* growing at 442 ft. above sea-level and the other growing at 3500 ft., had, respectively: d_{15}^{20} 0.8959, 0.8976; α_D –0.35°, –0.50°; n_D^{20} 1.4655, 1.466; acid value 0.6, 0.3; ester value 22.0, 17.8; ester value after acetylation 165.1, 150.9; esters (as linalyl acetate) 7.7%, 6.2%; solubility in 70% alcohol at 20°, both soluble in 2.3 vols. The marked odour of cineole rendered them inferior in aroma to English and French lavender oils. **Peppermint oil.**—Two samples of peppermint oil from plants grown in Cyprus had the following respective constants: d_{15}^{20} 0.961, 0.928; α_D +14.5°, –10.9°; n_D^{20} 1.482, 1.468; ester value after acetylation 132.5, 140.0; menthol 41%, 43.5%. **Oil of pennyroyal from South Africa.**—Distillation of the fresh pennyroyal plant grown in Cape Province yielded 0.47% of a clear, pale yellow oil having d_{15}^{20} 0.9378, α_D +20.7°, n_D^{20} 1.4855, ketones (as pulegone) 93%.

solubility in 70% alcohol at 15° 1 in 1.8 vols. *Palmarosa oil (Rosha) from India*.—A sample of palmarosa oil prepared by steam-distillation of plantation grass had d_{15}^{20} 0.8861, α_D^{20} +0.35°, n_D^{20} 1.4745, acid value 1.3, ester value 11.6, ester value after acetylation 266.5, total geraniol 91.6%, and solubility in 70% alcohol at 15° 1 in 1.8 vols. *Cuscuta (vetiver) roots from Travancore*.—Steam-distillation of ground vetiver roots (moisture 5%) yielded 1.6% of a dark, reddish-brown, viscous oil having d_{15}^{20} 1.0377, n_D^{20} 1.525, acid value 74.7, ester value 15.0, ester value after acetylation 108.3. *Anise-scented bay oil from Dominica*.—The clear, reddish-brown oil distilled from the "Bois d'Inde Anise" grown in Dominica had d_{15}^{20} 0.9968, n_D^{20} 1.527, phenols (by absorption) 10%, methoxyl 17.9%, and consisted chiefly of myrcene, estragol (about 15%), and methyleugenol (13%). It contained only a small quantity of eugenol. *Pimenta acris* leaves (moisture 18.9%) from Egypt on steam-distillation yielded 3.06% of oil having d_{15}^{20} 0.9956, α_D -1.82°, n_D^{20} 1.522, and phenols 70%. The aroma was good and the quality comparable with that of West Indian bay oil. *Cumin oil from Cyprus*.—A pale, yellowish-brown sample of cumin oil from Cyprus had d_{15}^{20} 0.9443, α_D +3.15°, n_D^{20} 1.5125, cuminaldehyde 40%, and solubility in 80% alcohol at 15° 1 in 1.2 vols.

E. H. SHARPLES.

Essential oils. A. M. BURGER (Riechstoffind., 1929, 4, 121; Chem. Zentr., 1929, ii, 2517—2518).—Huo pine oil has d_{20}^{20} 1.034, α_D^{20} +0.6°, n_D^{20} 1.535; the chief fraction (90%, b.p. 133—135°) is eugenol methyl ether. Massoia bark oil has d_{15}^{20} 1.075, and contains eugenol (62%), safrole, and hydrocarbons. A. A. ELDRIDGE.

Determination of ascaridole in chenopodium oil. T. T. COCKING and F. C. HYMAS (Analyst, 1930, 55, 180—186).—About 2.5 g. of the oil are dissolved in sufficient 90% acetic acid to produce 50 c.c. of solution, and 5 c.c. of the solution are added to a mixture of 3 c.c. of 5*N*-potassium iodide, 5 c.c. of concentrated hydrochloric acid, and 10 c.c. of glacial acetic acid cooled to -3°. After mixing, the stoppered tube is left for 5 min. and the liquid titrated with 0.1*N*-thiosulphate. Each c.c. of thiosulphate is equivalent to 0.00665 g. of ascaridole. A blank test should be made; the experimental error should not exceed $\pm 1\%$. Dilution of the reaction mixture before titration gave low results. The small liberation of iodine by non-ascaridole constituents of essential oils may be safely ignored. D. G. HEWER.

See also A., Apr., 469, **Brucine and strychnine salts of inositolphosphoric acids** (SELIGSON). 486, **Compounds of phenols with quinine and cinchonine** (TOMCSIK). **Microchemical reactions of aconitine** (WAGENAAR). **Potentiometric determination of alkaloids** (MARICQ). **Arsenic derivatives of pyridine** (BINZ and others). 498, **Toxicity of lead compounds** (BUCK and KUMRO). 503, **Disinfecting properties of chloropicrin** (SMORODINCEV). 504, **Assay of thyroid gland** (HARRINGTON and RANDALL). 505, **Determination of iodine in thyroid gland** (MIDDLETON). **Follicular hormone: its preparation and determination** (FRATTINI and MAINO). 506, **Determination of antineuritic vitamin-B** (EVANS and LEPKOVSKY). **Determination of vitamin-D**

(HELLER and CASKEY). 507, **Ethereal oil and crystalline ester from heart wood of *Vouacapoua americana*** (SPOELSTRA). 508, **Pyrethrin content of pyrethrum flowers** (GNADINGER and CORL). **Occurrence of *d*-norisoephedrine in *Catha edulis*** (WOLFES).

PATENTS.

Manufacture of [therapeutically active] basic nitro-derivatives of 9[ms]-aminoacridine. I. G. FARBENIND. A.-G. (B.P. 304,280, 4.1.29. Ger., 18.1.28. Addn. to B.P. 283,510; B., 1929, 577).—A nitrated 10-chloro- or 10-alkoxy- (or aryloxy)-acridine is condensed with an amino-acid, -ester, or -nitrile, and the nitroacridylamino-acid is converted into its (β -diethylaminoethyl)amide or other basic amide. 10-Chloro-3-nitro-8-ethoxyacridine condenses with glycine to give 3-nitro-8-ethoxy-10-acridylaminoacetic acid, m.p. 274—275° (decomp.); the corresponding acid chloride hydrochloride, m.p. 217—218° (decomp.), is converted into the β -diethylaminoethylamide, m.p. 153° [$+2H_2O$; dihydrochloride, m.p. 233° (decomp.)], the γ -diethylamino- β -hydroxy-*n*-propylamide, m.p. 143° (decomp.), the *N*-methyl-*N*-(β -diethylaminoethyl)amide, m.p. 120° (dihydrochloride, m.p. 123—124°), the bis-(β -diethylaminoethyl)amide [trihydrochloride; m.p. 229° ($+4H_2O$, decomp.)], and the β -dimethylaminoethylamide, m.p. 175° (decomp.) [dihydrochloride, m.p. 198° (decomp.)]. 3-Nitro-10-phenoxyacridine, m.p. 195—186°, with glycine gives 3-nitro-10-acridylaminoacetic acid, m.p. 218—219° [hydrochloride, m.p. 235° (decomp.)]; acid chloride hydrochloride; β -diethylaminoethylamide m.p. 177° (decomp.) (dihydrochloride, m.p. 220°). The new diamine intermediates described are: ethyl-(β -diethylaminoethyl)amine, b.p. 154—156°; bis-(β -diethylaminoethyl)amine, b.p. 76°/2 mm.; and β -dimethylaminoethylamine, b.p. 106—107°. C. HOLLINS.

Manufacture of synthetic menthol. HOWARDS & SONS, LTD., J. W. BLAGDEN, and W. E. HUGGETT (B.P. 325,509, 19.11.28).—Crude menthol (e.g., from hydrogenation of thymol, menthone, etc.) is converted by treating with phthalic anhydride into hydrogen phthalates which are cooled to 80° and crystallised by addition of light petroleum. C. HOLLINS.

***N*-Hydroxyethyl derivatives** (B.P. 300,503).—See III. **Caffeine from coffee beans** (B.P. 314,059 and 314,308).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Influence of the sensitisation of photographic plates on their spectral sensitivity and gradation. A. F. BÜRKI and V. R. PFRUNDER (Helv. Chim. Acta, 1930, 13, 237—267).—The ordinary methods of sensitometry have been compared with one another and the sources of error investigated. A source of light similar to daylight has been obtained by the use of a 3.5-volt filament lamp and a filter of ammoniacal copper sulphate solution. With the aid of this lamp the sensitivity of different photographic emulsions has been measured both by the Scheiner sensitometer and the Eder-Hecht wedge sensitometer, and the results are compared with one another and with the commercial data. The sensitising

influence of a number of substances on different emulsions has been examined. Dyes such as erythrosin, pinachrome, pinachrome-violet, and chlorochrome increase the total sensitivity. The greatest increase is observed in the yellow, and emulsions originally only sensitive to blue become sensitive to green, yellow, and red at the expense of the blue. An aqueous dye solution may act as a sensitizer, whilst the alcoholic solution may be inactive. Dyes belonging to the safranine and eosin group have no effect. Dilute hyposulphite solutions also slightly increase the sensitivity. Pinacryptol-green is a powerful desensitizer. Experiments have been made in hypersensitisation with ammonia, and in ultrasensitisation with ammoniacal silver chloride solution, of various kinds of plates. The sensitivity for all wave-lengths appears to be increased without increasing the time of fixing. By a preliminary illumination with a faint green light of the plate to be ultrasensitized the sensitivity is still further increased, but the stability of the emulsion is not very great, the sensitivity being reduced with time. A method for determining gradation for any wave-length, and dispensing with the use of filters, has been devised. A number of gradation curves have been obtained for different plates sensitized by different methods, and the influence of different methods of sensitisation on the blackening curves has also been examined. Curves showing change of gradation with time of illumination for a constant intensity of light, and change of gradation with intensity of light for equal times of illumination, have been obtained for different coloured light. The intensity curves are found to be steeper than the time curves. The absorption spectrum of the new sensitizer chlorochrome has been determined, and its maximum absorption has been found to correspond to maximum sensitisation.

M. S. BURR.

See also A., Apr., 396, **Sensitising photographic plates** (LUEG).

PATENTS.

Manufacture of a dye for colouring photographic prints. M. MICHELS (G.P. 458,989, 7.8.26).—Finely-divided starch loosely coloured with a basic dye is made into a paste with water and hydrochloric acid, or substances which produce this acid are added. The product serves to colour copper-toned papers. A. R. POWELL.

Production of pictures consisting of dyes in photographic manner. F. LIERG (B.P. 298,979, 20.8.28. Austr., 18.10.27).—Dye components in insoluble form are added to the silver emulsion; after exposure the silver image is first developed and the coloured image is then produced by suitable treatment, e.g., by diazotisation or oxidation. Thus benzidine urate or resinate with a metal salt of β -naphthol, or benzidine phosphotungstate with the aluminium salt of H-acid, benzidine abietate with ethyl salicylate, or ethyl anthranilate with aluminium naphthoxide, are added separately as fine dispersions or solutions in alcohol to the sensitising bath. After exposure and hardening, the print is immersed in nitrous acid, which penetrates only the unhardened parts, forming a reversed colour image. Insoluble salts of aminodiphenylamine and *p*-toluidine may be oxidised to give a red image. The nitrite for azo dye development may

be added to the emulsion in the form of insoluble cobaltammine salts etc.

C. HOLLINS.

Production of light-sensitive layers by means of diazo compounds. KALLE & Co. A.-G. (B.P. 302,184, 10.12.28. Ger., 10.12.27).—Paper etc. is impregnated with diazo compounds of aminohydroxynaphthoic acids, e.g., 1-amino-2-hydroxy-3-naphthoic acid, in acid, neutral, or alkaline solution; if an acid solution is used the paper must be treated with ammonia before exposure. Washing with water develops a bluish-black positive (after exposure under a negative) which may be toned with a solution of a copper salt.

C. HOLLINS.

Production of photographic plates, films, or papers for photography in natural colours. W. R. B. LARSEN (B.P. 316,277, 8.11.28. Denm., 28.7.28).—The colour screen is prepared from a mixture of red-, green-, and violet-dyed gelatin particles which have been partially decolorised by a chemical treatment in such a way that the full colour may be later restored; e.g., if aniline-red, acid-green, and crystal-violet be the dyes used, the colour may be weakened by treatment with an alkali and subsequently restored by an acid. At the same time a red sensitizer, e.g., pinacyanol, and a green sensitizer, e.g., erythrosin, are incorporated in the red and green screen particles, respectively. The silver halide emulsion is coated upon the screen prepared from an even layer of these particles upon a suitable base, and by diffusion of the dyes into the emulsion during the drying process becomes sensitized to red and green light under the red and green particles. Soluble bromides or alkali salts may also be incorporated in the screen particles to exert a control, by simultaneous diffusion with the dyes, on the sensitivity of the emulsion in the region of the filter. A slow-acting hardening agent is used in the preparation of the screen particles in order that the diffusion of the sensitizing dyes may take place before the hardening is complete. After exposure, development, and fixation the tint of the screen dyes is adjusted to the required intensity.

J. W. GLASSETT.

Decoration of fabrics by the action of light and the reduction of silver salts. M. MICHELS (G.P. 460,309, 13.5.27. Addn. to G.P. 441,150; B., 1927, 861).—The silver image produced on the material in the manner described in the prior patent is treated with lead ferricyanide, which converts the silver into a mixture of lead and silver ferrocyanides, the material is then dipped into cupric chloride solution to produce copper ferrocyanide, and the silver chloride is removed by means of thiosulphate. The image can then be coloured with any suitable basic dye.

A. R. POWELL.

[Production of positives in] colour [for kinematographic or still] photography. W. CHAPMAN (B.P. 327,200, 3.1.29).

Cellulose films (B.P. 301,878).—See V.

XXII.—EXPLOSIVES; MATCHES.

Propagation of detonation across a gas-gap between two cartridges of explosive. G. ST. J. PERROTT and D. B. GAWTHROP (J. Franklin Inst., 1929, 208, 643—660; cf. B., 1927, 350).—The method used was mainly that previously described. The initiating cartridge, 17.8 cm. long, was placed either in a paper

tube (3.2 cm. diam.) or in one of Shelby steel, 3.5 cm. internal diam. and 0.6 cm. thick. The gap was filled with air, hydrogen, carbon dioxide, or helium. The explosives used were (i) "40% straight dynamites," and (ii) "permitted" ammonium nitrate compositions. In paper tubes, with initiating and receiving cartridges of the same explosive, detonation of the latter always occurred in hydrogen, air, and carbon dioxide with (i) at distances up to 137, 107, and 61 cm. respectively, and with (ii) up to 25, 51, and 30 cm. It failed to occur with (i) at over 203, 178, and 122 cm., or with (ii) at 38, 64, and 46 cm. Detonation of (ii) by (i) occurred at distances up to 107, 76, and 61 cm. Corresponding distances in the steel tube were about 10 times as great. The velocity of transmission of the disturbance across the gap is greatest in gases of low density, and with hydrogen is at first greater than that of detonation, decreasing less in the steel than in the paper tube. It is concluded that there is for each explosive a critical velocity of the shock-wave above which this detonates the receiving cartridge, and below which detonation may be effected by a later disturbance—perhaps by the gaseous products of the explosion.

C. A. SILBERRAD.

See also A., Apr., 438, **New phosphonium salt** (EVRARD).

PATENTS.

Acceleration of the precipitation of nitroglycerin.

CARBONIT A.-G., Assees. of G. VON LÖBBECKE (G.P. 458,520, 30.7.28).—The glycerin or the acid mixture is treated with acetamide, dicyanodiamide, or carbamide, or with other organic compounds which during the nitration are decomposed completely with the evolution of indifferent gases.

A. R. POWELL.

Manufacture of self-combustible mixtures. (A)

MINIMAX A.-G., and (B) DEUTS. PYROTECHN. FABR., Assees. (B) of J. THIECKE and H. HERZOG (B.P. 299,396, 24.10.28. Ger., 25.10.27. Cf. B.P. 314,604 and 315,232; B., 1929, 706, 769).—A combustible mixture for the generation of heat, pressure gas, or nitrogen consists of finely-divided animal charcoal and ammonium nitrate, mixed preferably in the proportion of about 15 pts. of the former to 85 pts. of the latter. Other kinds of charcoal may be added to modify the rate of combustion. The mixture is compressed into solid bodies which are given a waterproof cover.

A. B. MANNING.

XXIII.—SANITATION; WATER PURIFICATION.

Chlorinated copperas in the treatment of soft, highly coloured water. A. C. DECKER (J. Amer. Water Works' Assoc., 1930, 22, 218—221).—Preliminary experimental work with a highly coloured creek water which it was desired to render potable showed that whilst good colour removal could be obtained by using 5.5 grains of alum and 3.0 grains of lime per gall., the substitution of ferrous sulphate for alum seemed to set the colour. If the ferrous sulphate were chlorinated, excellent results were obtained in conjunction with either lime or sodium aluminate. On the large scale satisfactory results were obtained, using 0.7—0.8 grain of ferrous sulphate per gall. oxidised with 0.11—0.126 grain of chlorine per gall. and succeeded by 0.33—0.4

grain of sodium aluminate or 0.55 grain of lime per gall. No difference in result was observable whether the plant was operated continuously or not, and it is estimated that the use of chlorinated copperas results in a saving of \$10 per million gallons. C. JERSON.

Peculiar lactose-fermenting anaerobe from filtered and chlorinated water. R. S. SPRAY and P. C. LAUX (J. Amer. Water Works' Assoc., 1930, 22, 235—241).—Positive "presumptive" tests in the drinking water of Williamson, W. Va., which had been previously chlorinated so as to have a residuum of 0.4 p.p.m. as indicated by the o-tolidine test, were shown to be due to the presence of a slender, Gram-negative bacillus of about 0.6 by 6—8 μ . The organism has not yet been recognised, and its significance in the water supply as well as its source is undetermined. It is apparently harmless, but causes inconvenience by producing numerous false "presumptive" tests. C. JERSON.

Rapid determination of the colon group [in water]. M. LEVINE (J. Amer. Water Works' Assoc., 1930, 22, 234).—The standard "presumptive" and "partially confirmed" *B. coli* tests may be obtained simultaneously in 24 hrs. if 0.1 c.c. of the preliminary enrichment broth (after about 10 hrs.' incubation at 37°) be smeared over the surface of an eosin-methylene-blue agar plate and both plate and tube incubated at 37° for the remainder of the 24 hrs. Those tubes which show gas (24 hrs.' presumptive test) should show colonies of the colon-aerogenes group (partially confirmed test) on the corresponding plate. C. JERSON.

Disposal of sugar-factory waste waters. MONTGOMERY.—See XVII. **Beer-slop waste.** HATFIELD.—See XVIII.

See also A., Apr., 434, **Routine preparation of conductivity water.** (STUART and WORMWELL). 443, **Determination of alkalis in waters** (MALJAROV). 477, **Rotenone and insect poisons** (BUTENANDT and HILDEBRANDT). 503, **Disinfecting properties of chloropicrin** (SMORODINZEV).

PATENTS.

Fungicides, disinfectants, and the like. NEWTON CHAMBERS & Co., LTD., and M. W. BLYTH (B.P. 326,460, 6.9.28).—An aqueous suspension of a neutral inorganic compound, e.g., lithopone or barium sulphate, stabilised by means of an organic colloid, is used as a vehicle for a phenolic disinfectant or fungicide, e.g., Izal or Lysol. The preparation may be coloured.

F. G. CLARKE.

Purification of boiler-feed water. P. MÜLLER GES.M.B.H. (Austr.P. 109,459, 2.5.21).—The water is passed through a softener which replaces the calcium and magnesium salts by sodium salts. When the water in the boiler contains a high proportion of these salts the pressure is reduced so as to evaporate as much more of the water as possible; the resulting steam is used for preheating fresh quantities of purified feed water, and the hot concentrated salt solution for preheating water passing to the purification plant.

A. R. POWELL.

Filtration of water (B.P. 325,937). **Air washing** (B.P. 325,884). **Air filter** (B.P. 326,047).—See I. **Purification of waste liquors** (B.P. 326,112).—See II.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 6 and 13, 1930.*

I.—GENERAL; PLANT, MACHINERY.

Trend in design and operation of industrial plants, with special reference to furnace volume.

H. KREISINGER (Proc. Eng. Soc. W.Pa., 1929, 45, 426—440).—A definite volume of combustion space is necessary in all furnaces to allow of almost complete combustion before the furnace gases enter the boiler. Any means to increase the rate of contact between the fuel and oxygen would permit the combustion space to be decreased, but limiting factors occur as, *e.g.*, the low heat value of blast-furnace gas and also when increased turbulence causes fuel ash to erode the combustion walls excessively. In water-cooled furnaces the limits are the accumulation of slag on the boiler tubes and the losses due to incomplete combustion. The rate of heat liberation in B.Th.U. per cub. ft. of combustion space per hr. for normal range of operation is given for various fuels: blast-furnace gas 7000—15,000, natural gas and fuel oil 10,000—30,000, powdered coal (low-fusion ash) 6000—14,000.

C. A. KING.

Pneumatic transport of granular and powdered materials. H. NEU (Bull. Soc. Ing. Civ. France, 1930, 82, 987—1041).—A pneumatic method of transporting materials requires a pressure difference of 100—450 mm. Hg and an air supply of 30—500 litres/kg. of material, according to circumstances. The calculation of an installation, which cannot be performed by theory alone, permits of two solutions: the minimum capacity and the most economical capacity. Various methods of feed and discharge are described together with types of pumps for the production of pressure or vacuum. Pneumatic transporters can deliver up to 300 tons per hr.; they are flexible and economical in labour, but they have a higher power consumption than other types. They are suitable for grain of all kinds, coal dust, cement, ammonium sulphate, etc. Portable transporters for discharging ships are described. Analysed costs of five transporters handling coal and grain are given. This method of handling has the convenience of taking up little room, and the crossing of obstacles such as railway lines presents no difficulty.

C. IRWIN.

High-pressure chemical engineering equipment of the Chemical Research Laboratory, Teddington.

H. TONGUE (Inst. Chem. Eng., Apr., 1930. Advance proof. 14 pp.).—A description of the apparatus at this laboratory, which includes low-pressure gas-storage, compression plant, a high-pressure gas-storage and distribution system, high-pressure autoclaves, and other apparatus for catalytic reactions.

C. A. KING.

Humidity chart for use with combustion gas [dryers]. S. HATTA (Chem. Met. Eng., 1930, 37,

165—166).—Humidity charts for air dryers cannot be used without error when the actual drying agent is flue gas. The author's chart is constructed on the assumption that the sp. heats of oxygen, nitrogen, and carbon monoxide are substantially equal. Humidity is expressed in lb.-mols. of steam per lb.-mol. of dry gas, and separate cooling lines are given for 0, 10, and 20% CO₂ content. In addition to the usual lines, a series of humid-volume lines are given for varying humidities. An example of the use of the chart is given.

C. IRWIN.

Use of thermal data in drying-tower design.

A. B. NEWMAN (Chem. Met. Eng., 1930, 37, 145—147).—The case is considered of a sulphuric acid drying tower without cooling coils, in which the whole of the heat of dilution of the acid is carried away in the effluent. Curves are given showing the variation of the sp. heat of sulphuric acid with concentration and the heat of dilution at various concentrations. By their aid the temperature of the effluent acid can be calculated, and from the water-vapour pressure of sulphuric acid solutions the necessary feed in any particular case can be deduced.

C. IRWIN.

Water-softening. JENKS.—See XXIII.

PATENTS.

[Open-hearth] furnace. M. C. STEESE (U.S.P. 1,737,392, 26.11.29. Appl., 13.12.20).—At each end of the furnace is a single uptake, used (at the firing end) for preheated air alone. Below an ordinary air port (leading to the furnace) is another containing a burner through which fluid fuel is injected under pressure. A preliminary combustion takes place in the port and is completed in the furnace with air from the upper port. The burner induces air proportional to the fuel supply, and the amount of air passing on to the upper port is determined by the total air allowed to enter the regenerator; the valve for this may be regulated (through electrical relays) by the pressure existing in the uptake.

B. M. VENABLES.

Pulverised-fuel furnaces. ASH CO. (LONDON), LTD. (B.P. 311,815, 26.4.29. U.S., 17.5.28).—The combustion space has its walls water-cooled to such an extent that a substantial amount of heat is extracted from the ash without causing it to solidify. The molten ash drops to a hopper-shaped zone, where it is cooled by jets of water and removed periodically through the bottom to a sluiceway.

B. M. VENABLES.

Coolers for rotary furnaces. N. YOUNG (B.P. 327,017, 8.4.29).—The product from a rotary roasting or clinkering furnace drops into a trough-like housing surrounding the kiln, and is propelled along it by inclined

* The remainder of this set of Abstracts will appear in next week's issue.

blades attached to the outer surface of the kiln. Air for cooling may be passed through passages formed in the trough, and a water spray may be used for "hydrating" the clinker. B. M. VENABLES.

Apparatus for heat-treating articles. C. E. BUYSSE, Assr. to INDUSTRIAL HEATING EQUIPMENT CO. (U.S.P. 1,744,949, 28.1.30. Appl. 21.12.27).—The articles are placed in cylindrical containers and the rollers thus formed run down a track (hairpin-shaped in vertical section), through an ante-chamber into a heating chamber, and return from the latter chamber through the lower part of the former. While in the ante-chamber the ingoing goods are heated by waste gases above and by radiation from the outgoing goods below. B. M. VENABLES.

Fluid heaters. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 326,470, 11.12.28).—A method of providing joints in U-tubes such as are used in economisers is claimed. Lugs are formed on the abutting ends of the tubes, loose flanges with corresponding recessed holes are slipped over the lugs in the manner of a bayonet joint, and the flanges are bolted together. B. M. VENABLES.

Heat-exchange apparatus. R. SAMESREUTHER and G. KRÄNZLEIN (B.P. 305,172, 29.1.29. Ger., 1.2.28).—A method of attaching a number of closely spaced tubes to a header entirely by means of welding is described. The ends of the tubes are flanged out and the adjacent edges of the flanges are welded together, thus forming one wall of the header. B. M. VENABLES.

Headers for heat-exchanging apparatus. SKODA WORKS (B.P. 310,819, 30.4.29. Czechoslov., 30.4.28).—A header, which may conveniently be manufactured as a steel casting, is formed with an inwardly arched end-cover, with or without stiffening ribs, and with outwardly arched side-walls with stiffening ribs of undulatory form. Preferably the thickness of the ribs and other parts is the same throughout, but in any case no portion is to exceed $2\frac{1}{2}$ times the thickness of any other. B. M. VENABLES.

Heat-exchange apparatus. W. H. OWEN (B.P. 326,844, 21.12.28).—Methods of securing a cellular-type air heater in a conduit for flue gases are claimed. B. M. VENABLES.

Plate heat-exchange apparatus employing condensable gas or fluid. R. SELIGMAN (B.P. 327,377, 6.3.29. Holl., 7.3.28).—On each side of the heat-transferring plate passages for the respective fluids are formed by ribs in conjunction with plain or similarly ribbed closing plates. The passages may be zig-zag or give controlled parallel flow from and to cross-passages at the top and bottom; the latter arrangement is suitable for clearing condensate rapidly. B. M. VENABLES.

Device for effecting heat interchange. [Immer-sion heater.] W. S. BOWEN (U.S.P. 1,745,204, 28.1.30. Appl., 11.4.24).—In the steam generator of the type where combustion of gaseous fuel takes place in a conduit which is jacketed by the steam and water space, a sleeve and baffle are provided in the latter to promote circulation and prevent priming; also a spiral baffle of refractory material is placed in the combustion conduit,

thereby improving the heat transmission both by scouring away the film and by radiating heat through the film. B. M. VENABLES.

Power plants and apparatus therefor. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GEŖ. (B.P. 315,274, 9.7.29. Ger., 10.7.28).—In a power station in which the feed-water heaters for the whole station form the condensers of the "house" turbines, under some circumstances there may not be enough cool feed-water to condense the exhaust from the house turbines. On the principle that it is wiser to waste heat rather than distilled water (by exhausting to atmosphere), some of the hot feed is cooled by running it back through a main condenser, which may be either that belonging to a turbine which has unexpectedly had its load reduced, or a stand-by. There is only one valve to regulate, viz., the water by-pass. B. M. VENABLES.

Crusher. W. A. BATTEY and C. A. JAMISON, Assrs. to PENNSYLVANIA CRUSHER CO. (U.S.P. 1,743,996, 14.1.30. Appl., 18.10.27).—A form of frame for a heavy-duty single-roll crusher is described. B. M. VENABLES.

Pulveriser. R. SINCLAIR, Assr. to UNITED COMBUSTION ENGINEERS, INC. (U.S.P. 1,744,927, 28.1.30. Appl., 19.6.28).—A series of pulverising zones is arranged longitudinally of the apparatus and without dividing walls. The interior of the casing is fluted except opposite the last beaters where, being smooth and volute shaped, that zone acts to a certain extent as a fan; there is, however, an additional fan drawing external air and delivering it into the outlet conduit at such an angle that it induces flow through the pulveriser. Arrangements are made at the feed end to trap, by inertia, any very heavy particles, the falling coal or other material being diverted sharply into the pulveriser by jets of high-pressure air. B. M. VENABLES.

Pulverising machine. P. A. HIRSCH (U.S.P. 1,744,895, 28.1.30. Appl., 15.11.28).—In a disintegrator suitable for powdered fuel the hammers work in annular zones spaced longitudinally of the casing and alternating with zones containing fixed vanes which tend to throw large material back into the pulverising zone from which it came. Beyond the last pulveriser is a double fan one side of which draws only just enough air through the machine to transport the fine powder, but the other side draws external air and dilutes the stream for transport to a distant point. B. M. VENABLES.

Pulverising and like mills. E. W. GREEN and G. R. UNTHANK (B.P. 326,662, 14.3. and 4.12.29).—The mill comprises a number of balls rotating between two tracks; the lower is rotated but unyielding, the upper yieldingly supported, but otherwise stationary. The ground material flows by centrifugal force over an upwardly and outwardly sloping lip on the lower race, and enters a rising annular stream of air which conveys the whole of the material to an upper separating zone from which the oversize drops back to be re-ground. The annular stream comprises practically all the air passing through the apparatus. B. M. VENABLES.

Apparatus for determining the volume and density of granular materials passing through it. A. HANIQUE (B.P. 326,583, 29.12.28).—The apparatus

comprises a measuring device, such as a rotating drum divided into four buckets by radial partitions, above which is a distributing hopper and above that a feed hopper with automatic-discharge valve. The drum and the distributing hopper are counterpoised and they drop together only when there is a certain weight of material present in the two combined; their downward motion cuts off the supply from the feed hopper and permits the drum to rotate one step and discharge itself with consequent return to the charging position. Since the drum delivers a definite volume every time, but only operates when there is a definite weight present, the excess material left in the hopper is a measure of the density of the material and can be read off on a scale down the side. A check may be provided by weighing the material into the charging hopper and dividing by the number of cycles. B. M. VENABLES.

Mixing machines. V. BOOTH (B.P. 326,969, 25.2.29).—The apparatus comprises a rotary drum with an axial conveyor which serves to charge the material, to withdraw partly mixed material and recharge it with a further quantity of original material, also to discharge the finished material. The conveyor extends beyond the drum at both ends, a hopper for original material being at one end and a final discharge at the other. The top run carries the material from the hopper in one direction, the bottom run scrapes it along a trough in the other. Buckets are provided on the interior surface of the drum which lift and drop the material into the trough. Sliding doors in the bottom of the trough determine the point of delivery.

Mixing machines. A. WALLACE and W. M. WALLACE, JUN. (B.P. 327,000, 21.3.29).—A mixer for plastic or fluid materials comprises a horizontal cylindrical chamber with filling and emptying apertures, and a stirrer with axial shaft and radial beater blades and spokes; the latter carry scroll-like scraper elements which sweep the entire internal surface as closely as possible. At least one end of the casing is removable and the stirrer is subdivisible. B. M. VENABLES.

Converting suspensions of solids into pieces [coherent masses]. L. B. GUNDERSEN (B.P. 326,999, 19.3.29).—The fluid is poured into porous moulds and the water removed by currents of air across the moulds, under pressure on one side and vacuum on the other.

B. M. VENABLES.

Magnetic separation of materials. F. KRUPP GRUSONWERK A.-G. (B.P. 303,513, 1.1.29. Ger., 5.1.28).—A magnetic separator of the type in which a drum rotates between two poles is arranged so that the magnetic material which adheres to the drum in the first air-gap is passed through the second and is there cleaned. If additional output rather than a very clean product is desired, some additional raw material may be supplied to the second separating zone.

B. M. VENABLES.

Art of separating [discrete particles] and apparatus therefor. F. R. JOHNSON (U.S.P. 1,744,967, 28.1.30. Appl., 25.8.27).—A mixture of discrete particles having differing physical properties is fed to an intermediate point on an inclined belt which runs upwards, at such angle and speed that some particles will

roll down and others will be carried up. The actual separation may be effected by their shape or the friction of their surfaces, "by an underlying solid body obstructing the direct pull of gravity," or by electric charges. In the last case the feed chute may be electrified to one polarity and a mass underneath the belt to opposite polarity; further, by the use of another charged mass at the lower delivery point, a separation may be made there, giving three products in all. B. M. VENABLES.

Apparatus for regulating, varying, or controlling the flow of solid, gaseous, or liquid media. H. C. HEAD (B.P. 326,546, 15.12.28).—A squirrel cage is formed of overlapping longitudinal slats and a pair of end rings; the joints are left loose, the cage is inserted in a conduit, and the end rings are rotated with respect to each other. The slats then form a false wall to the conduit in the form of a Venturi throat. The rotation of a ring may be effected by a Bowden wire, and should it be desired to put the fluid in rotation the slats may be provided with radial projections. B. M. VENABLES.

Centrifugal machines. SHARPLES SPECIALTY CO., and L. D. JONES (B.P. 327,079, 11.7.29).—The apparatus is suitable for the separation of solids and liquids by straining. The rotor is constructed of a number of rings separated by circumferential drainage slits, the interior is conical, either smooth or stepped, and the solid matter is worked towards the small end by means such as helical blades rotating at a suitably different speed; this inner rotor also carries thin ploughs which extend into the slits and keep them clear. Since the solid matter moves in the opposite direction to the freely flowing pulp, which naturally moves to the large end, the product is drier. B. M. VENABLES.

Centrifugal machine. T. A. BRYSON, ASSR. to TOLHURST MACHINE WORKS, INC. (U.S.P. 1,743,753, 14.1.30. Appl., 8.7.21).—A self-balancing centrifuge is constructed of the following parts, in order downwards: a motor with centrifugal clutch; a driving spider having freedom to move radially and longitudinally; a spherical bearing permitting longitudinal and angular, but not radial, movement and a brake drum at the same height; then, some distance down, the basket; and finally a double spherical thrust-bearing permitting the shaft to gyrate. B. M. VENABLES.

Removal of vaporisable constituents from liquid, pasty, or solid matters. J. BRABAK (B.P. 326,829, 20.11.28).—The material is spread in a thin layer on a rotating drum and subjected to currents of air or other drying medium which arrive radially, flow a short distance circumferentially under increased pressure due to the convergence of the nozzles, and then depart radially under reduced pressure. The inlets and outlets are formed from the assemblage of a number of ribbed plates and are long and narrow, a large number of alternate ones being provided to cover a considerable arc of the drum. The condition for maximum efficiency is that the width of the inlet nozzles should be greater than twice the radial distance between the nozzles and material on the drum, but less than the width of the outlet nozzles; the circumferential distance between nozzles should also be greater than twice the above-mentioned radial distance. B. M. VENABLES.

Evaporators. H. WADE. From CHEM. FABR. J. BELLAK (B.P. 326,510, 14.12.28).—In an evaporator with steam-heated tubes to which the liquid is supplied as a drizzle, the heating surface of the horizontal rows of tubes decreases in succession downwards. Preferably they are arranged in groups having equal numbers of tubes in the horizontal rows of any one group, and below each group are collectors from which the liquid drips in fewer rows on to the tubes of the next group. Splashing is not relied on to distribute the liquid and the tubes are not staggered.

B. M. VENABLES.

Combined purifying and distilling apparatus. K. E. BUETTNER (U.S.P. 1,744,747, 28.1.30. Appl., 13.3.28).—A still suitable for the purification of dry-cleaning solvents, by means of caustic soda and decolorising carbon or other reagents, is constructed with a conical lower part for settling purposes; this part is provided with a steam jacket and with a live-steam injection pipe, together with means for admitting the dirty liquid and drawing off sludge. The distilling chamber above is provided with a steam-coil and with means for sampling and drawing off clean, settled liquid and for adding reagent. There is an overflow at the top of the distilling chamber and another near the top of a continuing stand-pipe, also a vent at the extreme top of the latter. The still is used either continuously or for batches in conjunction with a heat-exchanging condenser, moisture remover, and pump.

B. M. VENABLES.

Apparatus for distilling liquids [especially crude glycerin]. W. E. SANGER, Assr. to O. H. WURSTER (U.S.P. 1,743,289, 14.1.30. Appl., 23.7.23).—A distilling system substantially as described in U.S.P. 1,452,617 (B., 1923, 536 A) may be used for the distillation of glycerin of either dynamite or pharmaceutical quality. The concentrator is provided with a superheating coil in its upper part, which gives a preliminary superheat to the mixed vapours from "sweet water" in the evaporator and from the glycerin in the concentrator prior to passing through the superheater-condenser and thence being injected into the bottom of the still. The efficiency of the vapours is increased and the amount of heat supplied to the still (closed coil and/or external) is reduced, but the amount of glycerin condensed in the superheater-condenser, hence its grade, is controlled by the amount of superheating effected in the top coils of the concentrator. The above method of operation is used for glycerin of dynamite quality; where a pharmaceutical product is desired, pure water is boiled in the evaporator and the concentrator is not simultaneously used as such, only its superheating coil being heated. Roses for injected steam are provided in the concentrator and evaporator for a preliminary removal of impurities. To allow for the difference in vacuum between the receivers and the concentrator and evaporator, the latter are at a lower level and the transfer pipes may be provided with siphons and non-return valves. For cleaning the main still a water spray is arranged above, and a steam or air spray below, the heating coil.

B. M. VENABLES.

Stuffing-box for pressure stills. F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE CO. (U.S.P. 1,745,200,

28.1.30. Appl., 5.6.22. Renewed 11.7.29).—The packing is divided into two portions by a hollow spacer, the interior of the spacer being put into communication with the interior of the still through a condenser, so that any leakage that does take place is of liquid.

B. M. VENABLES.

Combination bubble cap and downflow. V. F. GRACE (U.S.P. 1,744,543, 21.1.30. Appl., 3.10.27).—The cap is H-shaped in vertical section and circular in plan, and a long downflow pipe is taken through the web; the lower flange surrounds a short rising pipe for vapour and the upper flange determines the depth of liquid in the tray. The edges of either or each flange may be serrated.

B. M. VENABLES.

Apparatus for separating dust from flowing gases. WASSMUTH, KURTH, & Co., A.-G., Assees. of A. WASSMUTH, GES.M.B.H. & Co. (B.P. 308,178, 18.3.29. Ger., 17.3.28).—A chamber is divided into two by a partition extending nearly to the bottom, and is provided with hopper-like outlets for dust. The gas is admitted downwards through a spreading device in the top of one compartment, and the clean gas exhausted through a similar device in the top of the other compartment.

B. M. VENABLES.

Removal of dust from gases, and more particularly producer gas, a reheating device, and a steam-mixing device for the combustion air. "MATECO" SOC. POUR LA CONSTRUCTION ET L'EXPLOIT. DU MATÉRIEL COLONIAL AU GAZ PAUVRE, SOC. ANON., Assees. of M. DELVAUX (B.P. 318,965, 29.8.29. Belg., 13.9.28).—The dust-laden gases are passed through a conduit bent to circular shape, so that the dust is concentrated into the outer layer, which travels into a large expansion chamber where the dust settles, and whence the portion of gas is withdrawn and caused to rejoin the main stream. Provision for preheating the air and adding steam to it is made in a conduit surrounding the cleaner.

B. M. VENABLES.

Filtering apparatus for air and other gases. S. DUNLOP. From W. BARTEL GES.M.B.H. (B.P. 327,083, 13.8.29).—In an apparatus comprising a number of filter bands which run one behind the other, the bands are driven at different speeds but preferably by a common mechanism. They may pass over rollers or the like, which are provided with peripheral openings for the emission of steam, air, water, or other cleansing fluid.

B. M. VENABLES.

[Heat exchanger for] liquefaction apparatus. J. W. DAVIS, Assr. to S. G. ALLEN (U.S.P. 1,744,108, 21.1.30. Appl., 3.7.24).—A heat exchanger suitable for separating undesired constituents from a gas by liquefaction, and comprising a bundle of twin concentric tubes, has the outer tubes threaded through and firmly fixed to a number of metallic plates which conduct heat transversely from tube to tube and equalise the temperature. The whole unit may be placed in a casing and surrounded by insulating material.

B. M. VENABLES.

Condensation of vapours from air and gases by cooling. M. FRÄNKEL (B.P. 308,228, 19.3.29. Ger., 19.3.28. Addn. to B.P. 294,354; B., 1928, 697).—Two

reversible, regenerative, cold-storage devices and a direct-contact cooler containing grids sprayed with chilled brine or the like are operated on the cycle described in the original patent to remove moisture from air. If non-return valves are used in the conduits the change-over may be effected by only one reversing valve.
B. M. VENABLES.

Separation of mixed gaseous components. E. MAZZA (B.P. 303,124, 28.12.28. It., 28.12.27).—The apparatus is intended to stratify the gaseous constituents by means of centrifugal force.
B. M. VENABLES.

Gas-analysing apparatus. E. PICK, Assr. to PERMUTIT Co. (U.S.P. 1,744,415, 21.1.30. Appl., 1.8.27. Renewed 24.9.29).—To protect the analyser proper, which may have metallic parts, the sample of gas is first passed through a compact mass of steel wool preceded, if desired, by a preliminary strainer.
B. M. VENABLES.

Carrying out of catalytic reactions. SELDEN Co., Assees. of A. O. JAEGER (B.P. 310,956, 6.4.29. U.S., 4.5.28).—In the catalytic air-oxidation of naphthalene to phthalic anhydride and in other exothermic vapour-phase catalytic reactions, the metal bath regulating the temperature forms a closed system in which actual boiling of the metal at the upper end of the reaction vessel (i.e., where fresh gases meet the catalyst) is prevented by the hydrostatic pressure of the metal in the higher portions of the system. The metal rises through tubes in contact with the catalyst, overflows at the top into a drum forming part of the closed system, and descends through an outer limb which is cooled so as to maintain a difference of about 100° between the bottom and top of the catalyst chamber. The drum contains an air space which may be kept at any desired increased or reduced pressure. Circulation is automatic and is accelerated, whenever incipient boiling takes place, by the "geyser" action of the bubbles of vapour. A suitable apparatus is described.
C. HOLLINS.

Apparatus for carrying out endothermic catalytic reactions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,025, 15.4.29).—The catalytic tubes or chambers are heated entirely or mainly by radiation; their outer walls and the inner walls of the surrounding heating chamber may be lined with material of high emissive power.
B. M. VENABLES.

Catalysing apparatus. SOC. ANON. D'OUGRÉE MARIHAYE (B.P. 327,378, 19.3.29. Belg., 7.4.28).—The catalysing agent (or its support) is shaped in the form of a surface of revolution of a catenary, the axis of revolution coinciding with the direction of admission of the gas to be treated. The containing chamber is of similar shape and the gas is led away through holes in the wall.
B. M. VENABLES.

Manufacture of catalysts of high mechanical stability. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,580, 24.12.28).—Finely-divided metals of groups II or III, or alloys or carbides thereof, are incorporated with the constituents of non-metallic catalysts by intensive mechanical intermixing at below 50°. *E.g.*, a catalyst for the destructive hydrogenation of hydrocarbon oils is prepared by the addition of

1—3% of aluminium or aluminium-bronze powder to a mixture of 65% MoO₃ and 35% ZnO; after thorough grinding below 50° until the metal has completely reacted with the other constituents the product dries to hard, stable granules which do not disintegrate during use.
A. R. POWELL.

Manufacture of catalysts containing free phosphoric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,185, 27.10.28).—The catalysts, which have prolonged activity, comprise a uniform dehydrated mixture of 10 pts. of a phosphate of an alkali or alkaline-earth metal, or a mixture of the two, up to 1 pt. of free phosphoric acid (or an equivalent amount of a substance, like ammonium phosphate, which produces phosphoric acid when heated), and, if desired, a carrier (graphite, pumice), with optional addition of a pyrogenic substance (*e.g.*, oxalic acid) which decomposes into gaseous products during the dehydration or practical application. The phosphate and acid may be dissolved and crystallised out after any of the optional additions. The catalysts are adapted for use in dehydration reactions, *e.g.*, the production of butadiene from $\alpha\gamma$ -butylene glycol, for acetal splitting, for the formation of ethylene from ethyl alcohol, etc.
S. K. TWEEDY.

[Carriers for] catalysts. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 304,654, 4.1.29. Fr., 23.1.28).—Catalyst carriers comprise material prepared by baking a mixture of refractory material, a binder, and organic material removed during the baking; *e.g.*, a mixture of dried sawdust, kaolin, water, Mexican mazout, and coal tar moulded by extrusion into hollow cylinders is baked at 1200—1400°, first in a reducing and later in an oxidising atmosphere. The product is impregnated with the desired catalyst in the usual manner.
L. A. COLES.

Preparation for removing or preventing boiler scale and other like deposits. H. KÖPPLINGER (U.S.P. 1,754,171, 8.4.30. Appl., 10.2.26. Ger., 4.1.26).—See B.P. 262,823; B., 1928, 110.

[Crushing or grinding] mill. A. STEINBRÜCKNER (U.S.P. 1,752,860, 1.4.30. Appl., 30.11.27. Ger., 8.12.26).—See B.P. 301,667; B., 1929, 154.

Grinding, pulverising, or disintegrating mill. W. A. CLOUD (U.S.P. 1,752,888, 1.4.30. Appl., 14.6.26. U.K., 20.7.25).—See B.P. 260,034; B., 1927, 31.

Discharge means for tube or ball mills. P. BODENSTEIN, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,753,685, 8.4.30. Appl., 18.6.28. Ger., 27.6.27).—See B.P. 292,941; B., 1929, 78.

Filtering and decanting apparatus. C. PICCARDO (U.S.P. 1,753,577, 8.4.30. Appl., 21.8.28. Ger., 13.2.28).—See B.P. 305,976; B., 1929, 499.

Apparatus for separating liquids of different densities. F. PINK (U.S.P. 1,754,119, 8.4.30. Appl., 29.3.28. U.K., 27.1.28).—See B.P. 298,683; B., 1929, 3.

Recovery of adsorbable substances [from gaseous mixtures]. A. B. RAY and G. T. FELBECK, Assrs. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,753,067, 1.4.30. Appl., 26.5.26).—See B.P. 291,277; B., 1928, 508.

Apparatus for separating dust from gases. A. STEVENART (U.S.P. 1,754,126, 8.4.30. Appl., 28.1.28. Belg., 4.2.27).—See B.P. 284,980; B., 1928, 432.

Gas washer. M. MALLET, ASS. to NEWTON CHAMBERS & Co., LTD. (U.S.P. 1,743,974, 14.1.30. Appl., 9.4.28. Fr., 27.9.27).—See B.P. 297,765; B., 1929, 4.

[Mechanical stoker for] furnaces. A. J. M. A. R. VAN DER DOES DE BIJE (B.P. 326,443, 11.10.28).

Closing shutters for tunnel ovens. EESTI PATENDI AKTSIASELT (B.P. 304,238, 15.1.29. Estonia, 17.1.28).

Continuously-acting absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 303,504, 11.12.28. Ger., 5.1.28).

[Mounting of capillary tubes of] thermometers. H. G. A. NEWMAN, and ACCURATE RECORDING INSTRUMENT Co., LTD. (B.P. 327,205, 7.1.29).

Fractional distillation (U.S.P. 1,744,261). Mixing devices (B.P. 327,339).—See II. Fire-extinguishing and -proofing compositions (B.P. 302,172 and 326,253).—See III. Cooling brine (B.P. 327,039). Water-softening material (U.S.P. 1,744,703).—See VII. Treating solids with liquids (B.P. 327,146).—See XV.

II.—FUEL; GAS; TAR; MINERAL OILS.

Primary thermal decomposition of coal. R. HOLROYD and R. V. WHEELER (Fuel, 1930, 9, 40—51, 76—93, 104—114. Cf. B., 1928, 880).—The distillation under low pressure of some bituminous coals and coal constituents has been studied; a very slow rate of heating was adopted and the temperature, which was raised in stages, was maintained constant at each stage for 120 hrs. The heavy oils, light oils (and water), and gases evolved at each stage were separately collected. No extensive decomposition of any coal constituent occurred below 300°. Occluded gases were driven off up to 150°, and slight changes in the external groupings of the ulmin compounds occurred, particularly with weathered coals, resulting in the evolution of water and oxides of carbon. Between 220° and 300° the major part of the free hydrocarbons in the coal (γ_1 -compounds) distilled unchanged. At 300°, or just above, any plant entities which the coal contained began to decompose, giving unsaturated hydrocarbons, neutral oxygenated compounds, and water. Above 300°, marked decomposition of the ulmin compounds occurred, the products being gaseous paraffins, water, phenolic oils, and liquid aromatic and hydroaromatic compounds. Above this "active decomposition point" of the coal (cf. B., 1929, 116) the ulmin compounds, organised plant entities, and resins decomposed and distilled simultaneously, yielding a complicated mixture of liquid products. The amount of resins which distilled unchanged was usually less than that extractable from the coal by solvents (γ_2 -compounds). As the distillation was carried beyond 400°, increasingly large quantities of gases were evolved, the proportion of hydrogen they contained rapidly increasing. Compounds containing nitrogen and sulphur appeared in small quantity in both gaseous and liquid products at all temperatures of distillation.

A. B. MANNING.

Coking value of coal. T. C. LLOYD (Chem. Met.

Eng., 1930, 37, 169—171).—The plastic range of various coals was studied by measuring the rate of gas flow through a tube charged with the sample under a maximum pressure of 2.2 in. of water. Results were recorded by graphs on the basis of 1 in. pressure, it being assumed that within the slight range of pressure variation the flow is proportional to the pressure. It was found that the plastic range of coking coals extends over a considerable temperature interval (up to 150°) and the width of this interval and the absence of pronounced rise in the rate of flow of gas on the high-temperature side of the plastic range are considered to be reliable indications of the coking quality. The advantage of blending two coals of different plastic ranges is apparent on this view. C. IRWIN.

Swelling of coals during the coking process. B. HOFMEISTER (Arch. Eisenhüttenw., 1929—1930, 3, 559—569; Stahl u. Eisen, 1930, 50, 391—392).—The influence of various factors on the "swelling pressure" of a coking coal has been studied. By Damm's method (B., 1928, 802) the swelling pressure of a coal, the period during which that pressure is exerted, and the subsequent shrinkage of the coke can all be determined. The pressure exerted by both dry and moist coals increased with the bulk density, which was itself dependent on the size of the coal and the closeness of packing. Diminution in the size of the coal particles at constant bulk density, however, produced a fall in the swelling pressure. Addition of moisture up to 5% also reduced the pressure, whilst a further increase in moisture had little effect. Addition of coke breeze reduced the pressure. The swelling pressure of a blend of two coals often departed very considerably (+150 to —50%) from the mean of the values for each coal. Increased rate of heating, in general, increased the swelling pressure. The mechanism of the coking process is very briefly discussed. A. B. MANNING.

Vertical-retort practice on a small works, with special reference to coal testing under normal working conditions. S. B. JONES (Gas J., 1930, 190, 96—97).—A small bed of four vertical retorts, which is alone in operation during the summer, is used to carry out thorough tests on different coals. Methods employed in weighing, sampling, and analysis are described.

R. H. GRIFFITH.

Determination of volatile matter in coal by the standard method, with a new and an old platinum crucible and a nickel crucible. D. J. W. KREULEN (Chem. Weekblad, 1930, 27, 227—229).—A new platinum crucible was found to give a figure for volatile matter 1% higher than that found with an old crucible; a nickel crucible gave intermediate figures, closer to those given by the new platinum. The differences are ascribed to varying surface activities. The greatest swelling of the coke was observed with the nickel crucible. S. I. LEVY.

Coke grading and coal blending. T. H. MADDEN (Gas J., 1930, 190, 95).—It has been found more satisfactory to produce graded coke by cutting large pieces than by screening the coke as produced from the retort; a plant installed for this purpose is described.

R. H. GRIFFITH.

Combustibility of coke. D. J. W. KREULEN (Chem. Weekblad, 1930, 27, 229—230).—The different rates of combustion of two cokes of the same analysis supplied with air at the same rate lead to differences in the maximum temperatures attained, on account of the difference in size of the zones of combustion. The factors influencing the combustibility are considered.

S. I. LEVY.

Domestic fuel by high-temperature carbonisation. J. J. BROWN (Gas J., 1930, 190, 92—94).—Tests on graded fuels were made on 12-lb. charges, ignited by a fixed gas burner in a special grate; temperature measurements were taken at 15-min. intervals after the fire had started. Ash and unburned fuel remaining at the end of the test were weighed and the general characteristics of the fire were noted, particularly after replenishing. The effect of coke size was studied in some preliminary experiments and pieces between 2 in. and $\frac{1}{2}$ in. were finally used in the comparison of cokes prepared by the high-temperature carbonisation of blended coals. The quality of the coke was influenced by changes in the proportion of caking to non-caking coal, and by altering the temperature or time of carbonisation. Large-scale production was accomplished on lines indicated by the experimental plant results, and a dry-quenched coke has been produced which is easily ignited and gives good radiation. For domestic use it compares favourably with raw coal or with a low-temperature semi-coke. The manufacture of a coke of this kind needs very careful selection of coals, and considerable supervision during their treatment.

R. H. GRIFFITH.

Pulverised fuel. J. T. DUNN and B. MOORE (Inst. Chem. Eng., Apr., 1930. Advance proof. 19 pp.).—The type of fuel, general principles and systems of pulverised-fuel firing are discussed. The degree of fineness of the fuel should be fixed in relation to the grade of fuel and the size of combustion chamber available, though particles larger than 100-mesh should be avoided as owing to incomplete combustion a loss as high as 4% may be involved. The direct or unitary system commends itself for small-sized plant on account of its relatively low initial cost and the short length of tube transport. Burners should possess high adjustability and capacity for operating with low air pressures, at the same time imparting a turbulent motion to the combustible mixture, as, e.g., by grooving the burner nozzle. The suitable ratio of combustion rate to furnace volume depends on various factors, but in modern boiler plants a combustion rate of 5—10 lb. of coal per cub. ft. of combustion space is not considered excessive, and shows good practice with 25—35% excess air and a flue gas with 13.5% CO₂. The trend of design to reduce erosion of furnace linings is in the direction of the replacement of refractory material by screens of cooled tubes, though the former possesses the advantage of providing an incandescent surface which aids the combustion particularly of fuels of low volatile content.

C. A. KING.

Peat. II. Dehydration of peat. G. STADNIKOV (Kolloidchem. Beih., 1930, 30, 297—333; cf., B., 1930, 354).—Direct measurements of the rate of dehydration of peat are difficult because the total loss from a 10-g.

sample in several hours is too small for differences to be observed in different kinds of peat, and if 200-g. samples be used other errors arise, for, in air drying, it is not possible to ensure even layering, and in dehydration in a desiccator the absorption of water by the sulphuric acid is so great that its vapour pressure rises continuously. Measurements are best conducted with samples of intermediate weight (about 60 g.). Such measurements carried out at a vapour pressure of 3—4 mm. or above 10 mm. indicated no difference in the velocity of dehydration of natural peat and a coagulated peat suspension, but at intermediate vapour pressures it seemed that the coagulated peat was dehydrated more rapidly. It is emphasised that this result cannot be regarded as conclusive. Both forms eventually reach the same equilibrium for a given vapour pressure. In any case the velocity of dehydration is proportional to the difference between the water-vapour pressure of the peat and that of the surrounding atmosphere. Peat placed on filter paper is dehydrated far more rapidly than when placed on glazed paper, and the rate on filter paper is reduced if the pores become stopped with the matter. These experiments reproduce to some extent the conditions of the drying fields in different weather conditions. So long as peat is left on the ground the process of dehydration comes to a more or less incomplete close, depending on the conditions. Artificial methods of dehydrating peat are described; the cataphoretic method of Schwerin is inefficient.

E. S. HEDGES.

Waste-wood utilisation by the Badger-Stafford process. W. G. NELSON (Ind. Eng. Chem., 1930, 22, 312—315).—The process of distillation of about 400 tons of scrap wood per day is described. Larger pieces are broken up and handled separately from sawdust and shavings. Six rotary dryers, 100 ft. long and 10 ft. in diam., reduce the moisture content to 0.5%, at which stage the wood is transferred through butterfly valves to three retorts which operate continuously by utilising the heat developed by the exothermic carbonisation of material near the centre. The direct products are charcoal, pyroligneous acid, and gases having an average heating value of 290 B.Th.U. per cub. ft. The bulk of the charcoal is pulverised and briquetted, or burnt for steam-raising purposes, and the liquors are distilled, yielding approx. 4.5% of methyl alcohol, 12% of acetic acid per ton of wood, together with other allied products.

C. A. KING.

Oxide purification [of coal gas]. S. K. HAWTHORN (Gas J., 1930, 189, 834—836).—A special iron oxide purifying material, prepared by treating spent oxide for sulphur removal and then for cyanide recovery, is found to give increased capacity to the plant. Further advantages are due to reduced handling of the ore, and improvements in working methods have greatly diminished the costs of purification.

R. H. GRIFFITH.

[Coal]-gas dehydration for small works. F. PORTEOUS (Gas J., 1930, 189, 816—817).—A brief summary of advantages resulting from dehydration of coal gas.

R. H. GRIFFITH.

Analysis of Sarmas natural gas. Z. KERTÉSZ (Petroleum, 1930, 26, 393—396).—Recent research in Turda shows that this gas is quite dry, and is the purest

natural methane gas. It contains no oxygen, carbon dioxide, carbon monoxide, or heavy hydrocarbons; the mean analytical result was CH_4 99.18%, H_2 0.43%, and N_2 0.39% by vol. It is used as a heating material in lime kilns. W. S. E. CLARKE.

Natural gas and "cracking" gas and their industrial utilisation. H. M. STANLEY (J. Inst. Petroleum Tech., 1929, 15, 516—552).—Recent advances in the subject are reviewed and a full bibliography is given.

Crystallisable phenols of brown-coal tar. F. SCHULZ and J. PRUNET (Paliva a Top., 1929, 10, 61—68; Chem. Zentr., 1929, ii, 3087).—Tar oil from coal from the Karl pit in Cukmantl contains a fraction, b.p. 254—257°, m.p. 59°, which appears to consist of a mixture of a dihydroxybenzene with a dihydroxytoluene.

A. A. ELDRIDGE.

Wood tar for making axle grease. A. DENNEMARK (Neff. Choz., 1929, 17, 236—240).—Wood tar is treated with kerosene to precipitate pitch, the acids are saponified, the soap is separated, and the residual mixture is steam-distilled. Axle grease is made from the tar (20%), lime (10%), and fuel oil (70%). Wood oil must first be eliminated. The grease should have m.p. 121°.

CHEMICAL ABSTRACTS.

Viscosities of [tar and] bituminous materials and the relationship between values obtained by different methods. H. METZGER (Z. angew. Chem., 1930, 43, 289—293).—There is a constant relationship between the temperature interval from the setting point, as determined by the Hoepfner-Metzger method, to the drop point and the interval between the first-mentioned temperature and the softening point. For the Kraemer-Sarnow softening point the factor is 0.6816, and for the ring-and-ball softening point 0.8721. The Hoepfner-Metzger system grades the setting point as 0.0 and the drop point as 1.0. If the viscosity-temperature curve of a tar or pitch on this system is depicted with right-angle co-ordinates, the softening points becoming lines parallel to the temperature axis. The Richardson penetration value has not a constant relationship to the Hoepfner-Metzger points, and is depicted by curves obtained by taking average results from a number of samples of bitumen. In a similar way curves are obtained giving the relation to the Hoepfner-Metzger values of viscosities by the Hutchinson, Engler, etc. methods, which are, of course, measured in seconds. The whole series of curves are exhibited on one diagram. The accuracy of the relationships depends largely on the accurate determination of the setting point.

C. IRWIN.

Ring-and-ball method of test for softening point of bituminous materials, resins, and similar substances. P. H. WALKER (Bur. Stand. J. Res., 1930, 4, 195—201).—Modified procedure and apparatus as used at the Bureau of Standards for carrying out this test are described.

C. J. SMITHELLS.

Magnetic double refraction of petroleum of various origin. A. COTTON and M. SCHERER (Compt. rend., 1930, 190, 700—702).—The magnetic double refraction (β) of six samples of petroleum from (i) Texaco-

West-Columbia, (ii) Pechelbronn, (iii) Mid-continent, (iv) Columbia, (v) Iraq, and (vi) Moreni (prepared by a preliminary distillation to 250°, treatment at 95° with 1% sulphuric acid, washing with alkali and water, and redistillation to 250°) has been determined at 14.8—16.7° for light of wave-lengths $\lambda = 578, 546$, and 436 m μ . The values of C_m/d (the magnetic double refraction for unit field and unit mass, C_m for nitrobenzene being taken as 253×10^{-14}) have been thence deduced for $\lambda = 578$. The density (d) of the oil, values of β for $\lambda = 436$ and 578, and $(C_m/d) \times 10^{-14}$ are (in this order): (i) 0.855, 0.53°, 0.86°, 6.25; (ii) 0.779, 0.43°, 0.64°, 5.53; (iii) —, —, 0.66°, —; (iv) 0.859, 0.70°, 1.01°, 8.15; (v) 0.767, 0.63°, 0.88°, 8.21; (vi) 0.840, 2.27°, 3.34°, 27.00. These figures show differences which may be of use in distinguishing the oils. C. A. SILBERRAD.

Determination of mol. wts. of non-volatile petroleum oils. J. M. DEVINE (Univ. Oklahoma Bull., 1929, 9, 131—137).—Determinations of mol. wt. by the b.p. and f.p. methods agree for the same solvent when the values are extrapolated to zero concentration. Ethylene dichloride as solvent gives values 5% higher, and carbon tetrachloride 5% lower, than does benzene.

CHEMICAL ABSTRACTS.

Mineral oil from new sources. B. HLAVICA and F. TRČA (Paliva a Top., 1928, 10, 101—102; Chem. Zentr., 1929, ii, 3087).—Analyses of oils from Turzonka and Hodonin are recorded.

A. A. ELDRIDGE.

Composition of tar from some North Bohemian coals. B. G. SIMEK (Paliva a Top., 1928, 10, 157—160; Zentr., 1929, ii, 3086).

Low-temperature tars. J. TICHÝ (Paliva a Top., 1929, 11, 33—37, 42—50, 60—61; Chem. Zentr., 1929, ii, 3086).

Pneumatic transport. NEU. **Combustion-gas dryers.** HATTA.—See I. **Absorption of carbon monoxide.** GUMP and ERNST.—See VII. **Road asphalts.** MACZYŃSKI and SKALMOWSKI. **Road emulsions.** SHIPEROVICH and others.—See IX. **Coal-fired annealing furnaces.** STÄBLER.—See X. **[Carbon from] rice husks.** DE MONGEOT.—See XIX. **Low-viscosity nitrocellulose.** MILLIKEN.—See XXII.

PATENTS.

Coking retort ovens. KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 302,365 and 315,847, [A] 14.12.28, [B] 11.9.28. U.S., [A] 15.12.27, [B] 20.7.28. [B] Addn. to B.P. 304,743; B., 1929, 967).—(A) The heating walls of a coke-oven battery are provided with vertical flame flues divided into groups in which the gases alternately ascend and descend. The flues communicate at the top and there is no dividing wall between the different groups. Below the chambers and parallel thereto are the regenerators, which are divided into pairs by a central transverse partition. In the brickwork separating the chambers and heating walls from the regenerators are transverse horizontal flues extending the length of the battery. Some of these flues connect inflow regenerators to certain of the flame flues of each heating wall, whilst the others connect the outflow regenerators to the other flame flues of each heating wall, and *vice versa* on reversing the operating conditions. (B) The

oven described in the prior patent is modified by so connecting the regenerators, their respective flue groups, and the gas supply that the respective groups of flues of each heating system in the same heating wall simultaneously convey either burning gases or waste gases.

A. B. MANNING.

Device for the utilisation of the sensible heat of the distillation gases from ovens for production of gas and coke. N.V. SILICA EN OVENBOUW MIJ. (B.P. 310,892, 2.5.29. Ger., 2.5.28).—A removable steam generator formed by two vertical, concentric tubes is arranged centrally in each of the ascension pipes leading from the oven to the gas main. The ascension pipes are provided with fireproof linings. The inner of the two tubes serves as a supply tube for the water to be evaporated, while the outer tube serves for the production of the steam. All the inner tubes are connected, through valves, to a common water-pipe, and all the outer tubes similarly to a common steam-pipe. Tubes from the common water- and steam-pipes lead to a steam-drum.

A. B. MANNING.

[Low-temperature] distillation of fuel. C. B. THWAITE (B.P. 326,492, 13.12.28).—The finely-divided fuel is charged into pockets in a horizontal tray within a heating chamber or retort, and is carbonised therein by passing hot waste gases, *e.g.*, from coke ovens or a blast furnace, through the retort. A charging hopper is provided at the top of the retort, and the fuel, which is delivered on to the tray, is spread evenly in the pockets by means of arms operated from outside the retort. When carbonisation is complete, the tray, which is rotatable about a horizontal axis, is inverted; the low-temperature coke falls to the bottom of the retort and is withdrawn through a double-conical discharge valve.

A. B. MANNING.

Distillation and gasification of solid fuel. T. LICHTENBERGER (B.P. 309,190, 6.4.29. Ger., 7.4.28).—A gas generator has a vertical shaft the lower part of which is surrounded by an annular trough or bath through which molten salt at 800–1000° is circulated. At the base of the shaft is a rotary grate, through which steam is supplied to the generator, and a water-seal which prevents access of air to or escape of gas from the generator.

A. B. MANNING.

Distillation of coal, shale, peat, wood, and other fragmentary solid materials. S. MOORE (B.P. 326,540, 15.12.28).—The material is conveyed in a series of separate charges, *e.g.*, by means of spaced push-plates attached to an endless chain, through a horizontal retort the floor of which is heated externally by contact with a bath of molten metal. The retort is provided with gas-tight delivery and discharge orifices and means for regulating the rate of delivery and discharge of the material.

A. B. MANNING.

Carbonisation or gasification of coal and the like. K. C. APPELYARD. From E. O'TOOLE (B.P. 326,493, 6.9.28).—The coal is charged into a vertical retort in the upper part of which are a number of vertical tubes which mould passage-ways in the coal when it becomes softened by heat and which serve to convey the heating gases and the products of distillation from the retort. The coal is distilled by passing up through it

the hot gases from a combustion chamber below the retort. The retort comprises a number of sections fitting one above the other, and preferably constructed of an outside shell of steel lined with firebrick. Each section is made up of two semi-cylindrical halves bolted together. When the coke in the bottom section is completely carbonised the section together with the coke it contains is removed to a quenching platform, the retort is lowered, and another section added at the top. Dowel pins hold the material in the upper part of the retort during this procedure. At the top of the retort is a hood to which is attached a ring of sheet iron which extends down through the first and a little into the second section; this prevents escape of gas when the upper part of the retort is lowered and before the new upper section is in place.

A. B. MANNING.

Apparatus for the carbonisation of coal and like material. ILLINGWORTH CARBONIZATION Co., LTD., S. R. ILLINGWORTH, SOUTH METROPOLITAN GAS Co., and E.V. EVANS (B.P. 326,247, 10.12.28).—The apparatus comprises a brickwork outer setting within which are retorts constructed of H-section metal conductors (cf. B.P. 245,190; B., 1926, 228); these are used for the low-temperature carbonisation of coal etc. The metal conductors are mounted on L-section seatings, detachably secured to the base of the brickwork setting, so that they may be readily withdrawn when it is desired to use the retorts for high-temperature carbonisation.

A. B. MANNING.

Plant for carbonisation of coal and like material. ILLINGWORTH CARBONIZATION Co., LTD., and S. R. ILLINGWORTH (B.P. 326,169, 29.11.28. Cf. B.P. 245,190 and 244,837; B., 1926, 228, 428).—A number of retorts in the form of vertical metal tubes are arranged in concentric rings within a brickwork setting having a central exhaust flue. The retorts are heated preferably by means of a system of flues similar to that described in B.P. 244,837 (*loc. cit.*). They are charged from chambers which are designed to deliver a measured quantity of coal into each retort, and which are mounted on a rotatable platform, one such chamber being provided for charging each ring of retorts. The measuring chambers, which are filled from a bunker overhead, are provided with inlet and outlet valve-plates. The rotatable-platform forms the top of the offtake chamber for the retorts, and is provided with a peripheral flange which runs in a water-seal. Discharge of the carbonised material under the action of gravity is effected periodically by means which are automatically operated in timed relationship with the charging means.

A. B. MANNING.

Apparatus for heat treatment of the volatile products from carbonisation of coal. ILLINGWORTH CARBONIZATION Co., LTD., and S. R. ILLINGWORTH (B.P. 326,135, 29.11.28).—A retort setting of the type described in B.P. 245,190 and 244,837 (B., 1926, 228, 428) is provided with a vertical coke chamber wherein the volatile products passing from the retorts are brought into contact with coke at about 700° and are thereby converted into useful hydrocarbons. On each side of the coke chamber are vertical heating flues from which the hot gases pass to horizontal flues for heating the retorts.

A. B. MANNING.

Refining of active carbon. I. G. FARBENIND. A.-G. (B.P. 301,313, 23.11.28. Ger., 26.11.27).—Active carbons containing adsorbed acid electrolytes, *e.g.*, hydrochloric acid, chlorides, or sulphates, which would tend to corrode the containing vessels, are treated with nitric acid or a nitrate, and the nitrate ions are subsequently removed by washing, or steaming, etc. [Stat. ref.]

A. B. MANNING.

Manufacture of fuel. R. W. STREHLENERT (U.S.P. 1,743,985, 14.1.30. Appl., 16.6.27. Swed., 18.5.26).—Dry sawdust is mixed with waste sulphite liquor from cellulose manufacture and containing 25–60% of dry materials, and, after partially drying, the mixture is heated with steam and pressed into briquettes, which are dried and heated at 200°.

F. R. ENNOS.

Destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,157, 3.9. and 19.10.28).—The yield of hydrogenated oils obtained from coal and heavy mineral oils, often low owing to the formation of sludges which cause the deposition of the catalyst, is improved by a continuous circulation or movement of the liquid. Pumps of various types may be used, such as geared, valveless, centrifugal, or reciprocating. The hydrogen is continuously introduced into the liquid through a number of jets. Light products are carried with the hydrogen in the form of vapour and are separated. From the hydrogenated, unvaporised residue lubricating oils may be separated. A portion of the circulating liquid may be continuously withdrawn and the catalyst reactivated. Fresh raw material and catalyst are continuously added.

T. A. SMITH.

Destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,184, 3.9.28. Cf. B.P. 326,157; preceding abstract).—The materials in the form of liquids or pastes are subjected to destructive hydrogenation under pressure in the presence of colloiddally dispersed catalysts which are immune from poisoning by sulphur, *e.g.*, mixtures of molybdenum, zinc, and magnesium oxides. A portion of the reaction liquid may be branched off, either continuously or periodically, and the catalysts contained therein may be regenerated without separation from the liquid.

A. B. MANNING.

Destructive hydrogenation [of oils, tars, suspensions of coal, etc.]. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,586, 31.12.28).—The material in the form of a continuously falling, annular, liquid film is submitted to the simultaneous action of hydrogen under pressure and heat from a central heating element which does not come into contact with the material. Forms of apparatus suitable for carrying out the process are described.

A. B. MANNING.

Hydrogenation of coal, oils, and similar materials. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,238, 8.12.28).—The reaction vessel is heated by circulating hydrogen at a suitable temperature, *e.g.*, 450°, through a surrounding jacket, and all or part of this hydrogen is then admitted into the reaction vessel together with the preheated coal, oil, etc. The jacketed reaction

vessel is mounted within a high-pressure vessel. The intervening space is packed with heat-insulating material and the high-pressure vessel is also provided with a jacket, through which cooling gases are passed.

A. B. MANNING.

Water-gas generators. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,293, 25.1.29).—Coke is charged into a deep vertical shaft and the lower portion is subjected to alternate blasting with air and steam. The blow gases are led off, without passing through the upper portion of the coke bed, into regenerators which are used for preheating the steam during the "run." Part of the gas resulting from the steam blast (the "run") is passed through the upper portion of the coke bed and the remainder through regenerators used for preheating the incoming air for the succeeding "blow." During the "blow," part of the preheated air is by-passed to form secondary air for the combustion of the blow gases in a chamber adjacent to the regenerators.

A. B. MANNING.

Production of gases rich in carbon monoxide. (S.I.R.I.) SOC. ITAL. RICERCHE INDUSTRIALI (B.P. 306,959, 26.2.29. It., 29.2.28).—Solid fuels are gasified by injecting oxygen and steam (or oxygen and carbon dioxide) into a producer at a pressure which is unable to cause agitation of the fuel, but is sufficient to bring about rapid combustion and to carry away the ash as a fine dust containing the alkali and alkaline-earth compounds in the form of the oxides or hydroxides. The gas is washed with water, which dissolves these alkaline compounds and fixes the greater part of the carbon dioxide and the sulphur compounds in the gas. The residual ash is removed in dust separators.

A. B. MANNING.

Purification of gases containing hydrogen sulphide. C. STILL (B.P. 297,061, 13.9.28. Ger., 13.9.27).—The gases are washed with a suspension, in an alkaline solution, of an oxide or hydrated oxide of iron containing a small proportion, *e.g.*, 5%, of an oxide or hydrated oxide of manganese. The addition of the manganese increases the rate of absorption of the hydrogen sulphide and the rate of regeneration of the spent liquor; it also considerably reduces the formation of acid by the oxidation of sulphur during regeneration.

A. B. MANNING.

Bituminous composition and its manufacture. L. KIRSCHBRAUN (U.S.P. 1,738,906, 10.12.29. Appl., 22.7.27).—A waterproofing composition is formed by agitating and thereby aerating an aqueous dispersion of asphalt or similar bituminous material, preferably containing bentonite, in the presence of an agent having a low surface tension, *e.g.*, an alkaline-earth oxide, albuminous substance, or soap. The resulting foamy mass, which has a substantially permanent cellular and sponge-like structure, contains gas, and retains its shape and characteristics after drying out, is incorporated with cementitious material.

H. S. GARLICK.

Restoring the quality of used gasoline. F. H. MCBERTY, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,745,108, 28.1.30. Appl., 13.6.27).—Mechanically removable impurities are separated from dry-cleaner's solvent, which is then treated with a mineral acid, the

insoluble products are removed, the oil is treated with an alkali to effect partial neutralisation, and the remaining acidity is neutralised with ammonia.

H. S. GARLICK.

Treatment of petroleum oils. W. M. CROSS (U.S.P. 1,744,262, 21.1.30. Appl., 17.12.25. Renewed 9.8.29).—Crude petroleum is cleaned and the higher fractions are extracted by preheating the oil to above 100°, separating the liquid from the vapour-phase material, and passing the latter to a fractionating stage. The liquid products pass to a secondary heating stage where additional heat is imparted, and then on to a combined re-boiler and partial cooler in which the temperature is controlled before being admitted to the vaporisation stage, where additional fractions of vapour-phase hydrocarbons are separated and a free-flowing, residual bottom is obtained from the oil. Excess heat in the oil prior to evaporation is utilised for reboiling in the re-boiler and partial cooler, a condensate separated out during fractionation, and the vapours evolved in the vaporising stage are subjected to fractionation and final condensation.

H. S. GARLICK.

Refining of hydrocarbon oils. W. A. STREET (B.P. 326,163, 5.11.28).—Used lubricating oils are treated with a mixture of 1 pt. of sulphuric acid (*d* 1.84) and 4 pts. of new or recovered lubricating oil. This mixture (1 vol.-%) is well stirred into the used lubricating oil with the addition of 20–50% of petroleum spirit to ensure rapid settling.

T. A. SMITH.

Production of refined hydrocarbon oils and the like. I. G. FARBENIND. A.-G. (B.P. 300,900, 6.11.28. Ger., 19.11.27).—The sulphur content of impure oils is reduced by hydrogenation at temperatures of 300–420° and pressures above 20 atm. in the presence of catalysts of group VI or of cobalt and its compounds. The unsaturated content of the oils is also reduced. The process is particularly applicable to the refining of cracked distillates and rendering the oils obtained by the low-temperature carbonisation of coal suitable for use in Diesel engines. Oils low in hydrogen content may be separated from other oils by means of liquid sulphur dioxide and then refined and hydrogenated. When benzene vapour, obtained by the low-temperature carbonisation of brown coal, and containing 1% S and 25% of unsaturated hydrocarbons, is passed at 410° and 200 atm. through a tube, 2.5 cm. diam. and 150 cm. long, containing a mixture of tungstic acid and magnesia, a saturated benzene free from sulphur is obtained. The time of contact is 30 sec.

T. A. SMITH.

Treatment of hydrocarbon oil. C. W. WATSON, Assr. to TEXAS Co. (U.S.P. 1,744,599, 21.1.30. Appl., 11.11.27).—A battery of shell stills is arranged for continuous distillation in a series of groups of stills, each group being maintained under conditions of distillation and provided with a fractionating zone. Distillates suitable for forming a fraction of the boiling range of gasoline are taken off from the first group of the series, those suitable for forming a kerosene fraction are removed from the next group of the series, and the oil itself is introduced into the last still of each group at an intermediate point in the corresponding fractionating zones.

H. S. GARLICK.

Cracking of mineral oil. J. H. PEW, Assr. to SUN OIL Co. (U.S.P. 1,744,574, 21.1.30. Appl., 10.2.26).—A stream of oil is flowed through a series of cracking zones in heat-exchange relation with a vaporised metallic heating medium, *e.g.*, mercury, each zone being maintained at a temperature high enough to effect cracking of the oil with formation and accumulation of asphaltene within the cracking zones, but below that required to form any substantial amount of insoluble coky residue. This is effected by maintaining the metallic vapour at progressively higher temperatures in the successive heating zones and arranging in each zone for condensation of metallic vapour and transfer of its latent heat to the oil. At definite periods the process is stopped and a solvent for the asphaltene is flowed through and out of part of the cracking zones.

H. S. GARLICK.

Carburetted or mixing devices for air and liquid fuel, applicable also to the mixing of liquids or gases together. J. W. SMITH (B.P. 327,339, 7.6.29).—The fluids are passed through an annular conduit which is provided with a number of baffles alternately and oppositely inclined from each wall. The free edges of the baffles are formed with a large number of pointed blades like multi-pointed stars; the blades may be given a twist alternately in opposite directions, and their number and angle of inclination may be different on different stars.

B. M. VENABLES.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,754,131, 8.4.30. Appl., 4.5.23).—See B.P. 215,306; B., 1925, 274.

Coking retort oven. J. BECKER and J. VAN ACKEREN, Assrs. to KOPPERS Co. (U.S.P. 1,752,363, 1.4.30. Appl., 27.9.26).—See B.P. 278,012; B., 1929, 160.

Binder for fuel agglomerates. F. M. CROSSMAN (U.S.P. 1,752,838, 1.4.30. Appl., 5.12.24).—See B.P. 244,053; B., 1927, 98.

Polymerisation of oils. S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,752,921, 1.4.30. Appl., 23.1.25).—See B.P. 246,491; B., 1927, 722.

Production of montan wax. F. ZSCHOCK and H. RODRIAN, Assrs. to A. RIEBECK'SCHE MONTANWERKE A.-G. (U.S.P. 1,753,816, 8.4.30. Appl., 13.3.28. Ger., 16.9.27).—See B.P. 297,102; B., 1929, 1007.

Pulverisers (U.S.P. 1,744,927 and 1,744,895). **Mixing of liquids** (B.P. 326,924). **Stuffing-box for stills** (U.S.P. 1,745,200). **Removing dust from gases** (B.P. 318,965). **Filtering gases** (B.P. 327,083). **Gas cleaner** (U.S.P. 1,743,344).—See I. **Hydrocarbons from methane** (B.P. 316,126).—See III. **Carbon monoxide** (B.P. 326,227).—See VII. **Catalysts** (B.P. 304,654).—See VIII. **Dispersions of bitumens** (U.S.P. 1,738,776). **Bituminous paving** (B.P. 326,863).—See IX. **Catalysts** (B.P. 326,580).—See X. **Insecticide** (U.S.P. 1,744,324).—See XVI.

III.—ORGANIC INTERMEDIATES.

Preparation of glycol by hydrolysis of ethylene [di]chloride. H. BAHR and H. ZIEGLER (Z. angew. Chem., 1930, 43, 286–289).—The manufacture of glycol in America owes its origin to the availability of ethylene

in cracking gases, but fractions rich in ethylene are also obtained in Europe in synthetic ammonia plants employing coke-oven gas from which the nitrogen-hydrogen gas is obtained by partial liquefaction. This may be converted into glycol either through ethylenechlorohydrin or ethylene dichloride. The latter is not decomposed by water under ordinary conditions and alkalis produce principally vinyl chloride and acetylene. It is necessary therefore to convert the ethylene dichloride into an ester by treatment with, *e.g.*, sodium acetate and then effect hydrolysis with alkali. Experiments showed that the direct hydrolysis of ethylene dichloride (b.p. 84°) was facilitated by increased temperature and pressure. The yield per hr. rose from nil at 120° to 34% at 180°. Aldehydes begin to be formed in quantity if the hydrochloric acid concentration rises above 1%. A method was therefore adopted of autoclave treatment with addition of alkali at intervals to neutralise the hydrochloric acid. Yields of about 70% were obtained, working at 150—170°, with only small quantities of aldehyde and vinyl chloride. A loss of 20—25% was attributed to aldehyde having been volatilised or resinified. On a larger scale a yield of 1.5—2.0 kg. of glycol per hr. per 100 litres of liquid was obtained, using a reaction mixture containing 4—5% of ethylene dichloride. It is scarcely practicable to replace caustic soda by lime, and little advantage would be obtained as the sodium chloride is required for electrolysis. C. IRWIN.

Vapour pressure and [latent] heat of vaporisation of toluene. D. S. DAVIS (Ind. Eng. Chem., 1930, 22, 380—381).—The following equation, relating the vapour pressure, p , of toluene with T , the absolute temperature, $\log (41.6/p - 0.28) = 3.106(593.7/T - 0.08158)$, is suggested to correlate recent vapour-pressure measurements on toluene. The different empirical equations used to calculate the latent heat of toluene are compared. H. INGLESON.

Waste wood utilisation. NELSON.—See II. **Citric acid.** CATULLO.—See VII. **Absolute alcohol.** GUINOT.—See XVIII. **Utilisation of rice husks.** DE MONGEOT.—See XIX. **Detection of isopropyl alcohol.** BOEHM and BODENDORFF.—See XX.

PATENTS.

Production of higher hydrocarbons from methane. F. FISCHER and H. PICHLER (B.P. 316,126, 14.12.28. Ger., 23.7.28).—Methane, in the pure state or diluted with other gases, is converted into higher unsaturated and aromatic hydrocarbons by subjecting it to a temperature of at least 1000° for a period not exceeding 1 sec., in the absence of catalysts or oxygen-containing gases. The process is carried out by passing the gas at a high velocity through narrow tubes maintained at the required temperature. The free hydrogen produced at the same time may be reconverted into methane, *e.g.*, by interaction with carbon monoxide in the presence of a suitable catalyst, and this again subjected to thermal decomposition. A. B. MANNING.

Manufacture of butylene from ethylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,322, 22.2.29).—Ethylene under a pressure of 55—60 atm. is led with a little boron fluoride over a nickel catalyst at

15° (contained, *e.g.*, in a 4-litre autoclave packed with Raschig rings); 1.5 litres of butylene per hr. are obtained. C. HOLLINS.

Preparation of higher alcohols from ethylene and its homologues. COMP. DE BÉTHUNE (F.P. 636,651, 20.10.26).—In the process of B.P. 273,263 (B., 1927, 859), one tower is charged with dilute acid, cooled to 15—20°, which after absorption is worked up for higher alcohols and ethers in the usual way.

R. BRIGHTMAN.

Concentration of aqueous acetic acid. I. G. FARBENIND. A.-G. (B.P. 302,174, 10.12.28. Ger., 8.12.27).—In the extraction method for the concentration of acetic acid a secondary liquid (light petroleum, petroleum, benzene, tetrahydronaphthalene, trichloroethylene) is used so that less water is extracted and on subsequent distillation of the extract all remaining water is carried over with the secondary liquid. The latter is preferably a liquid boiling below 119°, and is used in conjunction with a high-boiling solvent. Alternatively, a low-boiling solvent and a high-boiling secondary liquid are used. Basic solvents are excluded. C. HOLLINS.

Hydrogenation of polyhydroxy-compounds [glycerol from dextrose etc.]. I. G. FARBENIND. A.-G. (B.P. 299,373, 24.10.28. Ger., 24.10.27).—Glycerin, or its reduction product, propylene glycol, is obtained by catalytic hydrogenation of polyhydroxylated compounds in presence of metals of the iron or platinum group (nickel). Sorbitol at 200—250° with hydrogen at 70—100 atm. gives first glycerol and then propylene glycol, b.p. 186—188°; dextrose or sucrose is reduced at 150° and 40 atm. to the hexitol and then at 200—250° and 100 atm. to glycerol and the glycol. Starch and cellulose are similarly reduced at 210—260° and 70—100 atm. C. HOLLINS.

Preparation of cyclohexylbenzene and its homologues. P. SCHVING (F.P. 636,686, 23.10.26).—*cyclo*-Hexanol or one of its homologues is heated with benzene or homologues in presence of sulphuric acid. *cyclo*-Hexanol and benzene give 56% of *cyclohexylbenzene* and 12—16% of *p*-*dicyclohexylbenzene*.

R. BRIGHTMAN.

Manufacture of triarylcarbinols. IMPERIAL CHEM. INDUSTRIES, LTD., and S. COFFEY (B.P. 325,933, 1.1.29).—A diaryl ketone (except tetra-alkyldiaminobenzophenones) mixed with an aryl halide is added to sodium or potassium in hot benzene or other diluent. The preparation of diphenyl-*p*-tolylcarbinol, m.p. 72—73°, from benzophenone and *p*-chlorotoluene, phenyldi-*p*-tolylcarbinol, m.p. 76—77°, from di-*p*-tolyl ketone and bromobenzene, triphenylcarbinol from benzophenone and chlorobenzene, *p*-methoxytriphenylcarbinol from benzophenone and *p*-chloroanisole, diphenyl- α -naphthylcarbinol from phenyl α -naphthyl ketone and bromobenzene, and 4:4'-dimethoxytriphenylcarbinol from 4-methoxybenzophenone and *p*-chloroanisole is described. C. HOLLINS.

Manufacture of organic phosphorus compounds [moth-proofing agents]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,137, 30.11.28).—Phenols are heated with triarylphosphine oxides, PAR_3O , to give products of the type, $\text{PAR}_3(\text{OH})\cdot\text{OAr}$, which are

useful moth-proofing agents. Compounds of triphenylphosphine oxide with phenol (m.p. 106°), *o*-, *m*-, and *p*-cresols (m.p. 67°, 89–90°, 44–45°), *o*- and *p*-chlorophenols (m.p. 105–106°, 80–81°), thymol (m.p. 67–68°), 6-chlorocarvacrol (m.p. 65–66°), 2:6-dichlorophenol (m.p. 97–98°), 2:4:6-trichlorophenol (m.p. 70–71°), *m*-5-xylol (m.p. 83–84°), 6-chloro-*o*-cresol (m.p. 91–92°), 3-chloro-*p*-cresol (m.p. 64–65°), ethyl *p*-hydroxybenzoate (m.p. 104–105°), *p*-nitrophenol (m.p. 107–108°), pyrocatechol monoethyl ether (m.p. 93–94°), resorcinol (m.p. 119–120°), *p*-hydroxybenzaldehyde (m.p. 66–67°), α - and β -naphthols (m.p. 106–107°, 89–90°), diethyl-*m*-aminophenol (m.p. 81–82°), *ar*-tetrahydro- β -naphthol (m.p. 120°), *p*-hydroxy-*n*-butylbenzene (m.p. 73–74°), 2:4-dichlorophenol (m.p. 54–55°), and of tri-*o*-tolylphosphine oxide with phenol (m.p. 149–150°) are described. C. HOLLINS.

Manufacture of aromatic nitriles. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,149, 4.12.28).—Aromatic amines are diazotised and treated with aqueous potassium cupridiammonium cyanide prepared by stirring a solution of copper sulphate (1 mol.) into aqueous potassium cyanide (4 mols.) and ammonia (2 mols.). A saving of half the copper salt and half the cyanide used in the ordinary Sandmeyer reaction is effected. The yield of *p*-tolunitrile from *p*-toluidine is 83%; of benzonitrile from aniline, 65%.

C. HOLLINS.

Manufacture of aromatic aminosulphochlorides substituted in the amino-group [*N*-substituted arylaminesulphonyl chlorides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,226, 30.11.28 and 27.7.29).—By the action of chlorosulphonic acid *N*-substituted arylaminesulphonic acids are converted, according to conditions, into corresponding sulphonyl chlorides or into compounds containing additional sulphonyl chloride groups. In particular the *sulphonyl chlorides* of acetyl-*J*-acid and acetyl- γ -acid are obtained for the first time. Other compounds described are: acetyl- α -naphthylamine-4-sulphonyl chloride, m.p. 170°; disulphonyl chloride of acetyl-*H*-acid, m.p. 188–189°; 1-acetamido-8-naphthol-4:6-disulphonyl chloride, m.p. 145–147°; benzoyl- α -naphthylamine-6-sulphonyl chloride, m.p. 185–186°; sulphonyl chlorides from phosphogenated *o*-aminophenol-4-sulphonic acid (m.p. 210–214°), 2:1-aminonaphthol-4-sulphonic acid [m.p. 262–263° (decomp.)], and *H*-acid (m.p. 214–216°); 2-methylbenzoxazole-5-sulphonyl chloride, m.p. 189–190° (decomp.); 2-hydroxyperimidine-5:8-disulphonyl chloride, decomp. 250°; 2-methylperimidine-6-sulphonyl chloride, m.p. 200–204°; carbanilide-4:4'-disulphonyl chloride; methyl-*o*-toluidine-4-sulphonyl chloride, m.p. 99–100°; dimethylaniline-*m*-sulphonyl chloride, m.p. 38°; isomeric dimethyl- β -naphthylaminesulphonyl chlorides, m.p. 86° and 108–110°, respectively, from the 5-sulphonic acid; 4-chlorosulphonylnaphthalene-1:2-diazo oxide, m.p. 131–132°. The products are of use as dye intermediates, for therapeutical purposes, and for combating insect pests.

C. HOLLINS.

(A) Fire-extinguishing and (B) fire-proofing compositions. I. G. FARBENIND. A.-G. (B.P. 302,172 and 326,253, 10.12.28. Ger., 10.12.27).—Alkylated or aral-

kylated aromatic sulphonic acids are added as wetting agents (A) to foam-producing fire-extinguishing materials and (B) to fire-proofing compositions. C. HOLLINS.

Manufacture of sulphonic acids of 1-aminomethylnaphthalene. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,022, 12.3.29).—Sulphonation of 1-aminomethylnaphthalene with 100% sulphuric acid below 50°, or with oleum at 60–70°, yields, respectively, the 2-sulphonic acid or the 2:4-disulphonic acid from which the corresponding aldehydes are obtainable.

C. HOLLINS.

Preparation of camphor by catalytic hydrogenation of borneol or isoborneol. Soc. NOBEL FRANÇ. (F.P. 636,809, 29.6.27).—Formation of camphene and the reverse reaction to borneol and isoborneol are reduced by the use of calcium carbonate as support for the catalyst. *E.g.*, reduced nickel oxide catalyst on powdered chalk gives 90% of camphor at 200–350°.

R. BRIGHTMAN.

Manufacture of nitro-compounds [of 2:4-dihydroxyquinolines]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,398, 2.5.29).—2:4-Dihydroxyquinoline or an *N*-substituted 4-hydroxy-2-quinolone is nitrated with nitric and sulphuric acids. The nitro-derivatives retain the power of coupling with diazo-compounds (nitration in acetic acid gives non-coupling products), and may be reduced and/or sulphonated. The nitrated 4-hydroxy-1-methyl-, -1-ethyl-, and -1-phenyl-2-quinolones melt above 300°.

C. HOLLINS.

Manufacture of condensation products of polynuclear hydrocarbon compounds and olefines [wetting agents]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,500, 11.12.28).—Solid hydrocarbons (or their halogen derivatives) containing at least 3 ring-nuclei are condensed with olefines in presence of aluminium chloride etc. and preferably hydrogen chloride. Examples are: phenanthrene with propylene or cyclohexene, acenaphthene or 5-bromoacenaphthene with propylene, crude anthracene with propylene.

C. HOLLINS.

Organic intermediate compounds and [vat] dyes [of the anthraquinone series]. (SIR) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 326,487, 8.12.28).—1-Chloroanthraquinone-2-carboxylic acid is heated with alkaline polysulphide and ammonia to give anthrathiazolecarboxylic acid, the chloride of which is condensed with amino- or diamino-anthraquinones to form vat dyes, *e.g.*, with α - or β -aminoanthraquinone (greenish-yellow), 1:5-diaminoanthraquinone (yellow), 1:4-diaminoanthraquinone (yellow-brown), 1:5-amino-benzamidoanthraquinone (greenish-yellow), 6-chloro-1-aminoanthraquinone (greenish-yellow), or 2-amino-1-thiolanthraquinone.

C. HOLLINS.

Manufacture of anthanthrone derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,797, 25.8. and 9.10.28).—The nitro-group or -groups in nitrated anthanthrones may be replaced by chlorine or bromine by the action of halogen or of benzoyl chloride or bromide, or may be reduced and the amino-group or -groups may be replaced by halogen, hydroxyl, carboxyl, cyano-, thiol, or thiocyno-groups. The hydroxy- or

carboxy-compounds may be etherified or esterified to give vat dyes. Mono- and di-chloroanthranthrones are orange-yellow vat dyes; the di-iodo- (bluish-red), dimethoxy- (bluish-red), mono- and di-cyano-, methoxy- (red), bromo- (orange-yellow), iodo- (orange), β -chloroethoxy- (red), dibromo- (orange-red), and thiol (brown) derivatives are also described. C. HOLLINS.

Compound from indene and phenols. L. ROSENTHAL and W. KROPP, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,052, 8.4.30. Appl., 22.6.27. Ger., 28.6.26).—See B.P. 297,075; B., 1928, 845.

Manufacture of naphthisatin compounds. J. HALLER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,754,098, 8.4.30. Appl., 29.11.26. Ger., 3.12.25).—See B.P. 286,358; B., 1928, 327.

Distilling apparatus [for glycerin] (U.S.P. 1,743,289). **Carrying out catalytic reactions** (B.P. 310,956). **Catalysts containing free phosphoric acid** (B.P. 326,185).—See I. **Vulcanisation accelerators** (B.P. 326,256).—See XIV. **Light-sensitive surfaces** (B.P. 326,287).—See XXI.

IV.—DYESTUFFS.

PATENTS.

Preparation of *o*-nitroso-[hydr]oxy dyes and their heavy-metal compounds. J. R. GEIGY A.-G. (B.P. 305,648, 30.11.28. Ger., 9.2.28).—Pyridacetyl or other basic derivatives of aminonaphthols are nitrosated and converted into heavy-metal salts which are soluble in water. Pyridylacetyl-2:7-aminonaphthol, $C_5H_5N(Cl) \cdot CH_2 \cdot CO \cdot NH \cdot C_{10}H_6 \cdot OH$, after nitrosation gives a copper salt which dyes brown-yellow. Dimethyl-aminoacetyl-2:7-aminonaphthol (yellowish-green iron salt), 7-hydroxy- β -naphthyltrimethylammonium chloride (blue-green iron salt), pyridylacetyl-7-amino- α -naphthol (green iron salt), pyridylacetyl-3-amino- α -naphthol (green iron salt), and pyridylacetyl-5-amino- α -naphthol (red-brown copper salt) are also nitrosated and converted into dyes. C. HOLLINS.

Manufacture of [vat] dyes [of the benzanthrone series]. I. G. FARBENIND. A.-G. (B.P. 308,651, 25.3.29. Ger., 23.3.28).—Benzanthrone-3:4-dicarboxylic anhydride (or a derivative) is condensed with an *o*-diamine to yield iminazole compounds. With *o*-phenylenediamine, 4-chloro-*o*-phenylenediamine, and 3:4-diaminophenetole orange vat dyes are obtained; with 1:2-naphthylenediamine a deep brown. C. HOLLINS.

Manufacture of vat dyes of the benzanthrone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,148, 4.12.28 and 3.9.29).—A mono- or di-halogenated benzanthrone, or 3:3'- or 4:4'-dibenzanthronyl, or 3:3'- or 4:4'-dibenzanthronyl sulphide is condensed with 1-amino-2-aldehydoanthraquinone (or an anil etc. thereof); or the corresponding mono- or di-amino-derivative is condensed with 1-chloro-2-aldehydoanthraquinone. Preferably a diluent (nitrobenzene), and an acid-binding agent (sodium acetate), and a catalyst (copper oxide) are used. The resulting acridines or acridones are brown vat dyes. 1-Amino-2-aldehydoanthraquinone is thus condensed with 3-bromo-, 11-chloro-, 9-chloro-, 4:9-dichloro-, 3:9-dibromo-, 3-bromo-

9-nitro-, and 3-bromo-9-benzenesulphonamido-benzanthrones, with dibrominated 3:3'- and 4:4'-dibenzanthronyls, and with dibromo-3:3'-dibenzanthronyl sulphide; 1-chloro-2-aldehydoanthraquinone is condensed with 9-amino- and 4:9-diamino-benzanthrones.

C. HOLLINS.

Manufacture of nitrogenous vat dyes [of the dibenzanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,217, 7.9.28. Addn. to B.P. 314,593; B., 1929, 710).—Grey vat dyes are obtained by heating nitrodibenzanthrones or nitrosodibenzanthrones alone or in nitrobenzene in the absence of alkaline condensing agents. C. HOLLINS.

Manufacture of vat dyes containing nitrogen [from pyrazolanthrone]benzanthrone]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,268, 19.12.28).—The condensation products from pyrazolanthrone and 4-chlorobenzanthrones carrying a 3-nitro- or 3-chloro-substituent are converted by alkaline fusion (e.g., alcoholic alkali at 110°) into violet vat dyes. Examples are the condensation products from pyrazolanthrone and 4-chloro-3-nitrobenzanthrone, m.p. 268°, or 3:4:9-trichlorobenzanthrone, and from 5-benzamidopyrazolanthrone and 4-chloro-3-nitrobenzanthrone. The products may be halogenated.

C. HOLLINS.

Manufacture of vat dyes [of the pyranthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,263, 15.12.28).—Reddish-brown vat dyes are obtained by oxidising di- or tetra-bromopyranthrone, e.g., with manganese dioxide or potassium iodate, in sulphuric acid. C. HOLLINS.

Manufacture of acid wool dyes [of the anthraquinone series]. I. G. FARBENIND. A.-G. (B.P. 302,928, 21.12.28. Ger., 23.12.27. Addn. to B.P. 282,409; B., 1928, 442).—A 4-halogeno-1-aminoanthraquinone-2-sulphonic acid is condensed with a *m*-aminoacetalkylanilide to give blue acid dyes.

C. HOLLINS.

Manufacture of monoazo [chrome wool] dyes. I. G. FARBENIND. A.-G. (B.P. 301,423, 26.11.28. Ger., 29.11.27).—Clear yellowish chrome-greens for wool are obtained by coupling 2:3-aminonaphthoic acid with a diazotised *o*-aminophenolsulphonic acid which may contain halogen but no nitro-group, e.g., 4-chloro-*o*-aminophenol-6-sulphonic acid. C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 302,601, 17.12.28. Ger., 17.12.27).—An arylamide of 2:3-hydroxynaphthoic acid is coupled in substance or on the fibre with a diazotised 3:6- or 5:6-dihalogenated *o*-toluidine. C. HOLLINS.

Manufacture of dyes [gallocyanine derivatives]. DURAND & HUGUENIN A.-G. (B.P. 301,329, 26.11.28. Ger., 26.11.27).—Readily dischargeable gallocyanines are prepared from *m*-substituted alkylanilines. Examples are dyes from: gallamide and nitrosodiethyl-*m*-toluidine (converted into anilide and sulphonated for chrome-printing greenish-blue); gallamide and *m*-chloronitrosodiethyl- or -dimethyl-aniline (anilide, sulphonated, redder blue); gallamide and nitrosoethyl-*m*-toluidine

(anilide, sulphonated); gallic acid and nitrosomethyl-*m*-toluidine (decarboxylated and reduced, violet).

C. HOLLINS.

Black sulphur dyes. R. VIDAL (F.P. 633,395, 14.8.26).—Nitroso-*o*- or -*m*-cresol is added to a solution of sodium sulphide and after cessation of the reaction 2 : 4-dinitrophenol and sodium sulphide are introduced. Sulphur is then added at 85°, the mixture being thereafter maintained at boiling temperature for 16–24 hrs. and the dye precipitated with the aid of a current of air. Liquid dyes of high concentration are obtained by mixing the moist precipitate with sodium sulphide and concentrating to a 50% content of dye. D. F. TWISS.

[Manufacture of] azo dyes. H. WAGNER, E. HOFFA, E. RUNNE, E. THOMA, and H. HEYNA, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,754,070, 8.4.30. Appl., 5.7.27. Ger., 9.7.26).—See B.P. 274,128; B., 1929, 351.

Manufacture of azo dyes. E. HOFFA and E. THOMA, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,745,676, 4.2.30. Appl., 24.2.28. Ger., 2.3.27).—See B.P. 313,865; B., 1929, 711.

[Manufacture of azo] dyes containing chromium. H. KAMMERER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,753,120, 1.4.30. Appl., 13.4.28. Ger., 14.5.27).—See B.P. 302,709; B., 1929, 165.

Anthraquinone dyes (B.P. 326,487). **Anthanthrone derivatives** (B.P. 325,797).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Commercial possibilities of the common milkweed. F. GERHARDT (Ind. Eng. Chem., 1930, 22, 160–163).—In an experimental crop from milkweed (*Asclepias syriaca*), 30 bushels of seed, 280 lb. of seed fibre, and 1 ton of air-dry stems were obtained per acre. The seed fibre is too brittle for use in the textile industry, but might be a substitute for kapok. The seed contains 21% of semi-drying oil and 47% of the remaining meal is crude protein. The dry stem contains 10% of bast fibre, which might be used in the textile industry. In view of the α -cellulose content (36.5%), the stems might be used for pulps for making paper or board.

E. B. HUGHES.

Silk—a field for research. E. M. SHELTON and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 387–390).—The present state of knowledge of the chemistry of silk is discussed, and some valuable lines of investigation are indicated.

F. R. ENNOS.

Calcium chloride and flue gas for waste disposal of kier liquors from textile plants. R. L. COPSON and H. A. CURTIS (Chem. Met. Eng., 1930, 37, 167–169).—The waste liquor resulting from the boiling of unbleached cotton with dilute caustic soda being highly charged with organic matter is an undesirable effluent to run into streams. The most feasible method of treatment is as follows. The liquor is run down a carbonating tower against a current of flue gases. The bicarbonate content is then neutralised with the requisite amount of untreated liquor, and a slight excess of calcium chloride is added. The solids are settled and the clarified liquor is run away. About 75% of the colour and organic matter are so removed as insoluble

calcium salts of organic acids. Variation in temperature of the liquor has little influence on the results.

C. IRWIN.

Evaluation of unbleached chemical wood pulps. G. HALL (Proc. Tech. Sect. Papermakers' Assoc., 1929, 10, 85–138).—The unbeaten strength test is not considered suitable for unbleached pulps, particularly sulphate pulps, owing to the difference in response to beating. The strongest pulp on unbeaten test is not necessarily the strongest after beating. Unbeaten tests depend more on felting capacity than on the ultimate strength of the fibres. For beating under standard conditions, the Abbé pebble-mill and the Lampén ball-mill are compared. The newer type of Lampén mill is recommended. After beating the pulps are made into circular sheets, 10 in. in diam., couched on to ferro-type plates, pressed at 214 lb./in.², dried at 50–60°, and tested for strength. The effect of varying the pressure on the waterleaf is discussed. Increasing pressure gives rise to increased tensile, folding, and bursting strengths, and lower tearing strength. Difference in strength of machine-made papers and laboratory-made sheets is attributed to the high pressures exerted by the press-rolls of the paper machine, the presence of fragmentary fibres in the back-water, and the more even distribution of the fibres in the laboratory method. Conditions may be chosen so that the different factors cancel out to give comparable results. Pulps beaten in a Lampén mill need only be pressed at 50 lb./in.² to give sheets of equal strength to machine-made paper from the same pulp. The lignin content of pulps is a measure of the degree of cooking, and determines the bleachability. The amount of rosin present is independent of the degree of cooking, but varies according to the process used. The cooking degree is determined rapidly by permanganate absorption tests. α -Cellulose tests are of little value for unbleached pulps. For a given bleachability, sulphite pulps hydrate more readily than do sulphate pulps; similarly, sulphite pulps cooked with indirect steam have the higher bursting and tensile strengths, but the lower folding and tearing strengths. Strength increases with lignin content, tearing strength showing a maximum at 5% of lignin. Porosity, or air-permeability, is a better indication of the condition of the fibres in a sheet than "beating degree." The same amount of beating produces a more porous sulphate sheet than sulphite sheet. The porosity of sheets made from sulphate pulps is proportional to the cooking degree, the harder the pulp the more porous is the sheet. Absorbency is proportional to cooking degree for both kinds of pulp.

T. T. PORTS.

Wood. II. Water-soluble polysaccharide of western larch wood. L. E. WISE and F. C. PETERSON (Ind. Eng. Chem., 1930, 22, 362–365; cf. Harlow and Wise, B., 1928, 636).—The presence of water-soluble α -galactan in western larch wood (*Larix occidentalis*) described by Schorger and Smith (B., 1916, 750) is confirmed. On complete hydrolysis it yields 11.95% of arabinose and 84.6% of galactose, no other hexoses being detected, and it appears to be an arabogalactan containing arabinose and galactose molecules in the ratio 1 : 6; the arabinose obtained on hydrolysis

accounts quantitatively for the furfuraldehyde yield on distilling with acid.

F. R. ENNOS.

Rayon as a paper-making material. M. B. SHAW and G. W. BICKING (Bur. Stand. J. Res., 1930, 4, 203—211).—It is shown that rayon is valueless in rag stock used for making high-grade paper, and may actually be detrimental, since the rayon fibres break into short lengths when wetted, without the fibrillation and fraying necessary for good felting.

C. J. SMITHELLS.

Rice husks. DE MONGEOT.—See XIX.

PATENTS.

Lubricating, conditioning, and laying the fly of raw cotton. G. J. EDWARDS. From E. F. HOUGHTON & Co. (B.P. 325,938, 5.1.29).—The raw stock is treated with a mixture containing at least 60% of mineral oil, 5% of soap, 2% of glycerin, 2% of sulphonated vegetable or animal oil, and the balance non-mineral oil, the whole being emulsified with not less than an equal quantity of water.

F. R. ENNOS.

Impregnation of textile filaments with rubber. C. H. GRAY (B.P. 325,916, 19.12.28).—The filaments are passed through a dispersion or solution of rubber and thence between a pair of rotating rubber-covered rollers.

F. R. ENNOS.

Means for testing the dryness of fibrous material. C. Q. IVES, Assr. to BROWN Co. (U.S.P. 1,744,120, 21.1.30. Appl., 12.12.24).—The relative dryness of similar materials is determined by comparing the electric resistance between a pair of pointed metallic contacts, connected to a source of electric current, when inserted in the materials to a fixed depth and at a suitable distance apart.

F. R. ENNOS.

Manufacture of cellulose esters. H. DREYFUS (B.P. 325,822, 21.11.28).—Cellulosic material is pre-treated with an organic base, e.g., methylpiperidine, above 100° and afterwards esterified with a carboxylic acid anhydride in the presence of the organic base above 100°.

F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 303,006, 6.12.28. Ger., 24.12.27).—Cellulose is treated for 1 hr. below 60° with air saturated with nitric acid vapour, so that its fibrous structure is preserved; the product is subsequently esterified in any desired manner.

F. R. ENNOS.

Manufacture of esters of cellulose or other carbohydrates. I. G. FARBENIND. A.-G. (B.P. 301,036, 22.11.28. Ger., 23.11.27).—Esterification of cellulose with a carboxylic acid anhydride and/or acid halide is carried out in presence of liquid sulphur dioxide, with or without the usual catalysts.

F. R. ENNOS.

Manufacture of acetylcellulose. I. G. FARBENIND. A.-G. (B.P. 301,755, 3.12.28. Ger., 2.12.27).—By incorporating a catalyst comprising zinc chloride, sulphuric acid, and hydrochloric acid or a substance yielding hydrochloric acid (acetyl chloride or tetrachloroethane), an ester soluble in acetone is produced.

F. R. ENNOS.

Manufacture of chlorinated organic acid esters of cellulose. I. G. FARBENIND. A.-G. (B.P. 306,132, 7.2.29. Ger., 17.2.28).—A cellulose ester of a saturated

organic acid is chlorinated, e.g., with phosphorus pentachloride, under suitable conditions according to the chlorine content desired, while suspended in an indifferent medium (tetrachloroethane); the product is soluble in organic solvents and stable towards acids.

F. R. ENNOS.

Manufacture of rayon of increased whiteness from viscose. DU PONT RAYON Co., Assees. of C. HENNINGSEN (B.P. 301,307, 30.10.28. U.S., 26.11.27).—Sufficient bluish colouring matter or dye is added to the viscose at any stage of its manufacture prior to coagulation and spinning.

F. R. ENNOS.

Manufacture of artificial silk. P. VANDEMAELE (B.P. 327,220, 22.1.29).—Artificial filaments produced from viscose etc. are prevented from stretching or slipping while passing over rotary spinning pots by an arrangement of three godet wheels.

F. R. ENNOS.

Production of artificial [silk] filaments. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 326,461, 28.9.28).—A fluid stream is suitably directed on to a number of artificial filaments, extruded from spinning orifices into a medium rich in the solvent employed, so that they converge together while in a tacky condition and unite to form one filament of large cross-section.

F. R. ENNOS.

Manufacture of artificial threads and webs of the same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,857, 27.8.28).—The solution of cellulose ether or ester in a volatile solvent is passed through nozzles, the straight bore of which is at least 10 times the internal diameter, and thence directly through a liquid containing an organic solvent and a coagulant for the cellulose derivative, e.g., aqueous acetone, so that the threads are only slightly coagulated superficially. The threads, which may be stretched during spinning if desired, are then solidified in the usual air-spinning space.

F. R. ENNOS.

Manufacture of artificial products from wood. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,502, 12.12.28).—Wood, straw, hay, rushes, etc. are treated with an organic acid halide in the presence of a basic reagent such as a tertiary amine, whereby a mouldable product softening below its decomposition point is obtained.

F. R. ENNOS.

Cellulose solutions and their applications. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 326,579, 22.12.28).—Cellulose is dissolved in anhydrous or aqueous methanesulphonic acid at 10–20°, and precipitated in any desired form by means of water.

F. R. ENNOS.

[Cellulose binding agent for] joining wood, metal, glass, etc. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS, Assees. of W. PLINATUS (B.P. 302,324, 14.12.28. Ger., 14.12.27).—A cellulose ester is stirred with an aqueous emulsion of a high-boiling solvent, e.g., an ester such as glyceryl benzoate, and the mixture is heated until the water evaporates; the powdery material obtained is pressed and rolled out to a thin adhesive layer, which is applied between the surfaces to be joined, the whole being subjected to gentle

pressure and increased temperature and allowed to cool under pressure.

F. R. ENNOS.

Fireproofing of cellulose. F. S. VIVAS, Assr. to INTERNAT. FIREPROOF PRODUCTS CORP. (U.S.P. 1,738,976, 10.12.29. Appl., 11.3.27).—Cellulosic materials are impregnated with a warm solution of boric acid, borax, ammonium sulphate, and sodium tungstate, and, after partially drying, are immersed in a solution of calcium chloride and dried.

F. R. ENNOS.

Production of paper. D. K. PATILLO and J. H. MACMAHON, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,737,330, 26.11.29. Appl., 23.12.27).—The stock is sterilised with chlorine while at a point of high dilution, subjected to a coagulating treatment, and formed into paper.

F. R. ENNOS.

Smoothing of paper. I. G. FARBENIND. A.-G. (B.P. 310,340, 23.4.29. Ger., 23.4.28).—After spreading with a melted hydrocarbon (paraffin wax), wax, resin, or a mixture of these, the paper is passed over a support heated above the m.p. of the smoothing agent, while removing the excess by means of a scraper.

F. R. ENNOS.

Production of [moiré-patterned fabrics] from organic derivatives of cellulose. C. DREYFUS (U.S.P. 1,754,164, 8.4.30. Appl., 4.11.26).—See B.P. 280,195; B., 1929, 279.

Coating of paper-pulp vessels for protection thereof against the action of moisture, fat, soap, etc. F. FRANK (U.S.P. 1,753,565, 8.4.30. Appl., 26.10.25. Ger., 27.10.24).—See B.P. 241,876; B., 1926, 783.

Combined acid channel and thread guide for artificial silk threads. O. (FRH.) VON KOHORN, and A. PERL (B.P. 316,253, 27.5.29. Austr., 26.7.28).

Spinning of artificial silk threads [on to a "ring" or "traveller" spindle]. C. C. TYRER (B.P. 326,452, 11.12.28).

Machine for production of wood pulp for manufacture of paper. MAGNET-WERK G.M.B.H. EISENACH SPEZIALFABR. F. ELEKTROMAGNET-APPARATE (B.P. 304,233 and Addn. B.P. 311,386 and 326,708, [A—C] 9.1.29. Ger., [A] 17.1.28, [B] 6.2.28).

Attrition mills (U.S.P. 1,744,226 and 1,744,235). **Pressure regulator for pumped liquids** (B.P. 309,086).—See I. **Fuel** (U.S.P. 1,743,985).—See II. **Hydrogenation of polyhydroxy-compounds** (B.P. 299,373).—See III. **Laminated glass** (B.P. 326,450).—See VIII. **Lead-coated paper** (B.P. 326,792).—See X. **Wool fat** (B.P. 303,890 and F.P. 569,624).—See XII. **Rubber-fibre articles** (U.S.P. 1,737,133). **Rubber goods** (B.P. 326,782). **Rubbering of fabrics** (G.P. 461,134).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of exposure of wool before dyeing. W. VON BERGEN (The Melliand, 1929, 1, 1084—1093).—Wool containing alkali is more rapidly affected by sunlight than wool which is slightly acid. The behaviour on dyeing of the exposed wool depends on the colour used.

CHEMICAL ABSTRACTS.

Action of light on cotton dyed with certain vat dyes. F. SCHOLEFIELD and E. H. GOODYEAR (The Melliand, 1929, 1, 1389—1393).—Cotton dyed with indanthrene-yellow G or certain other yellow or orange vat dyes is more susceptible to tendering by light than that dyed with blue or violet vat dyes. The effect, which is attributed to the formation of hydrogen peroxide, is increased by alkali.

CHEMICAL ABSTRACTS.

PATENTS.

Increasing the wetting and penetrating capacity of liquors employed in the textile industry. H. T. BÖHME A.-G. (B.P. 290,256, 10.5.28. Ger., 11.5.27).—Sulphonated fats and oils prepared as described in B.P. 284,280 (B., 1928, 943) are more suitable than is Turkey-red oil as wetting-out agents for addition to liquids which are acid or alkaline or which may contain soluble salts, since they are stable under such conditions and produce a greater corresponding degree of penetration. [Stat. ref.]

A. J. HALL.

Immunisation of [cellulose] threads [in wound packages]. CHEM. WORKS, FORMERLY SANDOZ (B.P. 325,961, 26.1.29. U.S., 22.10.28).—Cotton or viscose wound yarn is treated with alkali and esterified in the usual manner without unwinding. Preferably the alkalis copers are esterified under pressure.

C. HOLLINS.

Bleaching of vegetable fibres. R. VIDAL (F.P. 634,962, 24.9.26).—A hypochlorite solution mixed with a soap obtained by the process of F.P. 584,738 (cf. B.P. 250,108; B., 1926, 553) is used.

R. BRIGHTMAN.

Bleaching of materials for the manufacture of hats. H. GOLDBARBEITER (B.P. 293,828, 27.6.28. Austr., 13.7.27. Addn. to B.P. 291,743; B., 1930, 54).—The process described in the prior patent is suitable for the bleaching of hats, and particularly of hare's fur, which have been previously mordanted with the salts of heavy metals such as mercury, zinc, and tin.

A. J. HALL.

Treatment [washing, bleaching, etc.] of skeins of artificial silk. J. P. BEMBERG A.-G. (B.P. 312,034 and Addn. B.P. 313,945, [A], 17.9.28, [B] 22.9.28. Ger., [A] 19.5.28, [B] 20.6.28).—(A) Cross-lacing of skeins of artificial silk previous to their treatment with liquids is avoided and entangling prevented by laying the cross-reed skeins in separate layers closely over one another and then exposing them to the wet treatment. The silk yarn may or may not be in a twisted condition, and complete permeation of the skeins by the liquid is obtained. (B) Cross-lacing may also be avoided if the skeins are treated in a stationary condition.

A. J. HALL.

Reserving of wool in dyeing with substantive dyes. I. G. FARBENIND. A.-G. (B.P. 304,742, 25.1.29. Ger., 25.1.28).—In the dyeing of mixed fabrics containing wool with direct cotton dyes the wool is reserved by addition to the bath of a small amount (about 5%) of a water-soluble salt of a sulphonic acid obtained (cf. B.P. 314,588; B., 1929, 728) by condensing a natural resin or mixture of resins with an aralkyl halide or substitution product and then sulphonating the product.

A. J. HALL.

Production of fast tints on chrome-mordanted fibres. SOC. CHEM. IND. IN BASLE (B.P. 303,384, 31.12.28. Switz., 30.12.27).—Improved fastness to rubbing, light, fulling, and potting results when fully prechromed azo dyes are dyed on chrome-mordanted wool or cotton. Examples are: 5-nitro-*o*-aminophenol \rightarrow Brönner or phenyl-J-acid; 4-nitro-*o*-aminophenol-6-sulphonic acid \rightarrow acetoacetanilide. C. HOLLINS.

Dyeing of fabric. SPECTRUM DYES PROPRIETARY LTD. (B.P. 305,922, 21.8.28. Austral., 11.2.28).—Coloured textile materials are obtained by saturating a dyed fabric with a solution obtained by treating sodium bisulphite with zinc, and then further treating it until of the desired shade with a similar solution after the addition of the product obtained by treating coal-tar naphtha with sulphuric acid. A. J. HALL.

Treatment [coloration] of fabrics. BRIT. CELANESE, LTD. (B.P. 316,983, 11.8.28. U.S., 13.8.27. Cf. B.P. 295,582; B., 1930, 54).—The process of spraying cellulose acetate materials with dyes in organic solvents described in B.P. 314,208 (B., 1929, 716) is suitable for use with textile materials containing other derivatives of cellulose. A. J. HALL.

Production of dyeings [ice colours from 4-hydroxy- α -naphthyl ketones.] SOC. CHEM. IND. IN BASLE (B.P. 303,179, 29.12.28. Switz., 29.12.27).—Fabrics padded with 4-acylated α -naphthols may be steamed before development; the process is thus suitable for production of variegated effects on mixed cotton-acetate silk goods, and for prints. Phenyl 4-hydroxy- α -naphthyl ketone and aminoazobenzene are padded with alkali on cotton-acetate silk fabric, which is then steamed to fix the aminoazobenzene and developed with diazotised 4-chloro-*o*-aminophenyl phenyl ether to give red (acetate silk) and yellow (cotton) effects. C. HOLLINS.

Dyeing of [cellulose] acetate silk. I. G. FARBENIND. A.-G. (B.P. 320,422, 10.7.28. Addn. to B.P. 293,766; B., 1930, 185).—Processes additional to those described in the prior patent include dyeing with Benzo Brown G, Rhoduline Orange NO, New Methylene Blue F, Bismarck Brown FR extra, Brilliant Rhoduline Violet R, Rhoduline Heliotrope 3B, Methylene Blue BB, and Safranine FF extra in the presence of 3% of hexahydroaniline. A. J. HALL.

Dyeing of esters and ethers of cellulose or of its transformation products. I. G. FARBENIND. A.-G. (B.P. 304,739, 25.1.29. Ger., 25.1.28).—Greenish-yellow shades on acetate silk etc. are obtained by the application of 4-amino-1:8-naphthalimide and its *N*-alkyl-, -aryl, or -aralkyl derivatives, *e.g.*, the *N*-ethyl or *N*-*o*-tolyl compound. C. HOLLINS.

Dyeing and printing. SOC. ALSACIENNE DE PROD. CHIM. (F.P. 636,665, 21.10.26).—Aqueous solutions of the sulphurisation product of phenols are mixed with the alkali salts of acid dyes and fixed with acid or acid salts. *E.g.*, a solution of alkali-blue in aqueous soda is mixed with a phenol sulphurisation product obtained according to F.P. 577,653 (cf. B.P. 215,012; B., 1925, 541), and thickened with neutral starch and tragacanth. The fabric is printed, dried, and the dye is then fixed with

dilute acetic, hydrochloric, or sulphuric acid, or with zinc sulphate or aluminium chloride solution. Sulphonated basic dyes can be used. R. BRIGHTMAN.

Printing of textile fabrics [resists against synthetic mordants]. IMPERIAL CHEM. INDUSTRIES, LTD., and L. SMITH (B.P. 325,935, 3.1.29).—Zinc chloride or other metal halides (except alkali halides) are applied as resists against sulphurised phenols. C. HOLLINS.

Preparations for finishing textile fabrics. H. T. BÖHME A.-G. (B.P. 291,094, 12.5.28. Ger., 27.5.27).—Epsom salt finishes on textile fabrics are produced by using the products obtained by sulphonating fats and oils in the presence of organic acid anhydrides or chlorides or of strong anhydrous organic acids (cf. B.P. 274,104; B., 1928, 613). Such sulphonated products are preferred to Turkey-red oil, since they hydrolyse less readily and acid-tendering of the fabric during calendering or similar treatment is thus avoided (cf. also B.P. 293,746; B., 1929, 640). A. J. HALL.

Stabilisation of bleaching liquors. F. DRAISBACH, Assr. to J. A. BENCKISER CHEM. FABR. (U.S.P. 1,754,163, 8.4.30. Appl., 16.8.26. Ger., 14.1.26).—See B.P. 265,417; B., 1927, 252.

Washing, dyeing, mixing, or other machines in which the materials treated are subject to kneading or squeezing action. E. FREUND (B.P. 302,316, 13.12.28. Austr., 14.12.27).

Making pattern collections of textile print and dye works. SOC. FINANCIÈRE & COMMERCIALE SOC. ANON. (B.P. 326,528 and Addn. B.P. 318,600, [A] 11.10.28, [B] 29.10.28. Ger., [A] 9.7.28, [B] 7.9.28).

Purifying etc. apparatus [for dry-cleaners' solvent] (U.S.P. 1,744,747).—See I. Restoring used gasoline (U.S.P. 1,745,108).—See II. Wetting agents (B.P. 326,500).—See III. Increasing the whiteness of rayon (B.P. 301,307).—See V. Bleaching of fatty acids (U.S.P. 1,740,012). Sulphonation product (U.S.P. 1,745,221). Bleaching of beeswax (U.S.P. 1,739,796).—See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Preparation of citric acid. M. CATULLO (Boll. Chim. Farm., 1930, 69, 234—238).—The manufacture of citric acid from crude calcium citrate in Italy is described. T. H. POPE.

Ammonium sulphate from gypsum. L. WASILEWSKI, A. KACZOROWSKI, and S. ZABICKI (Przemysl Chem., 1930, 14, 150—158).—The following apparatus for the continuous preparation on a semi-technical scale of ammonium sulphate from gypsum is described. A suspension of gypsum in ammonia is passed through a series of horizontal tubes containing stirrers fixed to a central axis. A temperature gradient is maintained from 14° at the point where the substrates enter to 120° at the point where the products leave the apparatus. The pressure maintained within the system amounts to 2—3 atm. Solutions containing 40% of ammonium sulphate are obtained, using 12 pts. of 18% ammonia to 50 pts. of gypsum, with a throughput

of about 800 kg./24 hrs. Of the sulphuric acid present as gypsum 96.5—98.5% is thus recovered, with a loss of ammonia not exceeding 0.2%.

R. TRUSZKOWSKI.

Preparation of pure monoammonium and monocalcium phosphate from bone phosphoric acid. H. J. BRAUN (Metallbörse, 1929, 19, 1912—1913; Chem. Zentr., 1929, ii, 2927).—Treatment with ammonia removes the impurities; evaporation affords ammonium dihydrogen phosphate. Monocalcium phosphate, however, does not crystallise from solution unless a large excess of acid is present. A. A. ELDRIDGE.

Removal of ions from solutions of calcium dihydrogen phosphate by treatment with hydrous gels of alumina, silica, and their mixtures. J. C. GHOSH and P. B. BHATTACHARYYA (Soil Sci., 1930, 29, 311—322).—Silica gel removes the ions Ca^{++} and $\text{H}_2\text{PO}_4^{--}$ from calcium dihydrogen phosphate solutions in equivalent proportions. The Ca^{++} ions are assumed to be absorbed by valency forces and the resulting electropositive gel removes the $\text{H}_2\text{PO}_4^{--}$ ions by electrical attraction. The absorption of these two ions by alumina gel is much greater than in the case of silica, and the p_{H} value of the gel is increased considerably. This is explained by chemical interaction. Results of the treatment of calcium phosphate solutions with mixtures of alumina and silica gels are recorded and discussed. A. G. POLLARD.

Treatment of sylvite above 100°. W. FROELICH and E. RITTER (Mitt. Kali-Forsch.-Anstalt, 1929, 67—89; Chem. Zentr., 1929, ii, 2485).—A study of the separation of salts on cooling saturated solutions of sodium and potassium chlorides and its application to the working-up of sylvite. The maximum solubility of sodium chloride in saturated potassium chloride solutions is at 80°. At solution temperatures below 150° potassium chloride can be obtained free from sodium chloride. A. A. ELDRIDGE.

Rotatory furnace for extraction of aluminium salts from clay. L. WASILEWSKI and J. Z. ZALESKI (Przemysl Chem., 1930, 14, 181—188).—A rotatory furnace for the continuous preparation by a modification of Rimman's method is described. A mixture consisting of 4 pts. of ammonium sulphate to 1 pt. of alumina present in the clay is passed through a furnace the distal three fourths of which is maintained at 400°; where the time of passage of the reacting mass is 30 hrs. 88% yields of alum are obtained, with only very small losses of ammonia. Higher temperatures cause decomposition of ammonium sulphate, but it is probable that prolongation of the duration of reaction would give practically theoretical yields. The emerging product (at about 95°) is at once placed in water, in which the alum dissolves, leaving a residue of silica, suitable for the preparation of water-glass. The extract, on keeping, deposits fairly pure alum, the iron content of which is, after one recrystallisation, 0.002% FeO; this degree of purity suffices for the preparation of pure aluminium. R. TRUSZKOWSKI.

Extraction of copper from residues of copper sulphate manufacture. C. FICAI (Rass. Min. Met. Ital., 1929, 69, 4—7; Chem. Zentr., 1929, ii, 2931).—A

method depending on the roasting of the residues and extraction of the cupric oxide with dilute sulphuric acid is described. Iron and arsenic are removed from the solution before crystallisation; the noble metals remain in fairly pure form in the residue. A. A. ELDRIDGE.

Determination of small amounts of silver in pyrolusite. A. KUNDERT (Chemist-Analyst, 1930, 19, No. 1, 11).—The silver is extracted by repeated treatment of the ore with concentrated hydrochloric acid; the solution is neutralised with ammonia, treated with hydrogen sulphide, the precipitate washed with cold 10% sulphuric acid, ignited, and the ash treated with nitric acid. The silver is precipitated with hydrochloric acid, freed from lead by means of ammonia solution, and dissolved in potassium cyanide solution, this solution then being electrolysed. CHEMICAL ABSTRACTS.

Solubility of salts in water and in lyes at temperatures below and above 100°. W. FROELICH (Mitt. Kali-Forsch.-Anstalt, 1929, 37—66; Chem. Zentr., 1929, ii, 2485).—The solubilities of potassium chloride, sodium chloride, magnesium sulphate, and of sodium and potassium chlorides in water, of magnesium sulphate in aqueous magnesium chloride solutions, of sodium and potassium chlorides in technical sylvite extraction liquor, and of that mixture and carnallite in van't Hoff's liquor Q20 have been determined. A. A. ELDRIDGE.

Absorption of carbon monoxide by cuprous ammonium salts. W. GUMP and I. ERNST (Ind. Eng. Chem., 1930, 22, 382—384).—A large number of absorbents for the extraction of carbon monoxide from water-gas were examined from the point of view of the commercial preparation of hydrogen or carbon monoxide. Various mixtures containing cuprous ammonium salts were studied and found to be unsatisfactory, and then a large number of organic acids and phenols as substitutes for the carbonate in cuprous oxide-ammonium carbonate-ammonia mixtures were examined. Cuprous ammonium lactate was found to be the most satisfactory absorbent, and the mixture with ammonia did not attack iron and was stable. The absorptive power remained the same after 20 regenerations, in which the solution was heated to 80° to expel carbon monoxide. A solution containing 13 g. of cuprous oxide, 25 g. of lactic acid, 85 c.c. of ammonia solution (d 0.910), and 100 c.c. of water absorbed 15 vols. of the gas at 0° or 0.99 mol./mol. Cu_2O . Experiments on a semi-works' scale showed that carbon monoxide of 97—99% purity could be produced. No trouble was experienced in an iron plant when run for several weeks, and no deposition of copper took place. The same method was then used successfully in a large-scale plant. H. INGLESON.

Reliability of the Reich-Raschig method of determining sulphur dioxide. H. E. WOISIN (Z. angew. Chem., 1930, 43, 293—294).—The reliability of Raschig's modification of Reich's method of determining sulphur dioxide, in which the error caused by the presence of oxides of nitrogen, as in sulphuric acid chamber gases, is claimed to be suppressed by the use of sodium acetate, was examined. It was found that if oxides of nitrogen were absent, identical results were obtained with or without sodium acetate, and whether

the gas was drawn through the iodine slowly or quickly. With concentrations of 0.7% N_2O_3 , however, results by Raschig's method were seriously below the truth, although not so low as in absence of sodium acetate, and the slower the gas current the higher was the sulphur dioxide figure obtained. For Gay Lussac exit gases the values were particularly faulty and this test should only be used with great caution in calculating the output of units in a sulphuric acid plant. C. IRWIN.

Volatilisation of phosphorus from phosphate rock. II. Volatilisation of phosphorus and potash in a blast furnace. III. Calculations of performance of a blast furnace for volatilisation of phosphorus and potash. R. D. PIKE (Ind. Eng. Chem., 1930, 22, 344–354).—II. Two experimental runs of a blast furnace indicated that most of the reduction of phosphate was effected in the bosh of the furnace, any reduction by carbon monoxide in the shaft being of minor importance. Cold oxygenated blast containing 40–45% O proved most suitable, though a hot blast sufficiently oxygenated to give the same theoretical temperature of combustion would be preferable. A free-running slag closely resembling that of an iron blast-furnace should be tapped at 1450–1500° and from smaller-scale trials extractions of 97% P_2O_5 and 92% K_2O can be reasonably expected with continuous smooth operation. As the only function of the shaft is to preheat the charge, a squat type of furnace, probably not taller than 20–25 ft., is indicated.

III. The design of a suitable furnace would be similar to but lower than a standard, iron, blast furnace except that the blast would contain 30% O. The gases would then pass through a waste-heat boiler to burn phosphorus to oxide and then to a Cottrell precipitator in which the mixture of phosphorus pentoxide and potassium phosphate would be collected in liquid form. The slag must be fluid and basic to volatilise potash, which is apparently eliminated by the direct replacement by lime without the intervention of carbon. When supplies of coke and potash-bearing flux are favourable, the blast-furnace process appears to offer distinct economic advantages over the present sulphuric acid method for making phosphoric acid. C. A. KING.

Pneumatic transport. NEU. Thermal data in drying-tower design. NEWMAN.—See I. Rare elements in clays. FIOLETOV.—See VIII. Electro-reduction of silver chloride. YAGI.—See X. Preparation of basic copper sulphate. HOLLAND and others.—See XVI.

PATENTS.

Separation of mixtures of sulphuric acid and nitric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,977, 28.2.29).—Alkali sulphate or nitrate is added in quantity sufficient to form alkali hydrogen sulphate with the sulphuric acid. The separation of the alkali hydrogen sulphate is preferably assisted by cooling, but in order to prevent separation of alkali nitrate as well it is frequently necessary to remove part of the water from the mixture by evaporation.

S. K. TWEEDY.

Production of hydrocyanic acid. T. S. WHEELER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 325,860, 3.9.28).—Hydrocarbons, such as methane or coal gas,

are heated to at least 800° before or after admixture with (preferably excess of) nitrogen or ammonia, and the hot mixture is subjected to an arc or spark discharge. The gas mixture is preferably free from oxygen.

S. K. TWEEDY.

Production of cooling brine. H. STRATMANN and F. G. WERNER (STRATMANN & WERNER) (B.P. 327,039, 1.5.29).—Brine is rendered permanently neutral by adding "buffers," e.g., an acid component such as sodium acetate, and an alkaline component such as a primary phosphate. Alternatively, an amphoteric electrolyte, such as an amino-acid, may be added.

S. K. TWEEDY.

Production of sodium metaborate. A. KELLY (B.P. 326,378, 9.4.29).—Boron mineral (e.g., crude borax) is heated for 5 hrs. at 80° with sodium carbonate, lime, and water. The sodium metaborate solution formed is filtered off, bleached with hypochlorite, if necessary, and crystallised.

S. K. TWEEDY.

Manufacture of potassium nitrate. KALI-FORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 327,047, 10.5.29, Ger., 11.2.29).—A solution containing nitric acid is treated with potassium chloride at ordinary or raised temperatures in quantities such that at ordinary temperatures merely potassium nitrate separates out, and approximate saturation exists with respect to potassium chloride. The solution, if desired after separation of the potassium nitrate, is treated with nitrous gas (e.g., produced by the combustion of ammonia) and air until the initial nitric acid concentration is regained, nitrosyl chloride being formed also. The latter, separated from inert gases by absorption in silica gel, ferric chloride, etc., is decomposed with formation of nitric oxide which is employed to enrich the nitrous gases. The process may be conducted continuously.

S. K. TWEEDY.

Production of zinc salt solutions. METALLGES. A.-G. (B.P. 302,924, 20.12.28, Ger., 23.12.27).—The zinc oxide material is treated with a quantity of acid which is less than the equivalent of the zinc present, in such a manner that excess of acid with respect to zinc is always avoided. Thus the acid may be added continuously or intermittently to the oxide material, e.g., in the form of a paste, with continuous stirring, the temperature preferably never exceeding 40°. Further zinc oxide and acid may also be added to the mass under conditions adapted to prevent local excess of acid. The well-stirred mixture is then immediately filtered or centrifuged.

S. K. TWEEDY.

Manufacture of anhydrous zinc chloride by reacting zinc oxide with chlorine. C. VON GIRSEWALD and H. NEUMARK (U.S.P. 1,743,740, 14.1.30, Appl., 27.3.28, Ger., 9.4.27).—The reaction is effected in the presence of hydrogen, or a gas containing hydrogen, e.g., water-gas, the zinc chloride being distilled off, or drawn off from the reaction vessel in the molten state. The zinc oxide, in a finely-divided condition, may be carried into the reaction vessel in the stream of hydrogen.

F. G. CLARKE.

Purification of natural heavy spar. SACHTLEBEN A.-G. F. BERGBAU U. CHEM. IND. (B.P. 316,542, 22.4.29).

Ger., 30.7.28).—The spar is calcined under reducing conditions at 1300–1350° and quenched. The barium sulphide formed in small quantity during the calcination converts the metal oxide impurities during quenching into metal sulphides, which are readily removed by treatment with acid. Any quartz present is also removed. If the spar is non-bituminous it is mixed with bituminous spar or carbonaceous material before calcination. However, the impure spar, bituminous or non-bituminous, may also be calcined at 1300–1350° in an oxidising or reducing atmosphere and quenched in dilute sulphide solutions, *e.g.*, barium sulphide solutions. S. K. TWEEDY.

Manufacture of chromium oxide. I. G. FARBENIND. A.-G. (B.P. 302,178, 10.12.28. Ger., 10.12.27).—Alkali chromate or dichromate is mixed with red phosphorus in quantity insufficient for the appreciable formation of chromium phosphates and the whole is ignited, with or without preheating at 300°. Reducing agents (carbon or sulphur) may be added to combine with the liberated oxygen. Preferably a mixture of phosphorus and potassium dichromate is made into a paste with at most 20% of water and the whole caused to react. The oxide produced is eminently suitable as pigment. S. K. TWEEDY.

Impregnation of porous gels with (A) metals or other insoluble material, or (B) solid material. H. N. HOLMES (U.S.P. 1,739,306—7, 10.12.29. Appl., 13.8.27).—(A) The gel is impregnated, cold, with a solution of a catalyst-yielding compound containing also a reagent, which at an elevated temperature reacts to give the required insoluble deposit upon the gel. *E.g.*, silica gel is soaked in a solution of ammoniacal silver nitrate containing formaldehyde. The temperature is then raised to 100° to precipitate the metal. The pores in the interior of the gel thus become impregnated, since the surface is not choked by deposited metal. (B) The gel is impregnated with a solution of the metal salt etc., *e.g.*, ferric chloride, and then dried. A gas, *e.g.*, ammonia, which will react with the metal salt when in solution to give the catalyst, is introduced into the gel, and the latter then immersed in water, washed, and dried. F. G. CLARKE.

Manufacture of water-softening material of the exchange-silicate type. F. K. LINDSAY, Assr. to ARIZONA MINERALS CORP. (U.S.P. 1,744,703, 21.1.30. Appl., 19.2.25).—Finely-divided non-reacting material, *e.g.*, sand or base-exchange silicates which have fallen to powder, is added to a well-stirred mixture of solutions containing, respectively, sodium silicate and a sodium salt containing another metal, *e.g.*, sodium aluminate, so that gel formation takes place around the added particles; the gel is drained, dried for about 12 hrs. at the usual temperature, and then at about 60° in a current of warm air, preferably in a revolving drum, so that the product is obtained in a granular, non-powdery form. L. A. COLES.

Manufacture of complex antimony salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,176 and 326,231, [A] 5.12.28, [B] 6.12.28).—(A) Antimonic acid, freshly precipitated from the penta-

chloride, reacts with aliphatic hydroxy-acids (tartaric, gluconic, mucic, lactic, citric, etc.) to give relatively non-toxic complex salts of quinquivalent antimony. An organic base (hydroxytriethylamine) may be present. (B) Complex metal salts of aliphatic hydroxy-acids are treated with oxidants, *e.g.*, hydrogen peroxide, magnesium peroxide, to give less toxic compounds. An organic base, *e.g.*, diethylamine, may be present.

C. HOLLINS.

[Removal of carbon monoxide and dioxide from gas mixtures for use in] synthetic ammonia process. J. G. DELY, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,739,217, 10.12.29. Appl., 19.9.23).—Gas mixtures produced, *e.g.*, as described in the De Jahn process (Can.P. 201,059; cf. B.P. 120,546; B., 1920, 231A) are brought, without pretreatment with hot alkali solutions, in contact with ammoniacal cuprous (carbonate) solutions at high pressure and at 0° to remove carbon monoxide and dioxide, final traces of the latter gas being removed, if necessary, by subsequent scrubbing with cold sodium hydroxide solution.

L. A. COLES.

Continuous manufacture of carbon monoxide. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,227, 1.12.28).—Oxygen is blown into coke packed in a generator at a velocity (*e.g.*, 50 m./sec.) such that the primary reaction zone is rendered conical in form and is blown away from the nozzle and kept within the coke away from the generator walls. The ash of the coke is carried off as a vapour, containing combined sulphur (for the combination of which basic substances, like quicklime, may be added), and is separated from the carbon monoxide by washing. S. K. TWEEDY.

Production of [volatile inorganic] acids. H. FRISCHER (U.S.P. 1,754,156, 8.4.30. Appl., 16.5.27. Ger., 26.5.26).—See B.P. 271,881; B., 1928, 90.

Catalysts containing phosphoric acid (B.P. 326,185).—See I. Acid-resistant lead (B.P. 326,818 and 327,106).—See X.

VIII.—GLASS; CERAMICS.

Dependence of transparency of copper ruby glass on annealing time and temperature and the concentration of copper. L. RIEDEL and E. ZSCHIMMER (Keram. Rundsch., 1929, 37, 197–200, 237–239, 270–272, 545–547, 581–582, 634–636; Chem. Zentr., 1929, ii, 2488).—The temperature function of transparency for λ 5500 Å. was determined; the minimum is at 685°. Debye diagrams showed that the colouring matter of the glasses is metallic copper. It is technically desirable that the transparency to green light should be small; this is attained by increasing the copper content and annealing time and maintaining the most favourable temperature as exactly as possible.

A. A. ELDRIDGE.

Occurrence of rare elements in clays. A. FIOLETOV (Keram. Rundsch., 1927, 35, 270–272; Chem. Zentr., 1929, ii, 2489).—Primary Russian clays contain 0.22–1.35% TiO₂, and secondary clays 0.94–2.72%. The clays and kaolins examined contained 0.004–0.075% V₂O₅. A method for the determination of vanadium in clays is described. A. A. ELDRIDGE.

Glass distillation apparatus. OTHMER.—See I. [Ash from] rice husks. DE MONGEOT.—See XIX.

PATENTS.

Treatment of glassware [to prevent "bumping"]. J. A. JOBLING & Co., LTD., and G. L. HOCKENYOS (B.P. 307,793, 22.2.29. U.S., 13.3.28).—The whole or a part of the inner surface of beakers, coffee urns, etc. is roughened by sandblasting, frosting, etching, etc.

L. A. COLES.

Manufacture of safety glass. T. O. HENZE (B.P. 326,926, 9.5.29).—Two sheets of glass, the inner surfaces of which have been treated with an aqueous solution of isinglass, and an intermediate sheet of cellulose which has been treated with a solution of celluloid, urea, and formaldehyde in amyl acetate, are united, after the solvents have evaporated, in a bath of alcohol by the application of a pressure of 2–3 lb./in.² at about 15°; the edges are subsequently sealed. L. A. COLES.

Cellulose method of laminating glass. L. BARTELSTONE (B.P. 326,450, 10.12.28).—Sheets of glass with their inner surfaces conditioned by spraying with a solution of a cellulose derivative (cellulose nitrate) free from camphor, and having an intermediate sheet of celluloid conditioned superficially by spraying with a celluloid solution, are cemented together under heat and pressure, after the solvents have evaporated, by an expressed vegetable oil, *e.g.*, castor oil. L. A. COLES.

Manufacture of white enamels [for sheet or cast metal]. DEUTS. GASGLÜHLICHT-ADER-GES.M.B.H. (B.P. 310,528, 18.4.29. Ger., 28.4.28).—The highly basic complex compounds of oxides of antimony, zirconium, tin, or titanium, etc., containing the radical of thermally decomposable acid or acids (*e.g.*, nitric or formic), with or without salts of such acids, may be employed in quantities of at most 6% for rendering white and opaque enamels which are free from, or deficient in, fluorine. The opaquing action of the media rises to a definite limit with increase in the content (up to 0.5–3%) of the acid or acids unstable at incandescence.

S. K. TWEEDY.

Removal of sulphates from clays or like argillaceous materials. A. L. MOND. FROM KALI-CHEMIE A.-G. (B.P. 326,236, 8.12.28).—Barium salts (*e.g.*, carbonate, or more particularly fluosilicate) are added to the argillaceous material and the precipitation of the soluble sulphates is accelerated by an addition of soluble chlorides (*e.g.*, sodium chloride). S. K. TWEEDY.

Ceramic mass. J. M. LAMBIE and D. W. ROSS (U.S.P. 1,745,102, 28.1.30. Appl., 16.10.24. Renewed 14.11.28).—A ceramic casting slip of suitable composition is prepared with the use of a deflocculating mixture which contains ammonia solution, a basic compound of a metal, *e.g.*, litharge, and an organic polyhydroxy-compound of an acid character having at least two adjacent hydroxyl groups. Suitable reagents include oxalic acid, citric acid, gallic acid, mucic acid, pyrocatechol, quinol, and pyrogallol. C. A. KING.

Drying of ceramic articles in drying chambers and apparatus therefor. K. LÜHMANN (U.S.P. 1,744,398, 21.1.30. Appl., 30.9.27. Ger., 2.10.26).—A transverse flue passes beneath a battery of parallel

drying chambers, by which means hot air may be introduced into any one chamber and successively into others according to the hygroscopic and temperature needs of the material, *e.g.*, clayware, to be dried.

C. A. KING.

Refractory materials. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 326,279, 1.1.29).—High spalling-resistance is conferred to articles of refractory material (*e.g.*, fused alumina) by bonding the latter with an interlocking crystalline material other than the said refractory material. The crystalline material may be a titanium compound (titania alone or combined with magnesia) or an alkaline-earth oxide mixed with titania, or with a silicate, or with calcium fluoride.

S. K. TWEEDY.

Manufacture of fire-resisting material. W. T. WINCKLER, ASSR. to VON WINKLER CHEM. Co. (U.S.P. 1,743,176, 14.1.30. Appl., 2.2.27).—A mixture of silica 10 pts., lead oxide 8 pts., borax 8 pts., and calcium carbonate 1 pt. is fused to a glass, pulverised, and mixed with a fibrous material, *e.g.*, asbestos. The mixture is then heated until the flux fuses and is then pressed.

C. A. KING.

Refractory articles. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 326,560, 20.11.28).—The articles consist of, *e.g.*, silicon carbide and a composite binder, both stable under reducing conditions at 1300–1600°, the constituents of the binder being mutually soluble in varying proportions so that the binder matures continuously over a range of at least 300° between 1150° and 1900°, giving a m.p. curve approximating to a straight line over the same temperature range, and yielding a binder having a porosity at 1300° differing by less than 1% from that at 1500°. Suitable binders comprise mixtures of a (calcium) fluoride and a metal oxide (alumina or magnesia), and of diopside and forsterite.

L. A. COLES.

Refractory structure and article and method of forming the same. F. L. ARENSBURG, A. J. JACKMAN, and C. L. JONES, ASSRS. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,743,803, 14.1.30. Appl., 21.8.28).—Crucibles particularly adapted for melting steel in induction furnaces are made from chrome ore, magnesite, etc., with a porosity of at least 20%, the voids being lamellar in character. The pores are formed by introducing flat or flaky organic material, *e.g.*, bran, flat seeds, or fish scales, into the raw mixture, orientating the flat plates in a direction substantially parallel to the surface of the crucible wall.

C. A. KING.

Furnaces for annealing glass and other articles. T. TEISEN (B.P. 326,636, 19.2.29).

Joining glass to wood etc. (B.P. 302,324).—See V.

IX.—BUILDING MATERIALS.

Asphalt road emulsions. V. SHIPEROVICH, G. SHAVIRO, and O. PIRIK (Azerbaij. Neft. Choz., 1929, No. 10, 36–46).—The production of stable asphalt-water emulsions with "kontakt," naphthenic acids, or acid fuel is described. To the bitumen at 105–120° is added the emulsifying agent, and the mixture is poured into aqueous sodium hydroxide at 50–60°.

CHEMICAL ABSTRACTS.

Standards for and methods of testing of Polish road asphalts. M. MACZYŃSKI and W. SKALMOWSKI (Przemysl Chem., 1930, 14, 121—131).—The following official standards are given for Polish road asphalts respectively for the surface and basis of the road: d_{25}^{25} 1.220, 1.235 (± 0.002); viscosity (Hutchinson) 3—15, 20—100; free carbon 5—16, 5—18%; water 0.05, 0.5% (± 0.05); distillates boiling below 170° 1, 1%; at 170—270° 12—17, 8—18%; at 270—300° 4—12, 6—12%; at 300—350° 14—27, 6—16%. In both cases the products had: residue 55—65%, sintering point 60—75°, phenols 4% \pm 1, naphthalene 4% \pm 1, and ash 0.5%. Standard methods recommended for the testing of such asphalts are given in detail. R. TRUSZKOWSKI.

Cement from pyrites cinder. J. A. ALEKSANDROV (Neff. Choz., 1929, 17, 193—197).—A cement made from limestone (13), clay (1.3), and cinder (1 pt.) was resistant to sulphur compounds and gave the following values: volatile matter trace, SiO_2 24.22, Fe_2O_3 6.15, Al_2O_3 3.05, CaO 66.18, MgO trace, SO_3 0.35, R_2O 0.05%; d 3.26, hydraulic modulus 1.93%, silicate modulus 2.63%, setting time (beginning) 4 hrs. 55 min., (final) 6 hrs. 20 min.

CHEMICAL ABSTRACTS.

Constitution of hydrated Portland cement G. ASSARSSON and N. SUNDIUS (Sver. geol. Unders., 1929, 23, 7 pp.; Chem. Zentr., 1929, II, 2490).—In setting and hardening, the compound $3\text{CaO} \cdot \text{SiO}_2$ liberates 1 mol. of calcium oxide to the water.

A. A. ELDRIDGE.

Pine wood. P. M. SOUM (Bull. inst. Pin, 1929, 221—230, 253—264, 313—316).—Chiefly an account of work already published.

CHEMICAL ABSTRACTS.

Pneumatic transport. NEU.—See I. Waste-wood utilisation. NELSON. Natural gas [for lime kilns]. KERTÉSZ.—See II.

PATENTS.

Production of cement. G. IMAI (B.P. 319,342, 15.2.29. Jap., 21.9.28).—A mixture of calcined igneous rock containing quartz and felspar with a substance containing large quantities of soluble acidic components, e.g., naturally or artificially weathered tuff, basalt, or andesite, is ground and mixed with Portland cement clinker, and the mixture is pulverised. L. A. COLES.

Manufacture of plaster for building and like purposes. E. JOSZ (B.P. 326,625, 5.2.29).—A mixture of plaster of Paris, 0.6% by wt. of powdered erythro-dextrin, and 0.5—1.2% of sodium borate is tempered by the addition of water, or, alternatively, the dextrin and sodium borate are dissolved in the water used for tempering the plaster.

L. A. COLES.

Production of moulded articles. BELL'S UNITED ASBESTOS CO., LTD., J. A. CANN, and E. R. HARRAP (B.P. 326,825, 16.11.28).—Compositions for moulding under heat and pressure comprise finely-divided mixtures of alkaline-earth or magnesium compounds which set by combination with water (e.g., plaster of Paris, Portland cement, hydraulic or fat limes, magnesia) together with salts containing water of crystallisation, especially heavy-metal sulphates, chlorides, phosphates, and nitrates; compounds which react under heat and pressure with the heavy-metal salts (e.g., zinc oxide or

magnesia when zinc sulphate is used), as well as fillers, colouring material, etc. may also be added.

L. A. COLES.

Manufacture of [waterproofing] aqueous dispersions [of bitumens etc.]. L. KIRSCHBRAUN (U.S.P. 1,738,776, 10.12.29. Appl., 6.10.27).—Aqueous dispersions containing, e.g., asphalt with clay as the dispersing agent are stabilised by the presence of a silica gel formed *in situ* by the addition of sodium silicate solution and hydrochloric acid to the dispersing agent or to the dispersion, the quantity of acid being such that the product has p_H 3.0—3.5 when normal brick-making clays are used, or somewhat higher (e.g., 6.0—6.5) with clay having poor suspensive properties.

L. A. COLES.

[Cold] bituminous paving. (SIR) G. C. MARKS. From ROL LISTER & CO. (B.P. 326,863, 23.11.28).—Preheated and dried mineral aggregate, which may contain 5—20% of fine dust or "filler," is coated with bitumen previously softened by heating with sufficient refined tar or bituminous or asphaltic base oil to yield a matrix of m.p. 15—25°.

L. A. COLES.

Preparation of cellular building material. G. M. THOMSON, ASSR. to CANADA GYPSUM & ALABASTINE, LTD. (U.S.P. 1,753,255, 8.4.30. Appl., 20.12.26).—See B.P. 305,806; B., 1929, 325.

Bituminous compositions (U.S.P. 1,738,906).—See II. Joining wood to metal etc. (B.P. 302,324).—See V. Insecticide [for wood] (U.S.P. 1,744,324).—See XVI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Electric-furnace iron. G. L. SIMPSON (Amer. Electrochem. Soc., May, 1930. Advance copy. 6 pp.).—The present status of the electric furnace in the iron foundry is discussed with special reference to continuous processes which have become important in recent years. Examples are given of the duplex process in which the metal is first melted in the cupola and of the process in which the charge is melted in the electric furnace itself.

H. J. T. ELLINGHAM.

Influence of phosphatisation and a finish on the electrical insulation of steel plates. J. COURNOT (Compt. rend., 1930, 190, 934—936).—As ferrous alloys phosphatised by the author's method (cf. B., 1928, 18) showed poor superficial conductivity for electrodeposition, the electrical effect of phosphatisation has been investigated. Phosphatised plates covered with a thin layer (0.15—0.3 mm.) of an insulating varnish or of bakelite, and an unphosphatised plate for comparison, were placed (i) dry, and (ii) after being 24 hrs. in a saturated atmosphere, between electrodes by which was applied (a) an alternating current (50 cycles) of voltage increasing until rupture occurred, and (b) a direct current of 300 volts, the current passing being measured. The voltage for rupture was lower (1/2 to 1/9), whilst insulation was much greater (10—100 times for dry, and more for moist, plates) in the phosphatised plates.

C. A. SILBERRAD.

Properties of special steels. K. SASAKAWA (J. Iron Steel Inst. Japan, 1929, 15, 201—224).—A study of

the change of structure and hardness on quenching specimens of nickel, chromium, nickel-chromium, and nickel-chromium-molybdenum steels.

CHEMICAL ABSTRACTS.

Permeability of electric steel. M. NAKAMURA (J. Iron Steel Inst. Japan, 1929, 15, 95—123).—In cast steel the permeability (determined with Baro's permeameter) increases with decrease (up to a point) of the carbon content; below 0.12% C the permeability is generally low. Silicon somewhat increases the permeability, the limit depending on the manganese and carbon contents; its effect is that of deoxidation.

CHEMICAL ABSTRACTS.

Dilatometric analysis of certain nickel, vanadium, and nickel-vanadium steels. J. CHALLANSONNET (Compt. rend., 1930, 190, 939—940).—The steels examined all contained approx. 3.75% C, (a) had no further addition; (b) and (c) contained, respectively, 1 and 2% Ni; (d) and (e) 0.25 and 0.5% V; and (f) 1 or 2% Ni each with 0.25 or 0.5% V. They were all poured at 1500° into cylinders 8 or 25 mm. in diam. and 95 or 90 mm. high. Photomicrographs showed the carbon in all cases to be combined, and (a—c) showed eutectic cementite and primary pearlite in dendrites. All those containing vanadium exhibited lamellæ of pre-eutectic cementite and a network of eutectic cementite. On examination with Chevenard's dilatometer (Pyros standard), it was found that nickel had no effect on the Curie point (200°), but 0.25 and 0.5% V lowered it, respectively, to 160° and 130°. Nickel lowers the Ac and Ar points; vanadium alone has no effect, but when present with nickel it cancels the effect thereof. Nickel lowers the temperature of graphitisation, vanadium in amount equal to one quarter that of the nickel cancels its effect; alone it prevents graphitisation, at least up to 1100°.

C. A. SILBERRAD.

Change in density and electrical conductivity of copper during polishing. P. SIEBE (Metall u. Erz, 1929, 26, 397—400; Chem. Zentr., 1929, ii, 2931).—The density is maximal at 0.2% O, falling on overpolishing owing to absorption of hydrogen and sulphur dioxide. The electrical conductivity increases with falling oxygen content, further rising and then falling on overpolishing. In general, the danger of overpolishing is the greater the purer is the copper.

A. A. ELDRIDGE.

Determination of copper in commercial aluminium. J. C. GHOSH (J. Indian Chem. Soc., 1930, 7, 125—126).—The aluminium is dissolved in dilute (1:5) sulphuric acid, when the copper remains, and is subsequently determined iodometrically, after washing and dissolution in nitric acid. Results are quoted showing that the method compares favourably with existing methods.

J. R. I. HEPBURN.

Influence of silicon on the mechanical properties of aluminium bronze. S. SHIOZAWA (J. Min. Met., Japan, 1929, 45, 217—220).—With 2% Al, the effect of silicon is small; with 4% Al, over 1.5% Si rapidly increases the tensile strength. Experiments with aluminium bronzes containing 0—3% Si and 2—12% Al are recorded.

CHEMICAL ABSTRACTS.

Analysis of aluminium alloys. F. L. HAHN (Z. anal. Chem., 1930, 80, 192—200).—The alloy (0.5 g.) is

placed in a boat in a silica tube and heated in a current of chlorine charged with the vapour of carbon tetrachloride, a receiver containing isopropyl alcohol being connected with the tube. The solid sublimate condensing in the cooler front portion of the tube contains the whole of the iron, nearly all the aluminium, and a little titanium, whilst the liquid in the receiver contains all the silicon and the remainder of the titanium; a small residue of alumina [? also calcium and magnesium] remains in the boat. The constituents present in the above three fractions are then determined by the usual methods. Calcium, sodium, and magnesium, if present, are determined in a separate portion of the alloy after dissolution in hydrochloric acid. Attempts to obtain a direct determination of the oxygen present were not successful, but it is concluded that the alumina remaining in the boat represents that originally present in the alloy as corundum. A detailed description is given of the method and of the apparatus employed.

H. F. HARWOOD.

Magnesium-manganese alloys. G. W. PEARSON (Ind. Eng. Chem., 1930, 22, 367—370).—As a method of preparing alloys, rather large pieces of manganese were immersed in molten magnesium and the temperature was maintained at 750° for some hours. The solid-solution area extends beyond 2.7% Mn, and in the higher ranges manganese may appear as angular masses. When the alloy contains 35% Mn or more the manganese shows as irregular masses in a background of magnesium.

C. A. KING.

The Betts electrolytic lead-refining process in practice. J. J. FINGLAND (Amer. Electrochem. Soc., May, 1930. Advance copy. 28 pp.).—Details of modern practice in the operation of the Betts process are described with special reference to the methods employed by the Consolidated Mining & Smelting Co. of Canada at Tadenac, B.C. Concrete cells lined with asphalt have now replaced wood, which is eliminated as far as possible from the cell room. The cells are arranged in cascade units through which the electrolyte is circulated by means of copper pumps with monel metal wearing parts. At Tadenac the anodes are arranged with their centres 10.5 cm. apart and usually have a life of 8 days, although 6-day anodes are sometimes adopted in obtaining specially pure lead. Two crops of cathodes are drawn from each set of anodes. The electrolyte in circulation contains an average of 12% of total hydrofluosilicic acid and 8% of lead. The anodic current density is normally 1.8—1.9 amp./dm.² To meet the stringent requirements of the white lead industry the silver content of the refined metal has to be kept extremely low. For this reason the percentage of silver in the anodes must not be too high and the anodes cannot be run below about 20% of scrap. On the other hand, the amount of impurities in the anodes should not be too low since this leads to a slime which does not settle well and may therefore be included in the cathode deposit. It is found that the best conditions are obtained when the anodes contain about 0.85% Sb, for this forms on the anode surface an adherent sponge, which includes most of the other impurities. Hence the anodes are made to contain this proportion of antimony, if necessary by adding antimonial lead or

antimony recovered from the slimes. The total impurities in the anodes are usually less than 1.25%. Under these conditions the initial potential drop between anodes and cathodes is 0.35–0.45 volt, but as the thickness of the layer of slime increases, the resistance rises and the potential-drop reaches 0.65–0.70 volt at the end of the life of the anode. This increase in resistance is partly due to the concentration of lead fluosilicate in the pores being greater than that in the bulk of the electrolyte while the concentration of free hydrofluosilicic acid is lower. The origin of these concentration differences is discussed. The electrolyte must therefore be regarded as consisting of two parts: a portion which circulates and a portion which is stationary in the slime sponge. At Tadenac the average proportion of total hydrofluosilicic acid in the former to that in the latter is 100:15. For economic success all the hydrofluosilicic acid entrained in the sponge must be returned to the electrolyte. The proportion of lead fluosilicate to free hydrofluosilicic acid in the filtrate from the slimes washing process is adjusted by addition of sulphuric acid. The slimes are dried and melted in a reducing atmosphere, whereby some lead antimonite is slagged off. Then by the action of a jet of compressed air the arsenic and most of the antimony are driven off as oxides, which are recovered by means of a Cottrell precipitator. The rest of the antimony is removed as a lead antimonite slag, after which bismuth and copper undergo oxidation yielding a slag which is worked up for bismuth. The bismuth is recovered electrolytically. The residue of doré metal is parted by the sulphuric acid process to obtain silver. The gold sludge is cast into anodes and refined by the Wohlwill process, whereby some palladium is recovered. The cathode lead obtained in the main process is melted and then treated with compressed air at 540°. Antimony is thereby reduced to 0.001% and small amounts of arsenic and tin are completely eliminated. The hydrofluosilicic acid is best prepared by passing hydrofluoric acid vapours, produced by the action of sulphuric acid on pure fluor-spar, into water containing finely-crushed quartz or silica in suspension. Best French glue is used as addition agent. The composition of the electrolyte must be carefully controlled and rapid volumetric methods which have been developed for determining the concentrations of the various constituents are described.

H. J. T. ELLINGHAM.

Electrolysis of gold, electrolytic reduction of silver chloride, bronze plating, cadmium plating, and plating with copper-cadmium alloy. H. YAGI (J. Mint, Japan, 1929, 2, 56–77).—The composition and method of stirring the gold electrolyte were studied. Electrolytic reduction of silver chloride, using a silver cathode and a lead anode in sulphuric acid in a clay crucible (diaphragm), is convenient, but more expensive than reduction with zinc and sulphuric acid. Conditions suitable for bronze, cadmium, and copper-cadmium alloy plating are recorded. CHEMICAL ABSTRACTS.

Use of steel in the oil industry, with special reference to heat-resisting steels. (SIR) R. HADFIELD and S. A. MAIN (J. Inst. Petroleum Tech., 1929, 15, 611–633).

Copper from copper sulphate manufacture. FICAL.—See VII. **Cement from pyrites cinder.** ALEKSANDROV.—See IX.

PATENTS.

Device for separating and concentrating ores. A. W. McTAGGART (U.S.P. 1,744,785, 28.1.30. Appl., 21.7.27).—Ore pulp is treated in a tank in which upper and lower baffles are set longitudinally and inclined so as to form almost an inner compartment. Paddles agitate the pulp, causing it to circulate upwardly in the centre and through perforations in the upper baffles, and a comminuted jet of air discharges close to the agitator.

C. A. KING.

Treatment of ores, metallurgical products, etc. METALLGES. A.-G. (B.P. 317,001, 3.7.29. Appl., 8.8.28).—The ore mixed with fuel, if necessary, is passed successively in a Dwight-Lloyd roasting furnace under a series of ignition furnaces of gradually increasing intensity. The process is especially applicable to the roasting of zinc blende, which is thereby prevented from slagging.

A. R. POWELL.

Roasting of [sulphide] ores and minerals. M. F. COOLBAUGH and J. B. READ, Assrs. to COMPLEX ORES RECOVERIES Co. (U.S.P. 1,744,867, 28.1.30. Appl., 13.6.24).—The sulphide is passed in direct current with air through a roasting furnace to obtain a mixture of oxides and sulphates, and this product is mixed with a raw sulphide ore or with crushed matte and the mixture sintered in Dwight-Lloyd or Huntington-Heberlein sintering plant preparatory to smelting in a reverberatory or blast furnace.

A. R. POWELL.

Production of metals from their oxide ores. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 306,949, 15.2.29. U.S., 28.2.28).—A mixture of oxide ore, powdered fuel, and flux is heated indirectly, by means of a number of tubes passing through the chamber, until substantial reduction is effected without sintering. The reduced charge is then smelted directly by combustion of the carbon in the charge, and the gaseous products are utilised in heating the tubes in the reduction chamber and for preheating air for combustion, which may be regulated by mixing the products of combustion with inert gases.

C. A. KING.

Melting and deoxidising metals and alloys. W. REITMEISTER (B.P. 327,570, 5.4.29).—The deoxidising agent comprises a mixture of a metal oxide and sufficient finely-divided carbonaceous material to reduce it with the formation of carbon monoxide. *E.g.*, copper alloys may be deoxidised with a mixture of zinc oxide and coal dust, and iron or steel with a mixture of hæmatite and coal dust. An excess of oxide may be used to prevent carburisation of the metal.

A. R. POWELL.

Transformation of pig iron into desulphurised and dephosphorised steel or into pure iron. CARBONISATION SOC. GÉN. D'EXPLOIT. DES CARBONES (B.P. 303,891, 3.1.29. Fr., 12.1.28).—The oval bottom of an acid-lined converter mounted on hollow trunnions is raised towards the middle to impart a gyratory motion to the molten charge when rocked. The trunnions end in the upper part of the converter as tuyères for the

admission of fuel and air. Pig iron is introduced into an atmosphere of carbon dioxide and the metal is freed, in turn, from silicon, carbon, sulphur, and phosphorus, finally being purified in an atmosphere of hydrogen.

C. A. KING.

Annealing furnace. J. R. GARNER and J. H. ROYSTON (B.P. 326,562, 20.12.28).—A furnace particularly adapted for annealing metal tubes or rods is of tunnel form with a longitudinally grooved floor. Straight tubes or rods are connected together temporarily before entering the furnace and a number of such continuous lengths are pushed along the channels in the furnace floor.

C. A. KING.

Manufacture of compact metal articles from metal [iron] powders. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,536, 13.12.28. Addn. to B.P. 311,141; B., 1929, 526).—Iron powder produced by thermal decomposition of the carbonyl is pressed into an ingot heated at 1200° preferably in hydrogen, and subjected at this temperature to a pressure of about 2000 kg./cm.² Alloys of uniform composition may be obtained in a similar manner by adding the powdered alloying element to the iron powder prior to subjecting it to the above treatment.

A. R. POWELL.

Welding of metals and alloys. I. G. FARBENIND. A.-G. (B.P. 305,999, 13.2.29. Ger., 13.2.28).—Thick metal sheets or parts may be welded by applying an oxy-hydrogen or oxy-acetylene burner to the place to be welded and at the same time generating a luminous electric arc in the flame between the work and the welding rod.

A. R. POWELL.

Welding method and composition. P. L. & M. CO., Assees. of H. J. MORGAN (B.P. 317,361, 8.2.29. U.S., 14.8.28).—A mixture consisting of 90% of tungsten, 10% of carbon, and a binder is fused on to the surface of a metal to form a hard layer of tungsten carbide. Other elements may be incorporated in the mixture to form hard alloys, and such a process may be applied in the manufacture or repair of tools, e.g., earth-boring drills.

C. A. KING.

Working of normally brittle sheet metal. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of W. E. RUDER (B.P. 311,290, 8.5.29. U.S., 8.5.28).—Metals which are normally brittle, e.g., iron containing chromium and aluminium, are heated to below 200° and bent round a die heated to the same temperature. Thus flat, metallic ribbon may be wound edgewise on to a heated die, or sharp bends produced on sheet metal.

C. A. KING.

[Copper]-aluminium alloys. O. REULEAUX (B.P. 309,586, 27.3.29. Ger., 13.4.28).—Copper-aluminium alloys with 3–5.5% Cu are treated with up to 0.6% Ti, Zr, or B in the presence of a molten alkali fluoride flux, whereby the solid solubility of copper in aluminium at high temperatures is increased and a greater hardness and tensile strength are obtained on subsequent ageing. The titanium may be introduced by aluminothermic reduction of an alkali fluotitanate on the surface of the molten alloy or by addition of a copper-titanium alloy to the bath.

A. R. POWELL.

Manufacture of aluminium-silicon alloys. Soc. D'ELECTROCHIM., D'ELECTROMÉTALL., ET DES ACIÉRIES

ELECTR. D'UGINE (B.P. 302,692, 19.12.28. Fr., 20.12.27).—Briquettes of kaolin, alumina, and petroleum coke containing an excess of 4% over the theoretical amount of carbon are heated in a refractory crucible by means of an arc of 6000–8000 amp. at 35–50 volts under an arch of semi-fused material of the same type. Alloys containing 65–70% Al, 30–35% Si, and small quantities of iron, titanium, and carbon are thus readily obtained with a loss of less than 5% of the aluminium and silicon by volatilisation.

A. R. POWELL.

Rosin-core solder. P. C. RIPLEY (B.P. 327,337, 7.6.29).—A self-fluxing solder comprises a tube of soft solder filled with a plastic flux made by melting rosin with 2–50% of its weight of a solvent, e.g., turpentine.

A. R. POWELL.

Obtaining lead from its ores or other compounds. G. L. OLDRIGHT (U.S.P. 1,744,174, 21.1.30. Appl., 22.11.26).—The ore, if necessary, after a complete or partial roasting, is heated in a reducing atmosphere or with a reducing agent to obtain the lead in the form of prills disseminated throughout the partially sintered gangue. A considerable proportion of the lead may be removed by liquation and pressing, and the remainder by smelting in the blast furnace the granular residue from the presses.

A. R. POWELL.

Improving [the resistance of] lead [to sulphuric acid]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,818 and Addn. B.P. 327,106, 18.12.28).—Soft lead is alloyed with (A) 0.01–0.05% Ni and 0–0.1% Se, or (B) with up to 0.1% Se alone.

A. R. POWELL.

Metal-coated articles [lead-coated paper]. L. W. CUTLER, C. A. KLEIN, and ASSOCIATED LEAD MANUFACTURERS, LTD. (B.P. 326,792, 14.12.28).—The paper is first impregnated with a naphtha solution of bitumen and oil-soluble synthetic resin ("Albertol") and dried at 30–60°, then sprayed with finely-divided lead (200-mesh), and finally burnished with steel shot or by passing the paper through burnishing rollers. The surface may subsequently be coated with a second metal, e.g., copper, by electrodeposition or by simple replacement.

A. R. POWELL.

Flotation process [for lead-zinc ores]. J. H. LANE (U.S.P. 1,743,463, 14.1.30. Appl., 3.8.27).—The ore is ground with 1–4 lb./ton of sawdust (preferably from pinewood) and the usual chemicals to prevent flotation of zinc blende, e.g., cyanide, xanthate, bicarbonate, and zinc sulphate. On subsequent agitation in the flotation machine the wood pulp carries the galena into a froth which is readily floated off. The zinc mineral is removed from the tailings by addition of copper sulphate and xanthate and re-treatment in the flotation machine.

A. R. POWELL.

Mercury-extracting apparatus. L. DAAMS (U.S.P. 1,744,827, 28.1.30. Appl., 25.5.27).—A combustion chamber communicates with the lower and upper end of a retort chamber containing one or more vertical retorts, with damper control of the heat entering the chamber. The upper end of the retort is surrounded by a superheating coil, and an entrainment gas may be passed through the retort.

C. A. KING.

Manufacture of articles from alloys. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,518, 15.9.28).—An article of the desired shape is made of one of the constituents of the alloy and the second constituent is applied to the surface of the article by electroplating or by coating with the powdered metal. The composite metal is then heated at such a temperature and for such a time that complete diffusion of the constituents into a homogeneous alloy takes place. *E.g.*, a nickel tube plated internally and externally with 25% of its weight of chromium is readily converted into a tube of nickel-chromium alloy at 1350° in a vacuum. The process is also applicable to the manufacture of thin-walled articles of cobalt-iron, silicon-iron, chromium-iron, and nickel-chromium-iron alloys.

A. R. POWELL.

Filtration of cyanide slimes and apparatus therefor. C. LINTECUM (U.S.P. 1,744,480, 21.1.30. Appl., 3.9.24).—In order to reduce the number of tanks and also the amount of pumping required, a number of filters or vats of equal size on the same level are operated in definite cyclic relation with one another, the pulp and wash solutions being transferred from one vat to another instead of from the filter to excess tanks and back again.

C. A. KING.

Galvanising bath. J. SUMMERS & SONS, LTD., and H. B. FREEMAN (B.P. 327,181, 3.8.29).—A galvanising bath containing spelter etc. is enclosed with a closed jacket containing a fusible metal, *e.g.*, lead.

J. S. G. THOMAS.

Production of steel in an open-hearth furnace. R. DURRER, F. C. SIEMENS, and A. SPRENGER, ASSRS. to F. SIEMENS A.-G. (U.S.P. 1,752,374, 1.4.30. Appl., 28.1.27. Ger., 26.9.25).—See B.P. 293,326; B., 1928, 676.

Manufacture of ductile iron-chromium-aluminum alloys. V. B. BROWNE (U.S.P. 1,754,008, 8.4.30. Appl., 19.12.27).—See B.P. 302,640; B., 1929, 781.

Manufacture of macrocrystalline [tungsten] ingot. H. ALTERTHUM, ASSR. to GEN. ELECTRIC CO. (U.S.P. 1,752,877, 1.4.30. Appl., 6.7.22. Ger., 13.7.21).—See B.P. 183,119; B., 1923, 175 A.

Non-ferrous welding rod. A. R. LYTLE, ASSR. to UNION CARBIDE & CARBON RES. LABS., INC. (Re-issue 17,631, 1.4.30, of U.S.P. 1,716,590, 11.6.29).—See B., 1929, 648.

Electrolytic recovery of metals [nickel]. S. C. SMITH (U.S.P. 1,754,125, 8.4.30. Appl., 19.4.29. U.K., 30.3.28).—See B.P. 314,579; B., 1929, 725.

Magnetic separator (B.P. 303,513). **Filtering medium** (U.S.P. 1,743,524—5).—See I. **Joining metal to wood etc.** (B.P. 302,324).—See V. **White enamels** (B.P. 310,528). **Refractory article** (U.S.P. 1,743,803).—See VIII. **Smelting furnaces** (B.P. 302,308). **Separation of materials** (U.S.P. 1,744,989). **Chrome baths** (B.P. 327,293).—See XI. **Protection from mercury vapour** (G.P. 455,974).—See XXIII.

XI.—ELECTROTECHNICS.

Lead accumulators. E. DENINA (Giorn. Chim. Ind. Appl., 1930, 12, 67—72).—An investigation has been

made of the reactions occurring at the plates of an accumulator during discharge, by a potentiometric and gravimetric examination of artificial electrodes consisting of platinum plates coated electrolytically with lead peroxide and formed in sulphuric acid. Measurements of the *E.M.F.* and temperature coefficients of the single plates have been made in order to calculate the heat of the reaction occurring during discharge. The results in each case are in agreement with the theory of double sulphation during discharge. Measurements of the variation of potential and of the plate resistance during charge, discharge, and inversion show that the resistance in the cell depends primarily on the acid permeating the active material in the plates. The lead sulphate formed during discharge, contrary to expectation, has little effect on the resistance. F. G. TRYHORN.

Chemistry in the telephone industry. R. R. WILLIAMS (Ind. Eng. Chem., 1930, 22, 316—322).—A general account is given of work carried out in the chemical laboratories of the Bell Telephone Company.

H. INGLESON.

Electric-furnace iron. SIMPSON. **Corrosion of pipes.** KUHN. **Insulation of steel plates.** COURNOT. **Electrolytic lead refining.** FINGLAND. **Electrolysis of gold and electroplating with bronze, cadmium, and copper-cadmium.** YAGI.—See X. **Polymerisation of oils.** GARDNER.—See XII. **Determination of copper in vegetables.** SPRINGER.—See XIX.

PATENTS.

Electric furnaces of the inductor type. E. F. NORTHROP (B.P. 316,662, 9.7.29. U.S., 2.8.28).—A magnetic core is inserted within the extension of the inductor coil unoccupied by the charge and the hearth or base of the crucible, so that the air gap traversed by the magnetic flux is reduced. J. S. G. THOMAS.

Electric induction smelting furnaces. J. HÄRDÉN (B.P. 302,308, 27.11.28. Swed., 14.12.27).—A main low-frequency furnace with closed iron core and an auxiliary high-frequency furnace without such core are connected, *e.g.*, by a duct, so that a charge, melted in the latter furnace, may be transferred to the former.

J. S. G. THOMAS.

Cathodes for electron-discharge tubes. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 319,652, 7.3.29. Holl., 25.9.28).—An indirectly heated cathode is introduced between the heating element and the emitting coating, the latter consisting, at least in part, of an alkali metal or compound, *e.g.*, caesium. J. S. G. THOMAS.

Determination of intensities of [ultra-violet] radiation. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,616, 15.5.29).—In a process carried out in accordance with B.P. 296,198 (B., 1928, 864), an optical filter, *e.g.*, composed of Uviol glass and cellophane coloured with picric acid, is inserted before the test liquids, so that a sharply-defined range of wave-lengths is transmitted through the filters, and is incident upon the test liquids. The radiations are preferably compared by observing the respective times in which the test liquids attain a standard coloration.

J. S. G. THOMAS.

organic salt) polymerised rapidly under these conditions and produced solid products. Tung oil treated with 0.05% S produced a wax-like mass resembling a hydrogenated fat. It is suggested that the tung oil (cobalt addition) product could be used in linoleum manufacture.

J. O. CUTTER.

Autoxidation of corn [maize] oil as related to its unsaponifiable constituents. H. A. MATTILL and B. CRAWFORD (Ind. Eng. Chem., 1930, 22, 341—344).—The initial stages of the oxidation, as measured by the decrease of the induction period of maize oil after being subjected to various commercial processes of manufacture, have been studied. The decrease of the induction period is, in general, parallel to the decrease in the amount of unsaponifiable matter (sterols) present. The unsaponifiable matter contains substances which act as antioxidising substances, and so lengthens the period of induction. This action disappears on acetylation of the unsaponifiable matter, and is probably associated with the presence of a hydroxyl group in this substance. Commercial corn oil has less satisfactory keeping qualities than the cold-solvent-extracted oil because of the loss of unsaponifiable matter during the processes of purification, and also to changes in this material rendering it less active as an antioxidant.

J. O. CUTTER.

Composition of commercial stearine and oleine.

V. VESELY (Chim. et Ind., 1929, 22, 681—688).—The characteristics and requirements of technical oleines are described, and the presence of isomeric acids in distilled oleines is discussed. From the distillation products of 10-hydroxystearic acid, Δ^9 (elaidic) and Δ^{10} (isooleic) acids, *trans*-octadecenoic acids have been isolated (solid portion) and the presence of Δ^{10} -*cis*-octadecenoic accompanying the Δ^9 (oleic) acid has been demonstrated; it is therefore considered probable that distillation oleine represents a solution, saturated in the cold, of all the components of the "stearine" in the liquid Δ^9 - and Δ^{10} -oleic acids (cf. Vesely and Majtl, A., 1926, 47; Chem. Listy, 1928, 325). The possibility of the occurrence of two other isomerides (Δ^8) derived from 9-hydroxystearic acid (probably formed during the sulphuric acid treatment), or of other sets of isomerides from isomeric octadecenoic acids (e.g., petroselinic) in the original oil is not disregarded. E. LEWKOWITSCH.

Milkweed. GERHARDT.—See V.

PATENTS.

Preparation of [vitamin-containing] margarine from vegetable fats. W. W. TRIGGS. From VAN DEN BERGH'S MARGARINE GES.M.B.H. (B.P. 326,742, 25.7.29).—Vitamin-containing extracts from rice husks, malt, etc. are emulsified with vegetable mucilages or gums, egg yolk, etc. before being added to the margarine during churning.

E. LEWKOWITSCH.

Neutralisation of free acidity of oils and fats. J. M. ZUMETA and A. MARTICORENA (YRASTORZA) (B.P. 308,250, 6.12.28. Spain, 20.3.28).—The oil is dissolved in carbon tetrachloride or trichloroethylene and neutralised by aqueous alkaline lye; the solvent is evaporated after washing the oil solution with brine.

E. LEWKOWITSCH.

Refining of wool fat. I. G. FARBENIND. A.-G. (B.P. 303,890, 3.1.29. Ger., 13.1.28).—Crude wool fat is esterified in excess of a lower aliphatic alcohol (e.g., by ethyl alcohol with concentrated sulphuric acid); the insoluble portion of the product is separated and constitutes an almost odourless product of low acid value and high m.p. (yield 65%, dropping pt. 45—46°).

E. LEWKOWITSCH.

Deodorisation of wool fat. M. GILLET (F.P. 569,624, 9.8.23. Belg., 2.8.23).—The wool fat, preferably in the form of aqueous emulsion, is treated with sulphur, sulphur compounds, sulphides, or polysulphides and an alkali; on stirring, mercaptans are liberated which are absorbed by the alkaline solution. After washing with water an odourless fat is obtained.

E. LEWKOWITSCH.

Production of vegetable fats from grape marc. SOC. ANON. FABR. CHIM. ARENELLA (F.P. 635,628, 8.6.27. It., 5.4.27).—By extracting the marc with sodium carbonate solution or carbon disulphide, 8—10% of fat, having f.p. 65° and containing about 50% of stearic acid, is obtained.

E. LEWKOWITSCH.

Bleaching of fatty acids [of marine origin]. A. GODAL (U.S.P. 1,740,012, 17.12.29. Appl., 11.2.26. Nor., 29.1.25).—The crude fatty acids are dissolved in a volatile solvent, e.g., benzine, benzene, tetrachloroethane, and agitated with small amounts of concentrated sulphuric acid at 15—20°. On keeping, the sulphonated acids settle out at the bottom, carrying with them the colouring matter; the oil is recovered by evaporation of the purified layer above.

E. LEWKOWITSCH.

Esterification of fatty acids and mixtures containing them. E. VECKER (F.P. 635,452, 2.6.27. Ger., 23.6.26).—Fatty acids are esterified in the usual way with deficiency of an alcohol, especially glycerol, the excess fatty acids being removed *in vacuo* by means of superheated steam or gases in the presence of liquid mists.

E. LEWKOWITSCH.

Manufacture of soaps. W. MANN (B.P. 325,865, 29.10.28).—Fatty acids (e.g., stearic, oleic) are saponified by milling with alkali carbonates, or fats with caustic alkalis, in the dry state, water being added afterwards to obtain the requisite moisture content for the finished soap. The saponification can be accelerated by the addition of a small proportion (5%) of alcohol.

E. LEWKOWITSCH.

Preparation of soaps containing a fat-solvent. S. ZIMMERMANN, and "HENRY" SEIFEN-, KERZEN-, UND FETTWARENFABR. G.M.B.H. (Aust.P. 109,404, 27.7.25).—Fats are saponified in the presence of, e.g., 3½% of isopropyl or *n*-propyl alcohol before the addition of benzine, or alkali may be added to a solution of the solvent, propyl alcohol, and fat.

E. LEWKOWITSCH.

Drying and conditioning of household soap. TOMLINSONS (ROCHDALE), LTD., and J. N. TOMLINSON (B.P. 325,807, 25.10.28).—Apparatus is described for conveying the raw soap through drying chambers against hot air currents and then through cooling chambers provided with circulating cold air.

E. LEWKOWITSCH.

Preparation of detergents and cleansers. D. D. BROOKS. (B.P. 326,755 and 326,759, 24.9.29).—Tri-sodium phosphate and sodium sesquicarbonate and/or saponin are added to soaps, abrasives, etc.

L. A. COLES.

Extraction of [oleaginous] materials. H. G. C. FAIRWEATHER. From PROSCO OILS CORP. (B.P. 324,681, 1.8.28).—The material is subjected to continuous countercurrent contact, descending by gravity through the ascending solvent: in the first (uppermost) zone the solvent is relatively quiescent and saturates the material, which acts as a filter bed to remove fines from the solution which flows off above; in the second zone the material is agitated with the entering solvent and then accumulates at the bottom of the container, displaces by its weight any contained solvent, and is removed by suitable extracting apparatus.

E. LEWKOWITSCH.

Treatment of oil-containing fruits [especially palm fruits.] F. KRUPP GRUSONWERK A.-G. (B.P. 317,366, 25.6.29. Ger., 14.8.28).—The pulp is detached from the nuts by simultaneous dry heat treatment and mechanical stirring devices, and the pulp (with or without admixed nuts) is subjected to a second dry heat treatment in another larger, shallow pan, to remove the remaining water before subjection to oil-recovery processes.

E. LEWKOWITSCH.

Refining sulphur olive oil and the like [extracted oils]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,539, 14.12.28).—The oils are stirred for 10–20 min. at 60–80° with 1–4% of a concentrated volatile acid, *e.g.*, hydrochloric acid (*d* 1.19) or formic acid, which precipitates the slimes in the form of flocks; the oil is subsequently washed by showering.

E. LEWKOWITSCH.

Production of lubricating oil or grease. P. H. SNOW (B.P. 326,359, 19.3.29).—Equal amounts (*e.g.*, 12½ pts.) of castor oil and wool grease are mixed by gentle warming (60°), a much larger amount (75 pts.) of castor oil is added, and the mixture maintained at about 54–4° till evenly mixed.

E. LEWKOWITSCH.

Manufacture of products resembling linoxyn or rubber oil substitute. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,094, 17.9.28).—The unsaturated acids obtained by splitting off water from the oxidation products of paraffin hydrocarbons etc. (*e.g.*, by distillation) are esterified with, *e.g.*, glycerol, sorbitol; the esters so formed yield linoxyn-like products on heating in air with driers (*e.g.*, lead oxide, resinsates, linoleates) and rubber oil-like derivatives on treatment with sulphur.

E. LEWKOWITSCH.

Hydroxylated sulphonation product. B. KEISER (U.S.P. 1,745,221, 28.1.30. Appl., 21.1.29).—By sulphonating the fatty acids derived from linseed or perilla oils with sulphuric acid etc., at about 55°, neutralising, and washing in the usual way, a non-stringy, non-polymerised, water-soluble product of relatively low viscosity is obtained which is similar to Turkey-red oil.

E. LEWKOWITSCH.

Manufacture of new products resembling wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P.

324,631, 24.9.28).—Montan wax, bleached by oxidation, or a conversion product thereof (cf. B.P. 305,552, 308,996, and 320,854; B., 1929, 273, 425; 1930, 156) still containing free acids, together with other fatty, aromatic, or hydroaromatic acids (*e.g.*, coconut oil fatty acids, salicylic acid, naphthenic acids), are esterified with a polyhydric alcohol (or other polyhydroxy-compound which is liquid at the reaction temperature of 100–200°). Hydrocarbons or oily, fatty, waxy, or resinous compounds are added as desired. Any free acid remaining in the mixture may be converted into salts (*e.g.*, of calcium) or other compounds (esters, amides, etc.) wherein the carbonyl group still persists.

E. LEWKOWITSCH.

Emulsification of substances of wax-like character. DEUTS. HYDRIERWERKE A.-G. (B.P. 307,472, 8.3.29. Ger., 8.3.28).—Neutral waxes and hydrocarbons of high mol. wt. are melted with alcohols of high mol. wt. (solid at ordinary temperatures) and higher fatty or resin acids (*e.g.*, 65 pts. of spermaceti, 25 pts. of cetyl alcohol, and 10 pts. of stearic acids); the products form stable emulsions with water or dilute alkaline solutions.

E. LEWKOWITSCH.

Bleaching of beeswax. P. MAHLER, ASSR. to DARCO SALES CORP. (U.S.P. 1,739,796, 17.12.29. Appl., 4.12.24).—Melted beeswax is agitated with 12–35% of a mixture of vegetable char and acid-treated bentonite or other inorganic colour-absorbent, at temperatures between 75° and 100° for about 30 min., and filtered. The wax in the filter-cake is recovered by agitating with hot water, and added to the next batch of crude wax.

E. LEWKOWITSCH.

Preparation of concentrated cod-liver oil. K. KAWAI (U.S.P. 1,753,790, 8.4.30. Appl., 28.9.26).—See B.P. 283,265; B., 1928, 273.

Mixing apparatus (U.S.P. 1,745,291). **Presses** (B.P. 306,931 and 326,741). **Distillation of glycerin** (U.S.P. 1,743,289). **Mixing of liquids and gas** (U.S.P. 1,740,441).—See I. **Preparations for finishing textiles** (B.P. 291,094).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Basic lead sulphate. H. WOLFF [with G. ZEIDLER and B. ROSEN] (Farben-Ztg., 1930, 35, 1307–1309).—The results of a series of normal and accelerated weathering tests on paints based on basic lead sulphate (of German and other origin), American "blue lead," and basic carbonate white lead are illustrated and discussed. Contrary to earlier statements (*e.g.*, those of Schulz), basic lead sulphate was not found to be noticeably inferior to basic lead carbonate; indeed the former shows better retention of colour and, under some conditions, less chalking than the latter. Although shown to be inferior to standard basic lead sulphate, "blue lead" gives satisfactory durability when correctly used. Further points of interest arising in the exposures were the advantages of a red lead priming coat and of the introduction of stand oil in the finishing coat.

S. S. WOOLF.

Durability tests of spar varnishes. C. L. CAME (Bur. Stand. J. Res., 1930, 4, 247–259).—Kauri

reduction values and accelerated weathering tests were found to give the same relative order for 50 commercial varnishes as was obtained by outdoor tests.

C. J. SMITHELLS.

Testing the softening point of resins etc. WALKER.
—See II.

PATENTS.

Production of priming composition [for paints and varnishes]. BERGOLIN-WERKE VAN DER BERGH, KOMM.-GES. AUF AKT. (G.P. 459,652, 1.12.22. Addn. to G.P. 427,411; B., 1926, 761).—In addition to the usual filling materials, about 10% of neutral metallic compounds of fatty acids are introduced. D. F. TWISS.

Production of paints and lacquers. H. E. POTTS. From H. PLAUSON (B.P. 326,216, 6.9.28).—Rubber, balata, or the like is dissolved in an organic volatile solvent, *e.g.*, a low-boiling saturated hydrocarbon or chlorinated hydrocarbon, and vulcanised under pressure at about 125°. The vulcanised product is colloidalised by heating at about 180° under higher pressure and pigment is suspended in the dispersion so formed. Organic anhydrides, *e.g.*, acetic or phthalic, or alkali or alkaline-earth peroxides may be added to improve dispersion. The addition of resins, soluble in the dispersion medium, *e.g.*, hardened rosin or coumarone resin, increases the ease of application of the lacquers produced, which are of special value as ships' paints.

S. S. WOOLF.

Rendering paint waterproof. DEUTS. GASGLÜH-LICHT-AUER-GES.M.B.H. (B.P. 300,580, 5.11.28. Ger., 15.11.27. Addn. to B.P. 288,601; B., 1929, 924).—Paints containing added fungicide, as claimed in the prior patent, are used to give a finishing coat only.

H. ROYAL-DAWSON.

Manufacture of artificial resins. E. VON HERZ (B.P. 301,429, 29.11.28. Ger., 29.11.27).—Pentaerythritol or the water-soluble condensation product obtained as a by-product in the manufacture of that compound from acetaldehyde and formaldehyde is condensed with phthalic acid or anhydride, the sublimed phthalic anhydride is reintroduced, and heating is continued until sublimation ceases. The reaction may be continued until the resin is insoluble and infusible, or it may be interrupted at an intermediate stage of solubility, *e.g.*, when the resin is insoluble in alcohol, but still soluble in acetone.

S. S. WOOLF.

Manufacture of hardened artificial masses. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 298,085 and Addn. B.P. 326,214, [A, B] 1.10.28).—(A) Phenol-formaldehyde condensation products containing carboxylic acid groups (obtained by reaction between a phenol-formaldehyde resin and a halogeno-fatty acid or salt thereof or between a phenoxy-fatty acid and formaldehyde or a substance yielding formaldehyde) are converted wholly or partly into a salt or derivative of ammonia and heated at above 100° in presence or absence of a polyhydric alcohol, and with or without the addition of a filling material. (B) The intermediate ammonia treatment may be dispensed with when a polyhydric alcohol is present.

S. S. WOOLF.

Manufacture of [horn-like] condensation products of arylamines [with formaldehyde]. P. HALLER and H. KAPPELER (B.P. 306,972, 28.2.29. Switz., 29.2.28. Addn. to B.P. 266,358 and 274,501; B., 1928, 224, 792).—A soluble inorganic salt which does not react with formaldehyde, *e.g.*, sodium chloride, ammonium chloride or nitrate, or calcium chloride, is added to the acid condensation mixture of 1 mol. of arylamine and more than 1 mol. of formaldehyde, either at the beginning or before solidification of the mass. The product is washed with water.

C. HOLLINS.

Manufacture of drying varnishes. H. BECKER, ASSR. to SIEMENS & HALSKE A.-G. (U.S.P. 1,754,186, 8.4.30. Appl., 7.10.26. Ger., 20.5.25).—See B.P. 275,813; B., 1927, 822.

Manufacture of horn-like material. J. SCHLINCK and G. MÜNCHMEYER (U.S.P. 1,753,626, 8.4.30. Appl., 17.2.26. Ger., 21.2.25).—See B.P. 247,955; B., 1927, 306.

Chromium oxide (B.P. 302,178).—See VII. Rosin-core solder (B.P. 327,337).—See X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Cultivation and preparation of rubber in the United States. D. SPENCE (Ind. Eng. Chem., 1930, 22, 384—387).—The first steps for the cultivation of the guayule shrub, *Parthenium argentatum*, in the United States were taken in 1912. By suitable treatment of the seed, the proportion germinating has been raised from 5% to 96%. From about 1000 strains a few have now been selected for high rubber content, hardness, and adaptability to various conditions, the rubber content amounting even to 16%. Under normal conditions there is a progressive increase in the total amount of rubber per plant with each year of growth. In a year an acre of newly-sown seed may represent 1164 lb. of pure caoutchouc, a rate of production greatly exceeding that for an average *Hevea* plantation; the optimum growth for harvesting, however, is four years. The rubber is removed from the shrub by grinding in revolving tube-mills of the continuous-feed type; it is then screened, washed, and submitted to a second grinding. The rubber thus collected amounts to within 0.25% of the total rubber in the shrub. Guayule caoutchouc has the composition C_5H_8 , and shows the same behaviour as *Hevea* caoutchouc towards bromine, chromyl chloride, and vulcanisation. By submitting the shrub to a "retting" process, a product is obtainable still more closely resembling rubber from *Hevea brasiliensis* and, if desired, 75% of the acetone-soluble constituents can also be removed in this process, giving a crude rubber with only 5% of acetone-soluble matter. It is believed that guayule rubber will compete successfully in the United States with the product from the Far East, and the area of cultivated guayule is to be increased by 2500 acres annually.

D. F. TWISS.

Conditions affecting the vulcanisation of rubber.
IV. Heat reaction during vulcanisation. Y. TOYABE (J. Soc. Chem. Ind., Japan, 1930, 33, 96—98 B;

cf. B., 1930, 339, 249).—Rubber obtained from latex by precipitation with acetone was mixed with various proportions of sulphur (14.7—47.1%), and vulcanised at 155°. An embedded thermocouple showed an internal rise in temperature in each mixture, a maximum being reached in all cases after 60—70 min., when the ratio of free sulphur to total sulphur was about 60%. Vulcanisation at 170° led to occurrence of a maximum internal temperature in 20 min., the proportion of the sulphur then in combination being about 40—50% of the total present. Assuming that maximum heat evolution is an index of maximum reaction velocity, the independence of the latter of the temperature of vulcanisation and its occurrence when approximately half the sulphur has entered into combination suggest that maximum reaction velocity coincides with maximum disintegration of the rubber micelles. D. F. TWISS.

Comparison of various organic reagents for accelerating the vulcanisation of rubber. Y. TANAKA and G. HARA (Bull. Osaka Inst. Ind. Res., 1929, 10, 1—29).—The order of accelerating action, and optimal percentage (on rubber), of the substances examined were: piperidyl pentamethylenedithiocarbamate 0.25—0.5, *p*-nitrosodimethylaniline 0.25—1.0, diphenylguanidine 0.5—2.0, *p*-phenylenediamine 0.5—3.0, hexamethylenetetramine 0.5—4.0, triphenylguanidine 0.5—4.0, aldehyde-ammonia 0.75—4.0, thiocarbanilide 1.0—4.0. The breaking strength, elongation, hardness, and elasticity varied according to the accelerator used. The vulcanisation time is considerably shortened when a small, but often not when a large, quantity of reagent is employed. The sulphur may be decreased to 1% of the rubber. CHEMICAL ABSTRACTS.

PATENTS.

Preparation of pure caoutchouc from rubber latex. R. PUMMERER (G.P. 460,950, 26.1.26).—Rubber latex is caused to cream by treatment with dilute caustic alkali in the warm, preferably with absence of air; if desired, the cream may be re-diluted and re-treated. After removal of residual alkali, *e.g.*, by washing, filtration, dialysis, or centrifuging, pure caoutchouc is obtained which yields vulcanised products of super-normal technical qualities. D. F. TWISS.

Production of floating rubber resistant to mineral oil. TOKYO GOMU KABUSHIKI KAISHA (F.P. 634,628, 18.5.27).—An oil-resistant rubber of sp. gr. below that of water is produced by mixing rubber with factis, sulphur, alkali soap, and diphenylguanidine and vulcanising. Mineral fillers should not be used. D. F. TWISS.

Production of aqueous dispersions from rubber substitutes, more particularly factis. K.D.P., LTD. (B.P. 309,167, 15.3.29. Ger., 7.4.28).—Rubber substitutes are mixed with a peptising agent, such as gelatin, casein, or soap, on a roller-mill adapted to exert heavy pressure and thereby render the material plastic. As soon as intimate mixing has occurred, the material can be dispersed in water. D. F. TWISS.

Regeneration of vulcanised rubber. K.D.P., LTD., Assees. of METALLGES. A.-G. (B.P. 311,268, 15.3.29.

Ger., 8.5.28).—Vulcanised rubber is mixed with a peptising agent, *e.g.*, glue, saponin, soap, or casein (which, if desired, may be in solution), in a roller-mill or other suitable mixing apparatus adapted to exert substantial pressure and friction on the rubber. A product is obtained which on addition of water dissolves to a pulpy, finely-dispersed mass. D. F. TWISS.

Manufacture of accelerators for the vulcanisation of rubber and allied substances. S. J. PEACHEY (B.P. 326,256, 13. and 21.12.28, and 3. and 4.1.29).—Vulcanisation accelerators are prepared by heating *p*-nitrosodimethylaniline or one of its homologues, *e.g.*, *p*-nitrosodiethylaniline, with an arylamine of the benzene, naphthalene, or pyridine group and sulphur. The products are free from the objectionable staining properties of the parent nitroso-compound. D. F. TWISS.

Manufacture of synthetic rubber. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,869, 20.12.28).— α -Butadiene, or a homologue or derivative thereof, such as phenylbutadiene or styrene, is polymerised by an alkali metal in the presence of a small quantity of ammonia or a free cyclic or saturated aliphatic base (*e.g.*, 0.01—1% by wt. of the hydrocarbon to be polymerised). Substances favourable to the polymerisation, *e.g.*, ether, and diluents such as light petroleum may also be present. D. F. TWISS.

Manufacture of synthetic rubber. I. G. FARBENIND. A.-G. (B.P. 302,733, 20.12.28. Ger., 21.12.27).—Synthetic rubber-like masses are produced by separately polymerising quantities of the same butadiene hydrocarbon under different conditions and mixing the products. D. F. TWISS.

Manufacture of resilient [rubber] tyres and treads therefor. I. G. FARBENIND. A.-G. (B.P. 326,202, 30.8.28. Addn. to B.P. 299,037; B., 1930, 71).—In the manufacture of tyres from the plastic polymerisation product of butadiene, carbon-black is used as a compounding ingredient in addition to any other customary materials. D. F. TWISS.

Manufacture of rubber articles. MORGAN & WRIGHT, Assees. of (A) E. HAZELL, (B) A. W. KEEN (B.P. 307,789 and 307,792, 7.1.29. U.S., 13.3.28).—In the manufacture of rubber articles from aqueous dispersions of rubber by direct deposition of the rubber on to a form of the desired shape comprising a perforated metallic foundation with a coating of filtering material, (A) fabric or fibrous material such as paper pulp in one or more layers is interposed between the foundation and the external filtering coating which may consist of kieselguhr, clay, etc. Sufficient of this filtering composition is applied over the fabric layers to give a smooth continuous coating. (B) Using a similar arrangement, provision is made for heating the form (*e.g.*, by introduction of a heating medium) and the rubber deposited on it. The form may be immersed in the latex and withdrawn with a deposit of the desired quantity of rubber; heat is then applied for the substantial removal of water from the deposit, vulcanisation being completed subsequently. D. F. TWISS.

Production of rubber articles. DUNLOP RUBBER CO., LTD., C. HAYES, and E. A. MURPHY (B.P. 326,496, 14.11.28).—Rubber articles, particularly inner tubes or treads and other articles of irregular cross-section, are produced from aqueous dispersions of rubber or similar material in concentrated and/or compounded condition, by passing a mandrel downwards through a bath of thick creamy consistency, the thickness of the resulting coating being regulated by a spreading device, and then passing the coated mandrel downwards through an air-gap into a fixing bath containing, *e.g.*, a dehydrating and setting medium. Vulcanisation is effected subsequently. D. F. TWISS.

Manufacture of articles of rubber or similar material. DUNLOP RUBBER CO., LTD., D. F. TWISS, E. A. MURPHY, and W. G. THORPE (B.P. 326,282, 5.1.29).—Rubber articles of moderate thickness are produced by dipping a former into latex which preferably has been concentrated and/or compounded, and then bringing the uncoagulated deposit on the former into contact with an atmosphere containing, or consisting of, a volatile coagulant or with a dilute aqueous solution of such a coagulant. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., W. H. CHAPMAN, D. W. POUNDER, E. A. MURPHY, and F. T. PURKIS (B.P. 326,210, 7.12.28).—Silicofluorides have a variable effect on a dispersion of rubber or similar material, according to the proportion added. With a proportion in excess of a certain minimum, *e.g.*, 0.1–1% for a particular latex containing 60% of rubber and 0.5% of ammonia, the salts render the latex heat-sensitive, so that gelling occurs when the temperature is raised. With quantities below these, *e.g.*, 0.05–0.1%, the viscosity of the latex may be increased without inducing marked heat-sensitiveness. With greater proportions, *e.g.*, 1–5%, uniform gelling can be effected at ordinary temperature; the presence of zinc oxide assists the gelling process. The salts may be added as a powder or an aqueous dispersion. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., W. H. CHAPMAN, and D. W. POUNDER (B.P. 326,497 and 326,782, 14.11.28).—(A) Aqueous dispersions of rubber or similar material, compounded or otherwise, which have been rendered heat-sensitive, are brought to the desired shape, *e.g.*, by spreading, moulding, or electrophoresis, and are then gelled by contact with a fluid heating medium such as hot air or water. (B) Rubber sheet, tyre treads, and similar articles are produced by calendaring a heat-sensitive latex mixing between two non-adhesive sheets and causing the latex layer to gel by leading the sheets into a heating medium; after gelling, the rubber is removed from the sheets and dried. Using sheets which have not been pretreated to prevent adhesion, a double-texture, waterproofed material is obtained. By modified procedure it is possible to provide cloth or fabric with a permanent rubber coating on one or each side. D. F. TWISS.

Manufacture of rubber-fibre articles. W. B. WESCOTT, Asst. to RUBBER LATEX RES. CORP. (U.S.P. 1,737,133, 26.11.29. Appl., 12.7.27).—The cord fibre,

ranging from 0.5 to 1.5 in. in length, remaining after the mechanical removal of the bulk of the rubber from comminuted tyre scrap and containing, *e.g.*, 3–5% of adherent vulcanised rubber, is wetted thoroughly with latex to which a protective colloid such as haemoglobin may have been added. Fillers and vulcanising agents may also be present. The roughly moulded mixture is dried to a pervious mass and, preferably while still warm, is compressed in the final mould and vulcanised. The minimum proportion of latex rubber necessary for satisfactory results is 10–15%. D. F. TWISS.

Rubbering of fabrics containing copper and manganese. W. ESCH (G.P. 461,134, 11.11.26).—The fabric is given a preliminary coating of a rubber mixture resistant to copper and manganese and capable of being vulcanised at a moderate temperature; additional coats are then applied of a rubber mixture which can be vulcanised with a solution of sulphur chloride. A suitable copper- and manganese-resisting mixing comprises rubber, zinc oxide, lithopone, calcium carbonate, sulphur, cotton-seed oil, organic colour, and tetramethylthiuram disulphide. D. F. TWISS.

Preservation and packing of rubber goods. M. G. GRAICIEN (G.P. 461,060, 16.9.26).—Rubber articles such as finger-stalls are packed in air-tight containers filled with a mixture of water, glycerin, and borax, whereby the quality of the material is retained and after-vulcanisation prevented. D. F. TWISS.

Treatment of gutta-percha, balata, and like thermoplastic natural and artificial products and manufacture of moulded articles therefrom. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 326,481, 12.12.28 and 14.5.29).—Gutta-percha is subjected to heat treatment at a temperature below that causing melting or softening; this raises the normal melting or softening point and improves the mechanical, electrical, and water-resistant properties. Articles made of or containing gutta-percha, *e.g.*, insulated wires, may be so treated. On account of the induced hardening, the temperature of the heat-treatment may be raised gradually. Occasional "quenching" in cold water during the treatment accelerates the hardening process. D. F. TWISS.

Rubberising fibres (B.P. 325,916).—See V. **Electrical insulation** (U.S.P. 1,736,899).—See XI. **Rubber oil substitute** (B.P. 327,094).—See XII. **Paints and lacquers** (B.P. 326,216).—See XIII.

XV.—LEATHER; GLUE.

Effect of post-mortem action on the nitrogen distribution of animal skin. E. R. THEIS (J. Amer. Leather Chem. Assoc., 1930, 25, 92–102).—Small cubes of animal skin cut immediately after flaying were kept in stoppered bottles for definite periods at 5°, 20°, 30°, and 37.5°, respectively. There was a gradual formation of ammonia at 5°, which must be due to enzyme activity. A normal amount of ammonia was evolved at 20° in the first 48 hrs., after which there was a greater increase probably due to the combined activity of bacteria and autolytic enzymes. A much increased amount of ammonia was evolved at 37.5°. The presence

of very active autolytic enzymes in the skin was demonstrated. It was shown that the amide nitrogen increased progressively as the post-mortem period was increased, whilst the melanin nitrogen decreased. The amount of basic nitrogen was constant during the early stages, but diminished with more prolonged action. The total non-basic nitrogen remained practically constant, but the amino-nitrogen gradually decreased, thus indicating progressive deamination during post-mortem action.

D. WOODROFFE.

Formaldehyde tanning. B. ZIROULSKY (Halle aux Cuirs, Suppl. tech., 1929, 163—170; Chem. Zentr., 1929, ii, 2527).—The tanning action of formaldehyde is rapid; the absorption increases with the concentration of the formaldehyde and is markedly increased by the presence of alkali.

A. A. ELDRIDGE.

PATENTS.

Depilation of hides. E. A. TAYLOR, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,743,938, 14.1.30. Appl., 4.2.28).—Hides are treated with an aqueous solution prepared by dissolving in water an alkaline-earth oxide (25 pts. of lime), an alkaline sulphide (6.5 pts. of sodium sulphide), and an alkaline hydrosulphide (16.5 pts. of sodium hydrosulphide per 1000 pts. of green hide), the molecular amount of which is greater than that of the sulphide. A mixture of sodium sulphide and sodium hydrosulphide (mol. ratio of which is within the limits 1:2 and 1:5) may be used.

D. WOODROFFE.

Unhairing of hides or skins. A. E. WHITE. From TANNERS' COUNCIL OF U.S.A. (B.P. 327,132, 27.10.28).—Hides or skins are treated at about 20° with a saturated lime liquor, to which has been added about 1% of a primary alkylamine not containing other polar groups, e.g., ethylamine and/or methylamine.

D. WOODROFFE.

Treatment of skins or hides previous to tanning. W. H. ALLEN, ASSR. to LEATHER MAKERS' PROCESS CO. (U.S.P. 1,743,647, 14.1.30. Appl., 2.7.28).—Hides etc. are disinfected by immersion in a solution of sodium chloride and hydrochloric acid, the solution with the hides therein is neutralised with sufficient alkaline material, e.g., calcium hydroxide, to neutralise the free acid, and the hides are then removed from the liquor and washed free from soluble salts.

D. WOODROFFE.

Tanning of hides. HEIN & Co. (G.P. 459,617, 5.4.23).—Chlorine is passed into solutions of sulphite-cellulose waste liquor or ligninsulphonic acid until no more is absorbed, the excess chlorine is removed by treatment with alkali, and the product is combined with aromatic sulphonic acids, e.g., *p*-toluenesulphonyl chloride, and used for tanning hides.

D. WOODROFFE.

Tanning of hides. W. HILDT and R. MALACHOWSKI (G.P. 458,338, 9.3.23. Poland, 24.3.23).—The alkali naphthenates obtained by the alkali treatment of mineral oils are sulphonated, after which they may or may not be combined with acid resins and neutralised with alkali. The product, with or without the addition of other tanning materials, is used for tanning hides.

D. WOODROFFE.

Treatment of leather. B. RUBENS, ASSR. to A. BRICK, W. HALLNER, I. PRITSKER, S. KAGAN, N. L.

COHEN, and S. HOROWITZ (U.S.P. 1,744,506, 21.1.30. Appl., 27.4.27).—Ground oak bark (5 pts.) is extracted with boiling water (95 pts.), filtered, sodium hydroxide (0.25 pt.) is added, the solution allowed to cool, and the leather sponged or saturated with the solution and then moulded or shaped as desired.

D. WOODROFFE.

Tanning and other processes for treating solids with liquids. V. A. COLLINS (B.P. 327,146, 28.9.28).—Hides are suspended in tanning liquor in a closed container on the top of which is a series of cylinders, each containing a piston; the pistons are suitably reciprocated by means of a crankshaft to provide a continuous series of variations in the pressure in the container. A storage tank and pressure accumulator are connected with the container and the liquor is circulated by means of a pump.

D. WOODROFFE.

XVI.—AGRICULTURE.

Chemical composition of colloidal material isolated from the horizons of various soil profiles.

I. A. DENISON (J. Agric. Res., 1930, 40, 469—483).—A number of detailed chemical analyses are recorded. In some profiles the colloids of various horizons were of practically constant composition; in others wide variations existed. The most variable constituents were silica, iron, aluminium, organic matter, and combined water. Where the colloid composition changed with depth, variations in silica and aluminium contents were regular, but not always in the same direction in all soils. Weathering may not be the chief factor in reducing the silica: sesquioxide ratio of colloids, since in some instances this ratio decreased with the depth of horizon. Evidence was obtained of the presence of free alumina in the colloids of some of the lower (C) horizons.

A. G. POLLARD.

Composition of soils of the Nile delta. P. KOENIG (Ernähr. Pflanze, 1929, 25, 417—428; Chem. Zentr., 1929, ii, 2597).—The soils, which vary considerably in mechanical composition, seldom contain more than N 0.1, K₂O 0.5—1, CaO 3, P₂O₅ 0.2% (of which 10—20% is soluble in citric acid); the content of assimilable potassium is small.

A. A. ELDRIDGE.

Base exchange and acidity [in soils]. II. Preliminary experiments with permutits. P. VAGELER and J. WOLTERS DORF (Z. Pflanz. Düng., 1930, 16A, 184—204; cf. B., 1930, 295).—The results of experiments with soils and permutits are recorded in confirmation of the authors' mathematical treatment of the course of the exchange of calcium and hydrogen ions in relation to the anions with which they are associated.

A. G. POLLARD.

Relation of the amount and nature of exchangeable cations to the structure of a colloidal clay. L. D. BAUER (Soil Sci., 1930, 29, 291—309).—The properties of electro dialysed clay saturated to varying extents with different cations are examined. The reaction of clay sols containing different cations varied according to the lyotropic series Li > Na > K > Mg > Ca > H, whilst their specific conductivities were in the order Na > Li > K > Mg > Ca, and flocculation values in the order Li = Na > K > Mg = Ca. Univalent

cations increased the charge on the particles to a maximum at the saturation capacity of the colloid. Still higher concentrations of ions decreased the charge. Bivalent cations decreased the charge. The average radius of the aggregates varied with the cation from 136 $\mu\mu$ for hydrogen to 148 $\mu\mu$ for calcium. Pore sizes in calcium-clay were larger than in a sodium-clay. Addition of increasing amounts of univalent cations to clay at first decreased its viscosity, which subsequently increased to a maximum corresponding to the saturation point of the clay with bases. Still higher proportions of cations caused a sharp decline in viscosity, followed by an increase at the point of flocculation. Bivalent ions decreased the viscosity of clay to the point of flocculation, where a sharp increase occurred. The hydration and viscosity of the aggregates were in the order $\text{Li} > \text{Na} > \text{K} > \text{H} > \text{Ca} > \text{Mg}$. Changes in these properties of clay sols are associated with the number of "active" ions on the particle (i.e., dissociated ions existing on the particle surface as distinct from those of the undissociated clay "salts"). The size, stability, and low hydration of the calcium and magnesium aggregates are important for maintaining good soil structure.

A. G. POLLARD.

Replaceable bases of irrigated soil. W. H. METZGER (Soil Sci., 1930, 29, 251—260).—The proportions of replaceable bases in a soil in the air-dry, moistened, and flooded condition are compared. No appreciable change in the amount of replaceable sodium and potassium with moisture conditions was observed. Flooding largely increased the replaceable calcium, magnesium, aluminium, iron, ammonia, and manganese. Moistening to 20% produced increases only in the exchangeable manganese and magnesium. Comparison is also made of the distribution of replaceable bases in the various horizons of irrigated and non-irrigated soils.

A. G. POLLARD.

Transition of quicklime and its influence on the solubility of phosphoric acid and potash in heavy soils. H. HEUSER (Z. Pflanz. Düng., 1930, 16A, 204—226).—Changes resulting from the addition of quicklime to soils are examined by electro-ultrafiltration methods. In heavy soils quicklime is not converted quantitatively into calcium carbonate. Quicklime does not affect the solubility of soil potash. The easily soluble phosphate content of some soils was decreased by liming, but in others the transition of insoluble to easily soluble phosphate was increased. Electro-ultrafiltration methods may be used to follow the initial podsolisation processes in soils, which cannot be detected by macroscopic processes.

A. G. POLLARD.

Colorimetric determination of soil reaction. C. J. SCHOLLENBERGER (Science, 1930, 71, 220).—A simple form of percolator giving a clear water extract of soil is described.

L. S. THEOBALD.

Biochemical researches on soil. A. J. J. VANDE VELDE and A. VERBELEN (Compt. rend., 1930, 190, 977—979).—The method devised by Skar (B., 1929, 263) for the determination of the number of micro-organisms in milk has been applied to soil. The earth is weighed aseptically and shaken with sterile milk for 30 min.

in the presence of 4% formaldehyde. No relationship was established between the p_{H} , which varied from 5.11 to 7.95, and the number of micro-organisms, which varied from 1280 to 21,600 millions per g. of the soil from 30 fields. Cultivation of barley or radish in the same soil, with or without sterilisation or treatment with lime, showed that the number of micro-organisms diminished during germination, except where sterilised soil was used, in which case the number increased. The p_{H} increased in the cultures of the radish and decreased in those of the barley. Liming raises the alkalinity of the soil, but the reaction of the plant juices is dependent more on the life of the plant than on the reaction of the substrate.

C. C. N. VASS.

Effect of leaking natural gas on the soil. C. J. SCHOLLENBERGER (Soil Sci., 1930, 29, 261—266).—The exchangeable manganese content of soil is considerably increased by the leakage to it of natural gas. There is a smaller increase in exchangeable ammonia and a decrease in hydrogen-ion concentration. It is suggested that the gas displaces air from the soil and the resulting biological reduction processes tend to render active the higher oxides of manganese known to be present in the soil. Aeration of the soil leads to rapid reversion of the manganese to an inactive form and subsequent plant growth is normal.

A. G. POLLARD.

Soil and crop studies with ammonium sulphate. A. L. PRINCE and A. W. BLAIR (Soil Sci., 1930, 29, 267—279).—Pot cultures of barley, rape, and lettuce fertilised with varying amounts of ammonium sulphate are described. On a heavy loam rape and barley were not injured by heavy dressings (up to 1800 lb./acre) of ammonium sulphate, and the nitrogen content of the crop was increased. On sandy soil dressings exceeding 350 lb./acre caused decreased crops and poor germination. Below this limit the dry weight and nitrogen content of the crops were increased by ammonium sulphate. With the larger amounts of ammonium sulphate applied, the p_{H} value of the soils decreased to an extent which was greater on heavy soil than on sand. Plant injury, however, was more marked on the lighter soil. The p_{H} values of treated soils were found to be correlated with the proportion of "active" aluminium present. The hydrolysis of the aluminium sulphate produced by base exchange probably explains the changes in reaction.

A. G. POLLARD.

Action of ammonium salts [on plants] in relation to hydrogen-ion concentration. H. ENGEL (Z. Pflanz. Düng., 1930, 16A, 226—233).—The work of Mevius and Engel (cf. B., 1928, 278, and Planta, 1929, 9, 1) is further discussed. Addition of ammonium salts of the stronger acids to plant-nutrient solutions results in the penetration of hydrolytically produced ammonia into the root cells, in quantities increasing with the p_{H} value and ammonia tension of the solutions. Within the root cell the ammonia is neutralised by organic acids and its subsequent conversion into asparagin, amino-acids, and proteins is controlled by the carbon dioxide assimilation of the plant. Because of its physiologically amphoteric nature, ammonium nitrate is suitable for the study of the ammonia absorption of plants, in which no "acidosis" (Pranishnikov) is caused.

A. G. POLLARD.

Determination of inorganic nitrogen in dried plant tissue. A. C. SESSIONS (Soil Sci., 1930, 29, 285—289).—The method of Sessions and Shive (A., 1929, 960) is modified to deal with dried plant matter. The sample is ground to pass 30-mesh and 2–5 g. are placed in a 300-c.c. aspirating flask with 70 c.c. of water. After setting aside for 1 hr. with occasional shaking, material adhering to the sides of the flask is washed down with not more than 30 c.c. of water. Then 15 g. of sodium chloride and 3–4 g. of Devarda's alloy (100-mesh) are added and sufficient sodium hydroxide to make the solution approx. N/7. Air is drawn through the apparatus for 12 hrs., the ammonia being trapped in acid. Frothing is checked by the addition of 2–3 c.c. of light paraffin oil or 2–3 drops of decyl alcohol. A. G. POLLARD.

New basis for fertiliser experiments. W. J. SPILLMAN (Science, 1930, 71, 135—136).—In field experiments on a soil containing n , p , and k available units per acre of nitrogen, phosphoric acid, and potash, respectively, the yield per acre $y = A(1 - R_1^{n+x_1})(1 - R_2^{p+x_2})_x \times (1 - R_3^{k+x_3})$, where A is a maximum yield constant, x_1 , x_2 , and x_3 are quantities of growth factors, and R_1 , R_2 , and R_3 are their related constants. L. S. THEOBALD.

Comparative [fertiliser] values of different phosphates. C. A. MOOERS (Tenn. Agric. Exp. Sta. Bull., 1929, No. 141, 1—18).—Basic phosphates were suitable for unlimed land and were superior to superphosphate. Under liming, Thomas slag and superphosphate were of equal value. Rock phosphate was in both cases inferior. The test crops were maize, oats, potatoes, and hay. CHEMICAL ABSTRACTS.

Effect of burning on the accumulation of organic matter in forest soils. R. M. BARNETTE and J. B. HESTER (Soil Sci., 1930, 29, 281—284).—Soil analyses show that considerable losses to forest soils of organic matter and nitrogen are incurred by the periodic burning of undergrowth etc. A. G. POLLARD.

Preparation and effectiveness of basic copper sulphate as a fungicide. E. B. HOLLAND, C. O. DUNBAR, G. M. GILLIGAN, and W. L. DORAN (Mass. Agric. Exp. Sta. Bull., 1929, No. 252, 124—149).—Commercial basic copper sulphate was effective for cucumbers and celery. The concentration of copper necessary may be greater than that in Bordeaux mixture owing to lower dispersion. The concentration of basic sulphate necessary in sprays differs from that in dusts owing to the large proportion of inert vehicle in the latter. For sprays the following concentrations of copper and arsenic, respectively, are recommended: apples and grapes 0.05, 0.07; cucumber 0.025, 0.09; potatoes 0.30, 0.09; celery 0.30%. — CHEMICAL ABSTRACTS.

Determination of the content of toxic substance in insecticides etc. V. Volumetric determination of formaldehyde in seed fungicides. J. BODNÁR and W. GERVAY (Z. anal. Chem., 1930, 80, 127—134; cf. B., 1928, 619).—In fungicides containing phenol, glycerol, soaps, or sodium chloride, formaldehyde may be determined directly by the hydrogen peroxide method of Blank and Finkenbeiner (B., 1899, 614) or by the sulphite method of Lemme (cf. B., 1903, 1107). If the material contains mercuric chloride, 5 g. of sodium

chloride are added to the solution and the formaldehyde is separated from the mercury by distillation, the distillate being analysed by either of the above-mentioned methods. A. R. POWELL.

Pneumatic transport. NEU.—See I. **Removal of ions from calcium phosphate solutions by gels.** GHOSH and BHATTACHARYYA.—See VII. **Cultivation of rubber.** SPENCE.—See XIV. **Plant colloids.** KAVČIČ.—See XVII. **Ripening of fruits.** OVERHOLSER. **Foods treated with arsenical preparations.** VON FELLEBERG.—See XIX.

PATENTS.

Insecticides. A. CARPMAEL. From I. G. FARBEN-IND. A.-G. (B.P. 325,910, 12.12.28).—Benzene derivatives containing one or more methoxyl groups and at least 2 thiocyno-groups are used for the destruction of plant lice. Examples are: 4-methoxy-*m*-xylylene dithiocyanate, m.p. 71—72°, 2:5-dimethoxy-*p*-xylylene dithiocyanate, m.p. 173°, and 6-chloro-4-methoxy-*m*-xylylene dithiocyanate. The product may be dissolved, with an acetone-soluble wetting agent, in acetone and diluted with water to give an emulsion ready for use. C. HOLLINS.

Insecticide and fungicide. R. H. MCKEE (U.S.P. 1,744,324, 21.1.30. Appl., 28.3.25).—The shale oil fraction distilling at 150—300°, consisting of bases and saturated and unsaturated hydrocarbons, is emulsified with soap solution; the product on dilution with water is used for spraying plants or for impregnating wood. L. A. COLES.

Manufacture of insecticides. W. H. GROOM-BRIDGE, Assr. to A. J. DICKINSON, LTD. (U.S.P. 1,753,887, 8.4.30. Appl., 23.11.27).—See B.P. 301,186; B., 1929, 106.

XVII.—SUGARS; STARCHES; GUMS.

Purification of sugar-cane juice. A. LITJAGO (Weissruss. Staatl. Akad. Landw., 1929, 7, 315—320; Chem. Zentr., 1929, ii, 2611).—By the use of magnesium sulphate in the clarification of diffusion juice according to the equation $C_{12}H_{22}O_{11} \cdot CaO + MgSO_4 + H_2O = C_{12}H_{22}O_{11} + CaSO_4 + Mg(OH)_2$, almost complete separation of the organic, and to some extent of the inorganic, non-sugar substances takes place owing to the formation of colloidal magnesium hydroxide. The sugar, however, contains 0.4% of calcium sulphate. A. A. ELDRIDGE.

Determination of the sugar content of carbonated scums. F. HERLES; also J. HAMOUS (Z. Zuckerind. Czechoslov., 1929, 54, 23).—Polemical (cf. Kadlev, B., 1930, 29).

Free water necessary to change β -anhydrous lactose into α -hydrous lactose. R. W. BELL (Ind. Eng. Chem., 1930, 22, 379—380).—Of the three known forms of lactose, viz., α -hydrate, α -anhydride, and β -anhydride, the first-named is stable at temperatures below 93° in the presence of small amounts of water (cf. Hudson and Brown, B., 1908, 699, 1169). Experiments are described in which the amount of free water necessary to convert the β -anhydride into the α -hydrate has been determined. This was found to be between 0.5 and 1.0%. H. INGLESON.

Plant colloids. XXV. Potato starch from different varieties of *Solanum tuberosum*. J. KAVČIČ (Kolloidchem. Beih., 1930, 30, 406—415).—Using potato starch from four different varieties of *Solanum tuberosum*, measurements were made of the mean diameter of the grains, water content in the air-dried state, ash content, content of phosphoric acid, swelling temperature, viscosity, electrical conductivity, hydrogen-ion concentration, capacity for iodine adsorption, optical rotatory power, aggregate weight, and fraction diffusible through a collodion membrane. Different values of these properties were obtained for the different varieties and the variations were consistent, indicating that potato starch cannot be regarded as a definite colloid-chemical individual. E. S. HEDGES.

Yield of potato starch. W. BIELICKI (Przemysl Chem., 1920, 14, 145—149).—Maximum yields of starch from potato flakes were obtained at p_H 5.8. The yields obtained from flakes containing 18.4—35.5% of water are the same, provided that the duration of drying does not exceed 30 min. at a sufficiently low temperature, and that the water used for extraction of the starch has a low mineral content. R. TRUSZKOWSKI.

Soluble starch. H. INUKAI (Bull. Kyoto Ind. Res. Inst., 1927, 1, 1—23; 1928, 3, 13—42).—The properties of soluble starch prepared by various methods are recorded. The use of glacial acetic and concentrated sulphuric acids results in partial esterification but gives a good product, whilst bleaching powder causes oxidation. CHEMICAL ABSTRACTS.

Polysaccharide of larchwood. WISE and PETERSON.—See V.

PATENT.

Glycerol from dextrose (B.P. 299,373).—See III.

XVIII.—FERMENTATION INDUSTRIES.

Simple connecting device for the pure culture of yeast. H. FINK and R. KÜHLES (Woch. Brau., 1930, 47, 152—153).—Two flasks, of 1 and 5 litres capacity, respectively, with side tubes similar to those of Pasteur flasks, are connected by short rubber tubes and a T-piece. The rubber connexions can be closed by screw clips, and the other branch of the T-piece by a clip and a glass stopper. The larger flask is filled with wort, a small quantity is placed in the smaller flask, and the whole sterilised. The smaller flask is opened once for inoculation, and the T-piece filled with alcohol and closed. When fermentation is well established in the smaller flask, this alcohol is removed, the clips between the two flasks are opened, and wort is run from the large to the small flask. Portions of culture can be drawn off through the T-piece without risk of infection, or the contents of the large flask can be inoculated to obtain a culture for inoculating technical fermentations. F. E. DAY.

Staining of yeast by methylene-blue and its relation to hydrogen-ion concentration and the problem of permeability. H. FINK and F. WEINFURTER (Woch. Brau., 1930, 47, 89—93, 110—116, 124—127).—The proportion of cells stained and the

intensity of staining are greater in suspensions in alkaline than in acid media. The effect is more marked in dilute solutions of electrolytes than in wort. Acid media extract the dye from stained cells more readily than do alkaline media. Yeast washed with distilled water and suspended in conductivity water is "sensitised" to staining, and a similar result is obtained if the latter medium be replaced by 20% solutions of dextrose, levulose, or sucrose in distilled water. Maltose (2 specimens) was less effective, glycerin much less, and mannitol had no effect. The presence of salts protects the cells against "sensitisation" even in the concentration present in (Munich) supply water, and treatment with such dilute salt solutions after sensitisation and before staining destroys the sensitisation. The production of carbon dioxide in fermentation experiments is affected in a similar manner, parallel fermentations in media prepared with distilled and supply water giving 7.7 and 14.4 c.c. of carbon dioxide, respectively, in absence of methylene-blue and 1.1 and 9.4 c.c. in presence of the stain. Pure water and carbohydrates appear to increase the permeability of the cell, and thus to facilitate the entry of the dye, which then acts as a poison. This has been confirmed by plate counts of variously treated yeasts, and a parallel is drawn between these results and the protective action of salts against the yeast poison of wheat flour, especially as bakers' and factory yeasts are less susceptible of "sensitisation." In this respect yeasts show considerable variation, and this may be associated with high salt content of the previous culture medium, and consequent storage of salts by the cells. The relative protective action of a number of salts has been studied, and it appears that the differences are relatively small, but that if the effects of the ions are separated they tend to follow Hofmeister's series; e.g., the degree of protection increases from lithium through sodium and potassium to ammonium. Reference is made to work in hand on the influence of peptones, and on a modification of the staining test for yeast, and the possible effects of the protective action of salts in fermentation technology is discussed (cf. B., 1929, 792, 1028). F. E. DAY.

Graphical evaluation of sieve analyses [of grist]. O. HALTMEIER (Woch. Brau., 1930, 47, 133—137, 149—151).—If the results of sieve analyses are plotted graphically—percentage of residue against diameter of perforations—curves are obtained which are independent of the sieves employed. By comparing the curves given by the products from each pair of rolls, the effect of each on the final grist can be studied and the grinding correctly adjusted. F. E. DAY.

Separation of the agitation effect in beer fermentation into aeration and effective yeast-surface factors ("spärende Wirkung"). F. WINDISCH (Woch. Brau., 1930, 47, 94—96).—Wort agitated during fermentation gives a higher degree of attenuation even when the aeration effect is eliminated by increasing the pitching rate till no yeast increase occurs. At normal pitching rates the addition of 20 g. of wood-wool to 1.5 litres lessens the difference of attenuation between unagitated and agitated fermentations. In the presence of wood-wool and at high pitching rates, stirred and

unstirred fermentations reach the same degree of attenuation.

F. E. DAY.

Gasometric method for the determination of carbon dioxide in beer. H. LUNDIN, O. ÖHLIN, and J. EILBURG (*Woch. Brau.*, 1930, **47**, 121—124, 137—140).—The method previously described (*B.*, 1928, 725) has been improved. The Van Slyke apparatus and that for rendering the beer alkaline without escape of gas have been modified in certain details, and a borer for piercing crown-cork capsules and withdrawing the contents of the bottle quantitatively is described. The contents of the bottle are rendered alkaline and made up to 750 c.c., and the carbon dioxide liberated on acidifying 1 c.c. of this is measured, which may be done in one extraction if the re-adsorption factor for the Van Slyke apparatus is known. This may be determined by comparing the results on a few beers with those obtained by Langer and Schultze's method, or by making a second extraction. For measuring under reduced pressure the small volumes of gas obtained in the latter case, a device consisting of a light rod supported vertically by a float on the mercury reservoir and carrying a pointer 500 mm. above the mercury surface is described. When the pointer is adjusted to the corrected level of the mercury in the pipette, the gas is at a pressure of 500 mm. below atmospheric pressure. Full details of technique and calculation are given, together with examples of the increase of the amount of carbon dioxide in beer during fermentation and storage.

F. E. DAY.

Importance of acidity in brewing processes. P. KOLBACH (*Woch. Brau.*, 1930, **47**, 145—149, 163—168).—A review and discussion of recent work. Since a correct acidity is of importance at every stage of the brewing operations, artificial acidification of mash or wort is specially considered. In the author's view, the former is best achieved by addition of cultures of *B. Delbrücki* or, alternatively, of lactic acid to the mash rather than to the water. Acidification of the wort improves colour and economises hops without affecting the buffering.

F. E. DAY.

Change of buffer action on saccharisation of rice malt. K. MATSUMOTO and Y. KISE (*Bull. Res. Inst. Fermentation*, 1929, No. 102, 63—84).—When rice malt is saccharised at 55° the buffer action of the solution first increases and then decreases (apparently owing to the presence of lactic acid bacteria), the *pH* remaining constant. The buffer action differs with different acids, and is weaker towards alkalis than towards acids. The titratable acidity normally increases gradually during saccharisation. The buffer capacity is increased by addition of calcium salts, and is maintained constant by addition of lactic acid. When soluble starch or rice paste is saccharised by diastase or malt extract the buffer capacity remains constant.

CHEMICAL ABSTRACTS.

Investigation and evaluation of wine distillates and wine brandies. II. G. BÜTTNER and A. MIERMEISTER (*Z. Unters. Lebensm.*, 1929, **58**, 628—635; cf. *B.*, 1929, 373).—Eight French wines, compounded with wine distillates, have been subjected to analysis, with particular reference to their contents of

aldehydes, esters, and higher alcohols. The samples containing significant amounts of furfuraldehyde were low in acetaldehyde, whereas those containing no furfuraldehyde had a high acetaldehyde content. By studying the course of the distillation of wine, it was found that acetaldehyde distils early and furfuraldehyde largely at the middle of the run, but the esters and higher alcohols distil more or less regularly and no appreciable partition is effected. The opinions of Wüstenfeld and Luckow (cf. *B.*, 1929, 574) and of Ruppig (cf. *B.*, 1929, 573), that tasting should be an essential part of the testing of wine brandies and distillates, are further criticised.

H. J. DOWDEN.

Composition of wines from Baden, with special reference to their arsenic content, and the use of Bezssonoff's reagent for the detection of vitamin-C. E. REMY and F. RICHTER (*Z. Unters. Lebensm.*, 1929, **58**, 624—628).—A systematic analysis of eight wines of different origin has shown that arsenic is not a normal constituent and occurs only in insignificant amounts. The authors have confirmed the observation of Glassmann and Posdeev (cf. *B.*, 1929, 575) that tannic acid gives Bezssonoff's reaction, and a further colorimetric test has been evolved for vitamin-C in the presence of tannin. When lemon juice treated with Bezssonoff's reagent, after keeping for 1 hr., is made feebly alkaline with 10% caustic soda and shaken, the violet colour changes to blue, which develops a violet tinge on re-acidifying carefully with 10% hydrochloric acid. Tannin solution with the reagent, when made alkaline, turns reddish-yellow, with a further change to green on re-acidifying. When a mixture of lemon juice and tannin solution is similarly treated, the colours produced are blue-green and dirty green, respectively. That the reaction is a test for vitamin-C has been confirmed by experiments on other antiscorbutic materials such as the juice of oranges, tomatoes, vegetables, etc.

H. J. DOWDEN.

Improved manufacture of absolute alcohol. H. GUINOT (*Z. Spiritusind.*, 1930, **53**, 101—102).—Absolute alcohol can be obtained directly from fermented liquids by the azeotropic method of distillation with the same expenditure of steam as is required to manufacture raw spirit, the costs of the two processes being about equal. Most of the water is separated, together with the slop, from the fermented liquid at the foot of the first of three distillation columns. Further amounts are removed at the bottom and top of the concentration column in which the dehydration hydrocarbon functions. In this column there is an intermediate zone from which the vapour consisting of 98.5—99.0% of alcohol mixed with a small amount of the dehydration liquid is led to the third column, which is smaller. The remaining moisture and hydrocarbon are removed and 99.8% alcohol is collected at the foot of the column. Dehydration is accompanied by purification, and the final alcohol is almost free from aldehyde and ethers.

C. RANKEN.

Examination of brewery products with ultra-violet light. T. KOANA (*Bull. Res. Inst. Fermentation*, Japan, 1929, **102**, 85—118).

Biochemistry of breadmaking. ELION. [Alcohol from] rice husks. DE MONCEOT.—See XIX.

PATENTS.

Fermentation process. W. L. OWEN, Assr. to CITIZENS OF U.S.A. (U.S.P. 1,744,001, 14.1.30. Appl., 21.12.27).—The seed yeast is retained for about 30 min. in the seed vat in contact with an amount of vegetable carbon equal to 5% by vol. of the wort to be fermented. The vat is then filled with freshly sterilised wort and, after the density of the wort is reduced by one half, the entire contents of the seed vat are transferred to a fermenter which is operated at 30–40° Brix until the fermentation is complete. C. RANKEN.

Treatment of wine residues. H. L. RICARD (F.P. 631,824, 30.3.27).—The wine residues, after pressing, are dried by an air current at 80–100°, and broken up into the separate constituents (skins etc.). C. RANKEN.

Jellification and solidification of alcoholic beverages, medicines, and alcohol. C. A. and G. COFMAN-NICORESTI (B.P. 326,447, 10.12.28).—Jellification etc. is effected by means of an alcoholic solution of pectin; gum arabic and gum tragacanth may also be added.

H. ROYAL-DAWSON.

Cooling of metal vessels (Austr.P. 109,381).—See X. **Fats from grape marc** (F.P. 635,628).—See XII.

XIX.—FOODS.

Storage of food-grain. G. R. DUTT and A. N. PURI (Agric. J. India, 1929, 24, 245–250).—Attack of wheat by insects is prevented in closed containers by the presence of a small quantity of mercury or tin amalgam; no appreciable amount of mercury is absorbed by the wheat, the germinating power of which is not depressed.

CHEMICAL ABSTRACTS.

Detection of flour bleached with chlorine or oxides of nitrogen. J. KULMAN (Z. Unters. Lebensm., 1929, 58, 635–649).—Solutions of potassium chromate or dichromate having been found unsuitable for colorimetric measurements on benzene extracts, a graduated scale was prepared from the most strongly coloured flour extract by dilution in stages of 10% with benzene, each dilution being evaluated in terms of a dye, with which the undiluted extract (No. 1 on the scale) was compared. Freshly milled flour had a colour value of 1–2, whilst flours bleached by chlorine or oxides of nitrogen had values between 5 and 9. To differentiate between chemically bleached flours and those bleached by natural ageing, it was found that when the benzene extract was evaporated at 110–115°, that from unbleached flour retains its original yellow colour, whilst that from chemically bleached flour changes to a reddish-brown. Spectroscopic investigations failed to reveal any differences in the absorption spectra of bleached and unbleached flours, as might be expected if the former contained nitro- or chloro-derivatives of carotene, and accordingly the behaviour of the extracting medium was examined. It was found that the brown coloration produced by evaporation of the extract at 110–115° was due to the action of chlorine or nitric oxide on toluene or xylene. Accordingly, it is recommended that the sample of flour (50 g.) be extracted with a mixture of benzene 85 c.c., toluene 10 c.c., and xylene 5 c.c., followed

by evaporation of the extract at 110–115° if the colour value on the scale described is within the limits 5–9. A brown coloration denotes a flour that has been bleached by chlorine or nitric oxide. H. J. DOWDEN.

Biochemistry of breadmaking. L. ELION (Chem. Weekblad, 1930, 27, 219–227).—The fermenting powers of brewers' yeast and bakers' yeast at different temperatures and for different flours are compared. The various factors influencing the fermentation and the baking are considered, and the importance of actual baking for testing purposes is stressed. The influence of bleaching agents, and of calcium and ammonium dihydrogen phosphates, is discussed. S. I. LEVY.

Utilisation of rice husks. L. B. DE MONGEOT (Giorn. Chim. Ind. Appl., 1930, 12, 74–76).—Rice husks consist of 60.04% of volatile matter, 24.79% of carbonaceous matter, and 15.17% of ash. Analysis of the ash gives SiO₂ 93.95, Fe₂O₃ 0.01, CaO 2.28, K₂O 3.15%, Mn₂O₄ and Al₂O₃ traces. The use of this silicious ash is suggested in the glass industry, the estimated annual production in Italy being 16,900 tons. Dry distillation of the husks gives a carbon of low density, and a liquid in which acetic and propionic acids, furfuraldehyde, phenol, and cresols were identified. Microscopical examination of the husks show them to be rich in cellulose, and they are suggested as a source of industrial alcohol. F. G. TRYHORN.

Milk. IV. Effect of heating on the constituents of milk. I. MATSUO (Osaka J. Med., 1929, 28, 555–562).—Raw and sterilised milk contained, respectively, total nitrogen 0.47, 0.449; albumin 0.410, trace; globulin 0.022, 0.014; caseinogen 0.370, 0.398%. Human milk (4–5 days after parturition) contained 1.41–1.66 mg. of ferric oxide per litre, in 13–44 days 1.12–1.57 mg., in 201–218 days 1.05–1.43 mg. Raw and boiled cow's milk contained 2.02 mg. (0.33 mg. only in a glass receiver); goat's milk contained 1.17 mg.

CHEMICAL ABSTRACTS.

Heat-coagulation of milk as a function of acidity. A. TAPERNOUX and K. KATRANDEY (Compt. rend. Soc. Biol., 1929, 101, 828–829; Chem. Zentr., 1929, ii, 2953).—A definite relationship was observed.

A. A. ELDRIDGE.

So-called low pasteurisation of milk. H. ZELLER, W. WEDEMANN, L. LANGE, and E. GILDEMEISTER (Arb. Reichsgesundh.-Amt, 1929, 61, 1–72; Chem. Zentr., 1929, ii, 2953).—Chemical and bacterial changes are recorded; the acidity is diminished, albumin and globulin are partly coagulated, the casein is altered, the catalase diminished, and the diastase destroyed.

A. A. ELDRIDGE.

Analysis of sheeps'-milk cheese produced in the Tatra mountains. H. RUEBENBAUER (Przemysl Chem., 1930, 14, 169–178).—The fat content of the milk of sheep bred in the Tatra mountains varies from 6.86% in May to 9.12% in August, whilst the content of solids-not-fat remains constant (10.99–11.33%). An analysis of cheese made from the milk gives (%): water content 36.81, fat 33.5, ash 4.54 (including 2.47% NaCl), casein 15.17, albumin 1.32, amino-compounds 6.69, diamino-compounds 0.72, ammonia 0.08, and lactose

1.17%. The fat has m.p. 28.2°; f.p. 18.9°; critical dissolution temperature (alcohol) 53°; refractivity 44.1°; acidity corresponding with a free oleic acid content of 0.7191%; saponif. value 229.1; soluble and insoluble volatile acid value, respectively, 28.2, 3.8; non-volatile acid content 86.4%; iodine value 35.2; thiocyanogen value 35.2; octoic acid value 2.4; lauric, stearic, and oleic acid contents, respectively, 6.95, 4.7, 38.2%; palmitic and myristic acid content 33%; unsaponifiable residue 1.8%. The fat gave a positive aldehyde reaction and a blue luminescence on exposure to the mercury-vapour lamp. Sheeps' cheese thus differs from that of cows chiefly in its albumin content and in the properties of the fat, which is distinguished by its low refractive index, high ratio of soluble to insoluble volatile fatty acids, and by the high saponification value. The microscopical flora of sheeps' cheese consists chiefly in *Oidium lactis*, together with *Penicillium glaucum*, *B. lact. acid.* *Leichm.*, and *B. casei*.

R. TRUSZKOWSKI.

Determination of moisture in cheese. F. H. McDOWALL (New Zealand J. Sci. Tech., 1930, 11, 293—294).—A suggested standardised procedure as to (a) sampling, (b) preparation of samples, and (c) determination of moisture in cheese by direct heating in aluminium or nickel dishes, as recommended by the New Zealand Dairy Science Association, is outlined.

B. W. TOWN.

Effects of temperature on the ripening and keeping of fruits. E. L. OVERHOLSER (Proc. Int. Cong. Plant Sci., 1929, 2, 999—1011).—The course or character, as well as the rate, of metabolism, both in storage and in the field, may be modified by temperatures above the optimal. CHEMICAL ABSTRACTS.

Arsenic content of foods and of foods treated with arsenical preparations. T. VON FELLEBERG (Biochem. Z., 1930, 218, 300—317).—Ordinary foods and drinks contain only harmless amounts of arsenic, and no danger is to be feared from arsenic consumed with fruit which has been sprayed with arsenical compounds, although lead from lead arsenate sprays is a source of danger.

W. McCARTNEY.

Influence on the softening of peas of calcium and magnesium hardness of the cooking water. A. MÜLLER (Z. Unters. Lebensm., 1929, 58, 608—623).—An instrument has been devised by which the hardness of peas may be measured by the loading required to drive a needle (0.7 mm. in diam.) through the peas. By means of this apparatus a study has been made of the rate of softening of peas, previously swollen, when boiled in distilled water and in water of various degrees of hardness due to magnesium chloride, calcium chloride, or calcium bicarbonate. An increase in the time required by the peas to become tender was caused by all types of hard water, the influence of calcium hardness being more pronounced than that of magnesium. The observations support the hypothesis of van der Marel (cf. Pharm. Weekblad, 1922, 59, 82) that sparingly soluble pectin compounds are formed with the salts present in the water.

H. J. DOWDEN.

Electrolytic determination of copper in preserved vegetables. J. W. SPRINGER (Z. Unters. Lebensm.,

1929, 58, 651—652).—The copper salts used for conserving the green colour of preserved peas, beans, etc. may be quickly determined by heating a sample of the pulped material (100 or 150 g.) on a water-bath with 10 c.c. of nitric acid (*d* 1.4). To the mash, made faintly alkaline with ammonia, are added 20 c.c. of sulphuric acid (50%), the mixture being then diluted to 300 c.c. with hot water and electrolysed at 80—90° by the method previously described for the determination of zinc (cf. B., 1925, 175). With a current of 4—5 amp., the deposition is complete in 10—20 min. and, after washing the electrode, the deposit is redissolved in 10 c.c. of nitric acid. This copper solution is made feebly alkaline with ammonia, acidified with sulphuric acid, and then re-electrolysed, using a freshly ignited, weighed, platinum electrode. The weight of copper deposited on the electrode yields results in good agreement with those obtained by ashing processes. H. J. DOWDEN.

Use of benzoic acid and sodium benzoate as ingredients of fresh sausages and fresh minced meat. H. EICHLER, G. ENDRES, G. GMINDER, O. MEZGER, and J. UMBRECHT (Z. Unters. Lebensm., 1929, 58, 553—584).—A collaborative study has been made of the effects of incorporating sodium benzoate and benzoic acid in the preserving salt used in the manufacture of sausages. The mixture, which consisted of benzoic acid 8.5 pts., sodium benzoate 8.5 pts., and common salt 83 pts., was added in the proportion of 4 g. of preservative per kg. of sausage meat to various sausages and to raw minced beef. Controls of identical composition were prepared without preservative and the samples were then stored at three different laboratories, in the dark, at room temperature, and in the ice chamber. Examinations of colour, taste, smell, bacterial content, and decomposition (Eber's test) were made at frequent intervals. A survey of the results showed that (i) a concentration of 0.06% of benzoic acid approximately doubles the keeping qualities of the sausages; (ii) the red colour of the meat is not enhanced, but is in some cases slightly diminished; (iii) the growth of all types of bacteria is retarded although none is entirely eliminated. H. J. DOWDEN.

Determination of sulphurous acid in minced meat. G. STEINHOFF (Z. Unters. Lebensm., 1929, 58, 649—650).—A suspension of benzidine sulphate undergoes quantitative hydrolysis and may be titrated with caustic soda (cf. Raschig, B., 1903, 1066). The sample of meat (25 g.) is digested on a water-bath with dilute alkali and filtered off, and to the filtrate, acidified with acetic acid, are added 15 c.c. of benzidine hydrochloride solution (20 g. of benzidine dissolved in 35 c.c. of hydrochloric acid [*d* 1.19] and diluted to 1 litre). Albumin is removed from the extract by Carrez's method (cf. A., 1909, ii, 625) and the resultant solution is diluted to 250 c.c. and filtered. To 100 c.c. of the filtrate are added 10 c.c. of 3% hydrogen peroxide, whereby the sulphurous acid is oxidised and precipitated as benzidine sulphate, which is collected, washed, suspended in water, and titrated with 0.1N-caustic soda against phenolphthalein (factor 32). The method is found to be unsuitable for the examination of gelatin or dried fruit. H. J. DOWDEN.

Detection of the commencement of putrefaction of flesh, and a method for the determination of ammonium salts of flesh. B. GLASSMANN and F. ROCHWARGER (Z. Unters. Lebensm., 1929, **58**, 585—592).—The ammonium salt content of meat and fish has been found to provide a useful indication of the onset of decomposition and a method is described for the determination of ammonium salts in the presence of protein degradation products. A representative sample (100 g.) is washed, dried, and minced finely, 5 g. of the minced meat being then ground with 50 c.c. of water and collected on a filter. 10 c.c. of the filtrate are shaken with sodium permutit previously purified by treatment with 2% acetic acid, to absorb the ammonium salts. The permutit is washed several times by decantation and then treated with 5 c.c. of 10% caustic soda solution followed by about 150 c.c. of water and 10 c.c. of Nessler's reagent, the whole being finally diluted to 200 c.c. The colour is compared in a Duboscq colorimeter with that of a solution of ammonium sulphate (1 c.c. containing 0.121 mg. NH_3) mixed with identical volumes of caustic soda and Nessler's reagent and diluted to 200 c.c. The critical value for meat was found to be 0.02% NH_3 , and for fish 0.02—0.025%, according to the conditions of storage. H. J. DOWDEN.

Carbon dioxide preservation of meat and fish. D. H. KILLEFFER (Ind. Eng. Chem., 1930, **22**, 140—143).—Meat of various kinds, sausages, butter, cheese, and eggs were in a good condition after storing at 4—7° for 2 weeks in an atmosphere of carbon dioxide and did not show spoilage until after 3 weeks, but in air many of the meat samples were spoiled after 2 weeks. Meat and fish stored in carbon dioxide keep 2—3 times as long as in air at the same temperature; at temperatures above the f.p. they showed a slight fall in p_{H} , which rose again on exposure to the air. Many bacteria (but not *B. typhosus*) show much slower growth on nutrient-agar plates in an atmosphere of carbon dioxide than in air; this is attributed to the lowering of the p_{H} of the medium. E. B. HUGHES.

Food preservatives. T. SABALITSCHKA (Pharm. Ztg., 1930, **75**, 454—456, 466—468).—Methods used for the determination of the activity and efficiency of preservatives and their bactericidal action under varying conditions are discussed, together with the influence of different physical factors and the character of the foodstuffs on the action of a substance as a preservative. The relative activity of a large number of substances including phenols and esters of *p*-hydroxybenzoic acid is described. Methyl *p*-hydroxybenzoate ("Nipagin") and particularly the higher esters of this acid have a much stronger action against a large number of organisms, especially moulds, than any of the usual preservatives. E. H. SHARPLES.

Digestibility trials on Indian feeding stuffs. VI. **Green fodders and silage.** P. E. LANDER and P. L. C. DHARMANI (Mem. Dept. Agric., India, 1930, 10, 193—208).

Milkweed. GERHARDT.—See V. **Effect of cooking**

salt on butter. BUDAGJAN and PAVLOV. **Butter fat in cooking fats.** PRITZKER.—See XII. **Saccharisation of rice malt.** MATSUMOTO and KISE. **Detection of vitamin-C.** REMY and RICHTER.—See XVIII.

PATENTS.

Whole-grain flour and the like. J. SCHNEIBLE, Assr. to C. B. SCHNEIBLE and K. F. SCHREIER (U.S.P. 1,744,581, 21.1.30. Appl., 7.12.23).—In the preparation of flour, the moistened whole grain is allowed to germinate and is then dried at 21—30° in such a manner that the enzymes are not injured, but insect eggs, larvæ, and moulds are destroyed. The product, containing less than 6% of moisture, is then ground. E. B. HUGHES.

Improvements of cereals, cereal flours, and food products therefrom. A. S. POLLAK (Aust.P. 109,163, 31.7.26).—The finely-divided material is placed in a sieve, and the sifted material, as it falls from the sieve, is treated with a vaporised solution of hydrogen peroxide. C. RANKEN.

Production of bread. A. M. J. SOREL (F.P. 569,990, 22.8.23).—Bread is manufactured without addition of leaven or yeast by introducing carbon dioxide under pressure into dough prepared with water saturated with carbon dioxide. C. RANKEN.

Preservation of eggs. T. M. RECTOR, Assr. to EMULSOL CORP. (U.S.P. 1,744,575, 21.1.30. Appl., 28.9.26).—Egg yolk is preserved in a suitable condition for use in mayonnaise manufacture by saturating it with salt (about 10%) and then storing it at a temperature slightly above the f.p. of the mixture. E. B. HUGHES.

Egg-preserving compositions. (Mrs.) C. C. ALEXANDER (B.P. 326,325, 25.2.29).—A mixture of lime, liquid petrolatum, paraffin wax, and formaldehyde is claimed. H. ROYAL-DAWSON.

Sausage products and their manufacture. MODERN FOOD PROCESS CO. (B.P. 326,847, 21.12.28. U.S., 10.11.28).—A protective membrane is produced on sausages by exposing the moulded sausage mixture to a damp atmosphere at about 35°, drying at 43° for a short time, and then smoking at 43—74°. The sausages may then be boiled or canned in the usual way. E. B. HUGHES.

Manufacture of food products. G. GRINDROD (B.P. 326,156, 29.8.28).—Liquids containing protein (pre-heated to about 43°) are placed in a closed container fitted with a hollow revolving arm carrying a large number of small nozzles. Steam at 25—50 lb. is passed through the nozzles into the liquid, which attains a temperature of about 110° by maintaining the container pressure at 6—10 lb./in.² It is claimed that in 3 min. the contents become sterile and their physical state is considerably changed. In the case of milk the globulin is destroyed, the albumin re-dispersed in some other colloidal form, and the casein becomes hydrated and resistant to coagulation. After this treatment milk may be concentrated to 50—65% of total solids, and when cooled it sets to a translucent yellowish gel which remains unchanged for months in an open container.

On dilution this gel reverts to ordinary milk having its appearance and flavour unchanged and complete solubility, but without the usual property of coagulation. By this process also (1) a concentrated ice-cream mix may be made as a gel; (2) gelatin may be sterilised without affecting its gelatinising power; (3) preparations of chocolate and candy products may be made as jellies; (4) preservation of ground meat, sausages, etc. may be effected by embedding in gelatin and subjecting the mixture to the treatment. E. B. HUGHES.

[Puffed] cereal foods. JERSEY CEREAL CO., ASSEES. OF C. E. LUKE (B.P. 299,803, 17.10.28. U.S., 1.11.27).

Margarine (B.P. 326,742).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Relationship between enzymes and alkaloids of *Atropa belladonna*, L. J. P. TODD (J. Roy. Tech. Coll. Glasgow, 1930, 2, 353—365).—Fresh belladonna leaves may be stabilised by heating in alcohol vapour at a pressure of 1.5—2.5 atm. with only slight loss of alkaloid, but this process has no advantage over careful drying, and the appearance is impaired. Losses of alkaloid of up to 20% occur when the leaves are allowed to ferment in presence of antiseptics at 32°. At 55° no loss occurs. The emulsification of the solvent in the process of determining the alkaloid content is most troublesome in carefully dried and enzyme-free leaves, and an improved method is described in which ether-soluble inert matter is first extracted from the powdered leaves moistened with acid. R. K. CALLOW.

Chemical and photochemical oxidation of commercial adrenaline solutions. T. VACEK (Compt. rend. Soc. Biol., 1929, 109, 583—584; Chem. Zentr., 1929, ii, 3026—3027).—The p_H values of commercial adrenaline solutions varied between 1.6 and 4.0. The rate of oxidation with hydrogen peroxide and in light is independent of the p_H , whilst an increase in the rate with increasing p_H was observed with pure solutions of adrenaline hydrochloride. The intensity of photo-oxidation depends on the intensity of the light and on the wave-length; irradiation with ultra-violet light alone causes less change than that with visible and invisible rays at the same time. A. A. ELDRIDGE.

Significance of the degree of dissociation of acids in the oxidation of adrenaline. T. VACEK (Compt. rend. Soc. Biol., 1929, 100, 585—586; Chem. Zentr., 1929, ii, 3027).—The speed of oxidation of adrenaline solutions is directly proportional to the hydrogen-ion concentration only when the same solvent is employed. A. A. ELDRIDGE.

Alkaloid determination in ergot. H. OETTEL (Arch. exp. Path. Pharm., 1930, 149, 218—239).—For testing ergot and its preparations the methods of D.A.B. VI (cornutin reaction and precipitation with Mayer's reagent) are inapplicable. A simple and reliable method using sodium carbonate is described for testing qualitatively the therapeutically active alkaloid content of *Secale cornutum* and preparations from it. For quantitative purposes the methods of Keller-Fromme

and of the German pharmacopœia are unreliable. Forst's method, slightly modified, gives unobjectionable results. The drug is first extracted by percolation with alcohol, the crude product purified by means of chloroform-ether, precipitated from a sulphanic acid solution with sodium carbonate, and weighed. The alkaloid contents of various commercial preparations varied greatly. T. R. SESHADRI.

Determination of morphine in opium. E. STUBER and B. KLJATSCHKINA (Arch. Pharm., 1930, 268, 209—223).—Morphine is most completely precipitated from its hydrochloride by ammonia at p_H 9.2, or in 2% ammonium chloride solution at p_H 8.9. For opium extracts the optimum p_H of precipitation varies with the sample. Precise directions are given for the determination of morphine in crude opium or opium powder by this method. The sample is extracted with water at 55° for 4 hrs. and kept overnight. After narcotine etc. have been removed from the extract by exact neutralisation with ammonia, three trials are sufficient to determine the maximum amount of morphine which can be precipitated by further addition of ammonia. The results, corrected for solubility, are higher than those given by Dieterich's method, and agree with those obtained when calcium hydroxide or borax is the precipitant. H. E. F. NOTTON.

Determination of morphine in tablets and pills. J. LAURENCE (J. Pharm. Chim., 1930, [viii], 11, 336—337).—About 10 g. of the powdered tablets are rubbed in the mortar with a mixture of 1 g. of calcium hydroxide and 15 c.c. of distilled water, the mixture is filtered, and the filtrate and washings are made up to 35 c.c. Hydrochloric acid is added until the liquid is just acid and then 1 g. of potassium iodide. When this is dissolved, exactly 15 c.c. of 0.1N-iodine are added and the mixture is agitated gently for 2 min. and set aside for 1 min. An aliquot of the supernatant liquid is filtered off and titrated with 0.1N-sodium thiosulphate. One mol. of morphine combines with 3 atoms of iodine.

E. H. SHARPLES.

Detection of nickel in drugs. L. ROSENTHALER (Pharm. Zentr., 1930, 71, 241—242).—The ash from 10 g. of the drug is warmed with aqua regia, the solution filtered, and the filtrate made alkaline with ammonia. The precipitate is filtered off and alcoholic dimethylglyoxime solution (Tschugaev) is added to the filtrate. The presence of nickel is indicated by a red colour. Results of the examination of a large number of drugs and herbs are given. E. H. SHARPLES.

Determination of the alcohol content of tincture of iodine. K. FEIST and F. KLATT (Pharm. Ztg., 1930, 75, 424).—The iodine is taken into combination by addition of water and iron powder; after reaction, the mixture is distilled, and the distillate analysed in the usual way. S. I. LEVY.

Detection of isopropyl alcohol in alcoholic pharmaceutical preparations. T. BOEHM and K. BODENDORF (Arch. Pharm., 1930, 268, 249—262).—Previously described methods are criticised (cf. Reif, B., 1928, 686).

When an aqueous solution of isopropyl alcohol is superposed on concentrated sulphuric acid containing *m*-nitrobenzaldehyde, a carmine-red ring appears at the point of contact. Methyl and ethyl alcohols and acetone neither give nor vitiate the reaction, but *n*-propyl, isobutyl, and amyl alcohols give a similar colour. The application of the test to tinctures and spirits is described. The detection of acetone in presence of acetaldehyde by means of sodium nitroprusside in presence of ammonia and ammonium chloride has been studied, and a method is given for the colorimetric determination of isopropyl alcohol, after oxidation, by this reaction.

H. E. F. NOTTON.

Chemical evaluation of Extractum Filicis maris. Z. CSIPKE (Magyar Gyó. Társ. Ert., 1929, 5, 425—438; Chem. Zentr., 1929, ii, 3045).—The pure filicic acid content is not an exact measure of the activity of the drug; the determination of the crude filicic acid content gives inaccurate values. The value of the extract is best expressed in terms of phloroglucin or phlorobutyrophenone. The extract is dissolved in ether, shaken twice with baryta water, the aqueous solution is filtered, and an aliquot portion treated with ammoniacal silver nitrate. After 6—10 hrs. in the absence of light the silver is collected, washed, dissolved in nitric acid, and titrated; 0.0108 g. corresponds with 0.00217 g. of phloroglucinol or 0.0196 g. of phlorobutyrophenone.

A. A. ELDRIDGE.

Adulteration of Flores Tilixæ, D.A.B. VI. H. KAISER and K. EGGENSBERGER (Süddeut. Apoth.-Ztg., 1929, 69, 450—451; Chem. Zentr., 1929, ii, 3045).—A taste test is needed; capillary and fluorescence analysis is insufficient.

A. A. ELDRIDGE.

[Biological] evaluation of digitalis. II. Comparison of Mansfeld's sinus method with the 6-hr. frog or cat method. A. STASIAK and B. ZBORAY (Magyar Gyó. Társ. Ert., 1929, 5, 400—406; Chem. Zentr., 1929, ii, 3045).

Detection of vitamin-C. REMY and RICHLER.—See XVIII. **Preservatives.** SABALITSCHKA.—See XIX.

PATENTS.

Extracting nicotine from tobacco in continuous operation. S. E. PAGE. From BIGOT SCHÄRFE & Co. CHEM. FABR. G.M.B.H. (B.P. 327,034, 22.4.29).—A mixture of tobacco with an excess of an alkali hydroxide is passed by means of a screw-conveyor through a horizontal, steam-jacketed cylinder in which it is treated with a countercurrent of superheated steam and/or hot, non-reacting gases, *e.g.*, furnace gases; nicotine is recovered from the gases and vapours leaving the cylinder.

L. A. COLES.

Treatment of tobacco. W. W. TRIGGS. From AMER. MACHINE & FOUNDRY Co. (B.P. 326,400, 6.5.29).—An apparatus is described whereby the volume of cured tobacco is increased (about 15%) by treatment (at 110°) under pressure (20 lb.) with air or steam, with subsequent sudden release of pressure.

E. B. HUGHES.

Preparation of thymol. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 298,600, 325,856, and 326,215,

30.8.28. Ger., 12.10.27).—*m*-Cresol, either (A) liquid, or (B) gaseous, is condensed with propylene, or substances which yield propylene by dehydration, in presence of dehydration catalysts such as alumina, zinc chloride, etc. Thymol is separated by distillation from two isomerides (m.p. 69°, b.p. 228.5°, and m.p. 112°, b.p. 245—246°, respectively). (C) The isomeric propyl- and isopropyl-*m*-cresols obtained as in (A) are converted largely into thymol by heating with dehydration catalysts (aluminium silicate) at 230°.

C. HOLLINS.

Separation of alkoxyisoeugenol from alkoxyisochavibetol and production of isoeugenol from the separated compounds. GRAESSER-MONSANTO CHEM. WORKS, LTD., Assees. of T. S. CARSWELL (B.P. 303,021, 24.12.28. U.S., 22.12.27).—The mixture of methoxymethyl ethers of 3:4-dihydroxyisopropenylbenzene, obtained, *e.g.*, from safrole or isosafrole by heating with methyl-alcoholic alkali at 135°, is converted into calcium salts by adding lime to the alcoholic solution. The insoluble portion is filtered off and acidified to yield the 4-ether ("methoxyisochavibetol"), and the 3-ether ("methoxyisoeugenol") is recovered from the filtrate. Each is then converted into isoeugenol, in one case by methylation and hydrolysis of the methoxymethyl group, and in the other by hydrolysis and methylation.

C. HOLLINS.

Manufacture of aqueous solutions of barbituric acids. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 325,847, 30.11.28).—Monoalkylamides of 5:5-disubstituted barbituric acids are sufficiently soluble in water to be useful for intravenous injection.

C. HOLLINS.

Manufacture of esters of [methyl-substituted] 2-phenylquinoline-4-carboxylic acids [atophans]. A. J. STEPHENS. From R. VON WÜLFING and E. MÜLLER (J. A. WÜLFING) (B.P. 325,985, 8.2.29).—Methyl-substituted atophans are heated at about 100° with 3—4 times the theoretical proportion of *n*-propyl, *n*-butyl, or isobutyl hydrogen sulphate. The three 2-phenyl-6-methylquinoline-4-carboxylates have m.p. 79—80°, 64—65°, and 74—75°, respectively; the 6:8-dimethyl compounds melt at 73—74°, 80°, and 83°, respectively.

C. HOLLINS.

Manufacture of alkylamine derivatives of organic compounds. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 326,553, 17.12.28).—The usual methods are applied to the production of β -diethylaminoethylamino-anthracenes or -phenanthrenes containing hydroxyl groups. *E.g.*, 1-bromoanthraquinone is condensed with β -diethylaminoethylamine and reduced to 1- β -diethylaminoethylamino-9:10-dihydroxyanthracene; 3-nitrophenanthraquinone is similarly converted into 3- β -diethylaminoethylamino-9:10-dihydroxyphenanthrene, b.p. above 250°/1 mm. The products have therapeutic value.

C. HOLLINS.

Manufacture of organic mercury compounds. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 325,846, 30.11.28).—Aromatic hydrocarbons (benzene) are refluxed with mercuric oxide or acetate in glacial acetic acid for 2—3 hrs. at 90—95°.

C. HOLLINS.

Manufacture of metal-organic complex salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,209, 4.12.28).—Carboxylic or sulphonic derivatives of resorcinol, particularly resorcinol-4:6-disulphonic acid, form therapeutically useful complex salts when treated with solutions of metal salts (silver nitrate, thallium chloride) and ethylenediamine. C. HOLLINS.

Production of a composition [which emits β -rays]. M. WRESCHNER and L. F. LOEB, ASSRS. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,752,826, 1.4.30. Appl., 4.3.26. Ger., 6.3.25).—See B.P. 248,765; B., 1926, 769.

Introduction of iodine into pyridine derivatives. G. KOCHENDOERFER, ASSR. to DEUTS. GOLD- U. SILBERSCHNEIDENSTALT VORM. ROESSLER (U.S.P. 1,753,170, 1.4.30. Appl., 1.2.26. Ger., 24.1.25).—See B.P. 246,501; B., 1927, 507.

Preparation of solutions of the active principle from animal organs. W. LUDWIG and O. SCHAUMANN, ASSRS. to WINTHROP CHEM. CO., INC. (U.S.P. 1,752,916, 1.4.30. Appl., 6.10.27. Ger., 15.10.26).—See B.P. 279,123; B., 1929, 226.

Distillation of glycerin (U.S.P. 1,743,289).—See I. N-Substituted arylaminesulphonyl chlorides (B.P. 326,226).—See III. Solidification of medicines etc. (B.P. 326, 447).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photosensitive preparations of selenium and tellurium. T. PAVOLINI (Giorn. Chim. Ind. Appl., 1930, 12, 72—74).—Mixtures of tartaric acid and ferric salts with selenious acid or with potassium tellurite are reduced in the presence of light with the formation of amorphous selenium or tellurium. The reaction proceeds in two stages, according to the equations $C_4H_6O_6 + 4FeCl_3 \rightleftharpoons 4FeCl_2 + 4HCl + C_4H_2O_6$, $H_2SeO_3 + 4FeCl_2 + 4HCl \rightleftharpoons 4FeCl_3 + 3H_2O + Se$, but in the case of tellurium the second reaction is easily reversible and the precipitated tellurium redissolves in the presence of excess ferric chloride solution. Details are given of the preparation of sensitive papers with these reactions as a basis. The selenium paper gives a bright red and the tellurium a black to brown tone. Development and fixation may be carried out with a dilute solution of hydrochloric acid, with the addition, in the case of the tellurium paper, of stannous chloride to prevent the destruction of the image by the unchanged ferric chloride. F. G. TRYHORN.

PATENTS.

Manufacture of light-sensitive surfaces for use in colour photography. K. BIALON (B.P. 326,287, 15.1.29).—Certain dyes form with silver bromide complex compounds which are sensitive to the light rays absorbed by the dye (acid-green, brilliant-blue, acid-violet, violamine, ponceau, azo-yellow), which, however, is not itself appreciably affected. Aqueous potassium bromide (1%) is added to a mixture of dye solution (0.5%) and silver nitrate (1%); the

precipitate, after washing with acetic acid, is mixed with gelatin solution (5%), a little ammonia is added, and the mixture, heated to 33°, is used for coating glass plates. C. HOLLINS.

Production of photographic images. H. D. MURRAY, ASSR. to NORTON & GREGORY, LTD. (U.S.P. 1,753,059, 1.4.30. Appl., 14.9.29. U.K., 14.7.28).—See B.P. 317,199; B., 1929, 872.

[Apparatus for] colour photography, in particular, cinematography. P. VEROLA (B.P. 308,973, 27.3.29. Fr., 2.4.28).

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose of low viscosity. M. G. MILLIKEN (Ind. Eng. Chem., 1930, 22, 326—328).—The reduction in viscosity of nitrocellulose from 500 to $\frac{1}{2}$ sec. by heating with water is carried out by a continuous process, the mixture passing through a tube 4 in. in diam. and 4000 ft. long, which is heated by a steam-jacket for a distance at the entrance, is heat-insulated along most of its length, and is cooled by a water-jacket near the exit end. Flashing of the water into steam is prevented by the back-pressure of a stand-pipe 200 ft. high connected to the exit, from which the treated mixture overflows into receiving tubs below. The process is safer and yields a more uniform product than that of batch-digestion in autoclaves. F. R. ENNOS.

PATENTS.

Blasting cartridge, percussion cap, detonator, detonating fuse, and the like. O. TUREK (U.S.P. 1,743,739, 14.1.30. Appl., 25.2.29. Czechoslov., 28.3.28).—A detonator contains as secondary charge compressed trinitrotoluene, tetryl, or 2:4:6-trinitro-1:3:5-triazidobenzene, and above this a priming charge of 0.05—0.2 g. of 2:4:6-trinitro-1:3:5-triazidobenzene pressed at a pressure not exceeding 300 kg./cm.² A perforated cap is finally inserted. W. J. WRIGHT.

Explosive powder. W. F. VAN VACTOR (U.S.P. 1,743,941, 14.1.30. Appl., 30.8.28).—The explosive is prepared by boiling sugar with water at 112.7—135° without stirring, and adding Irish moss jelly, potassium chlorate, and crude fuel oil. It contains potassium chlorate 40—60%, sufficient sugar to make the total with the chlorate 97%, Irish moss jelly 0.5%, fuel oil 0.5%, and water 2%. W. J. WRIGHT.

Low-velocity ammonia-dynamite. W. H. WARD, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,743,172, 14.1.30. Appl., 14.8.23).—For the manufacture of non-inflammable blasting explosives, containing nitroglycerin, ammonium nitrate, sodium nitrate, and a combustible, "relatively coarse" ammonium nitrate is used, its fineness being such that not more than 5% is retained by a 10-mesh, and not more than 20% passes a 60-mesh sieve. The strength of these dynamites lies between 10 and 60%, and their velocity of detonation does not exceed 2000 m./sec. A 20% dynamite contains nitroglycerin 4—7%, "relatively coarse" ammonium nitrate 23—27%, and sodium nitrate and combustible 68—74%. W. J. WRIGHT.

Percussion fuses for projectiles. SCHNEIDER & CIE. (B.P. 315,852, 23.5.29. Fr., 20.7.28).

Distillation of glycerin (U.S.P. 1,743,289).—See I.
Separation of mixed acids (B.P. 326,977).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Effect of certain acids on [sewage] sludge digestion. H. W. CLARK and G. O. ADAMS (Sewage Works J., 1929, 1, 393—397).—Lactic acid is less readily attacked by the bacteria of digesting sludge than is acetic, butyric, formic, or oxalic acid, but under certain conditions it is readily attacked. Unless present at the beginning of fermentation organic acids are not necessarily injurious to digesting sludge.

CHEMICAL ABSTRACTS.

Effect of iron compounds on [sewage] sedimentation, digestion, and ripe-sludge conditioning. W. RUDOLFS, L. R. SETTER, and W. BAUMGARTNER (Sewage Works J., 1929, 1, 398—410).—The addition of 5% of ferric chloride to raw, screened, domestic sewage increased the rate of settling of suspended solids from 24% to 53%. Small quantities of ferric chloride shortened the digestion time, but decreased the quantity of gas formed and the percentage reduction in volatile solids. Large quantities were beneficial to sludge digestion. The drying of digested sludge was accelerated by addition of ferric chloride. CHEMICAL ABSTRACTS.

Comparison of diffused-air and stream-flow aeration in purification of packing-house wastes. M. LEVINE, H. N. JENKS, and F. G. NELSON (Sewage Works J., 1929, 1, 425—430).—The latter gave a more rapid reduction of organic nitrogen, oxygen consumed, and five-day biological oxygen demand, whilst the former gave an effluent of better appearance.

CHEMICAL ABSTRACTS.

Engineering studies of municipal zeolite water-softening. H. N. JENKS (J. Amer. Water Works' Assoc., 1930, 22, 342—356).—The efficiency of a zeolite water-softening plant depends largely on the intimacy of the contact of the water and the medium. Experimental studies with a practical-scale plant indicate that at low rates of flow an upward-filtration plant loses efficiency owing to "channeling," whilst at high rates a downward-filtration plant suffers from an excessive loss of head. It is suggested that large plants should be designed so that they can be operated in either direction, according to the rate of flow, or if the latter is constant they should be either shallow beds employing downward filtration or deep beds with an upward flow. The zeolite may be regenerated more economically if the brine solution can be recirculated and the volume of wash-water used can be considerably reduced by replacing the usual 18—22-in. bed of gravel on which the sand rests by a bed of asphalt-bound gravel about 8 in. thick. For use in a large plant the asphalt-gravel bed may be made of slabs instead of being constructed *in situ*. In the discussion of the paper a simple form of apparatus is described in which the softening efficiency of the zeolite may be determined. C. JEPSON.

Scientific control [of water-purification plant]

from the operator's viewpoint. R. G. YAXLEY (J. Amer. Water Works' Assoc., 1930, 22, 387—395).—The plant at Waterford, N.Y., obtains a supply of soft water from the Hudson River which is badly polluted with paper-mill wastes and sewage. The effective coagulation range is limited, a divergence of 0.15 either way from the optimum p_H value of 5.95 being noticeable in the basin reactions. As these changes occur without any apparent alteration in flow, colour, turbidity, etc., p_H determinations are regularly made and sulphuric acid is used to reduce any excessive alkalinity. By this means it has proved possible to obtain excellent coagulation over a wide range of water conditions without a radical increase in the rate of alum feed. C. JEPSON.

Colour test of floc in treating coloured water. G. G. NASMITH (J. Amer. Water Works' Assoc., 1930, 22, 396—398).—The length of filter runs in an experimental plant at Ottawa was shown to be directly dependent on the quantity of floc leaving the settlement basins. As the colour removed by the floc constitutes part of the floc, the quantity present may be estimated by replacing the colour in solution by acidifying and comparing it with a sample similarly treated but containing the whole of the floc. By this test much time was saved in discontinuing unsuccessful experiments, and filter runs were increased finally from 5 to 65—70 hrs.

C. JEPSON.

Disposal of waste liquors from textile plants. COPSON and CURTIS.—See V.

PATENTS.

Removal of odours from gases and air. J. T. TRAVERS, ASSR. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,738,543, 10.12.29. Appl., 18.5.28).—Waste gases from packing houses, fertiliser or rendering plants, etc. may be deodorised by passing through an absorption tower in countercurrent to a creamy mixture containing calcium hydroxide, calcium sulphate, ferrous hydroxide, and marl, a suitable mixture containing 1 pt. of solids to 4 pts. of water. The proportions of the solid ingredients may be varied within wide limits, but the marl should be 50—80% of the whole, should contain 70—95% of calcium or magnesium carbonate and not less than 5% of matter insoluble in hydrochloric acid (1:1), and should have a "flotation value" (rate of settling in still water) of less than 4 in./min. The amount of lime used may be 15—35% provided there is always sufficient to react completely with ferrous sulphate (5—15%) to produce the remaining ingredients.

C. JEPSON.

Protection against the noxious effect of inhaling mercury vapour. GEBR. MERZ MERZ-WERKE (G.P. 455,974, 6.5.26).—Air to be breathed is passed through permeable material having a large superficial area, e.g., fibrous or powdered material, covered with a very thin layer of metallic gold, and contained in a gas-mask.

J. S. G. THOMAS.

Mixing apparatus (U.S.P. 1,745,291). Filtration medium (U.S.P. 1,743,524—5).—See I. Moth-proofing agents (B.P. 326,137). N-Substituted arylamine sulphonyl chlorides (B.P. 326,226).—See III. Water-softening material (U.S.P. 1,744,703).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 20 and 27, 1930.*

L—GENERAL; PLANT; MACHINERY.

Transfusion of matter from one solid to another under the influence of heat. New factor in the process of metamorphism. F. D. ADAMS (Canad. J. Res., 1930, 2, 153—161).—A brick (*A*), composed of synthetic clinker made from dolomitic magnesite and mill scale (and hence rich in lime and ferric oxide), which had been fired side by side with one (*B*) composed of dead-burned Austrian magnesite, was found to have bent over under the influence of heat and its own weight, so that one edge (which remained sharp and unflattened) touched the brick *B*, which remained unaltered in shape. By analysis of successive portions of brick *B* situated radially from the line of contact it was shown that a considerable alteration in composition had occurred; three well-defined zones (*a*, *b*, and *c*, the last being that immediately around the line of contact) of different composition being observed. The average percentages of SiO_2 , Al_2O_3 , Fe_2O_3 , and CaO in the original brick *A*, the zones *c*, *b*, and *a*, and in the original brick *B* were, respectively: 1.0, 1.0, 22.4, 8.4; 0.6, 0.5, 17.2, 4.0; 1.6, 1.3, 15.6, 5.4; 3.5, 1.7, 12.4, 6.3; and 2.3, 0.9, 8.3, 2.4%. Thus the silica, alumina, and lime in the altered portion of brick *B* increase progressively on receding from the line of contact from which the transfused elements came, whilst the amounts of ferric oxide (and magnesia) increase proportionately in the opposite direction. Furnace conditions suggest that the transfusion took place between two solid bodies, and that the period of contact was probably not greater than 20 hrs. The results are considered in the light of those of Hansen and Brownmiller (*A.*, 1928, 480) and Greig (*A.*, 1928, 132), and their bearing on metamorphosis of rocks is discussed.

J. W. BAKER.

Pulverised fuel for the small unit-shell-type boiler, metallurgical and chemical processes. H. W. HOLLANDS and E. C. LOWNDES (J. Inst. Fuel, 1930, 3, 225—230).—The use of powdered coal in various industrial processes such as cement and macadam manufacture, and in chemical works, is described.

C. B. MARSON.

Rapid bulk-sample dryer. T. H. HOPPER (Ind. Eng. Chem. [Anal.], 1930, 2, 198).—A motor-driven blower having a capacity of about 300 cub. ft. of air per min. drives air through a baffle box which has an opening in the top on which are placed a number of screen-wire trays containing the material to be dried. For drying wet and sticky substances a special box with glass plates is used. Diced, fresh potatoes, sugar beets, and mangel beets can be dried with little, if any, oxidase reaction, and the dried, ground sample is almost white. Lean meat can be dried to an extent which permits grinding in 20 hrs.

E. H. SHARPLES.

Graphical rectifying-column calculations. T. BAKER and J. S. STOCKHARDT (Ind. Eng. Chem., 1930, 22, 376—377).—A method is explained by which the number of plates required in a fractionating column may be determined. The method, which is a modification of that due to McCabe and Thiele (cf. *ibid.*, 1925, 17, 605), requires experiments to be made in order to determine the efficiency of the fractionation of the plates employed.

H. INGLESON.

Large glass distillation apparatus. D. F. OTHMER (Ind. Eng. Chem., 1930, 22, 322—325).—Distillation apparatus is described in which corks and rubber stoppers are eliminated by the use of flanged glass tubes with rubber-cushion backing-rings. The largest column mentioned measures 3 in. by 72 in., and is suitable for use with 22-litre flasks.

H. INGLESON.

Temperature changes in the formation of solutions. K. M. WATSON and O. L. KOWALKE (Ind. Eng. Chem., 1930, 22, 370—376).—A method is explained by which the quantities of heat associated with the adiabatic dissolution of unit mass of solute in an infinite mass of solutions at various concentrations and temperatures may be calculated from thermochemical data. Graphs are given from which the temperature attained in the adiabatic formation of solutions may be predicted. Experiments have been made in which crystals of sodium carbonate decahydrate having thermocouples in their centres were suspended in solutions of sodium carbonate of different concentrations and temperatures. Measurements of the temperature difference between the crystal and the solution were difficult to duplicate when the crystal was stationary or when it was moved through the solution at a velocity of 15 m./min.

H. INGLESON.

Determining air flow in agitation problems. H. L. KAUFFMAN (Chem. Met. Eng., 1930, 37, 178—180).—For a depth of liquid of 9 ft. moderate agitation requires 0.65 cub. ft. of air/min./sq. ft. of cross-sectional area, complete agitation 1.3 cub. ft., and violent agitation 3.1 cub. ft. Charts are given showing (1) the relation between linear velocity and volume in pipes of different diameters; (2) the pressure loss due to friction per 100 ft. of pipe length of various diameters relative to volume passing, and estimates are given of lengths equivalent to elbows in respect to friction; (3) the volume of flow through orifices of various diameters under pressures up to 10 lb./in.² These data require correction by a constant varying with the form of the orifice if this is not circular. The degree of agitation in any tank depends on both the quantity of air and the velocity with which it leaves the orifices. It increases more rapidly than the rate of air flow. The same

* The remainder of this set of Abstracts will appear in next week's issue.

quantity of air gives better agitation with deep tanks than with shallow tanks. C. IRWIN.

Is smoke responsible for property damage?

R. D. MACLAURIN (Chem. Met. Eng., 1930, 37, 157—159).—Although the filtration of smoke particles at the source is a more rational procedure than the filtration of air to protect merchandise and internal decorations, the latter procedure is necessary at present. Photomicrographs show the effect of smoke particles (diam. 0.1—50 μ) on silk etc., and the comparative air-cleaning efficiency of oil filters, water washers, and fabric filters. The first two are shown to remove dust, but to fail in the separation of the smaller particles from smoke.

C. IRWIN.

Tentative standards (American Society for Testing Materials, 1929, 901 pp.).

Heat-balance of a boiler plant. STEIGER.—See II.
Drying of pulp etc. SHERWOOD. **Heat economisers for drying pulp etc.** ADAMS and COOPER.—See V.

PATENTS.

Fuel-burning systems. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of J. V. BREISKY and T. DRAPER (B.P. 314,757, 1.7.29. U.S., 29.6.28).—A fuel-burning system, e.g., an oil-fired central-heating installation, is operated automatically by electrical controls. The fuel-ignition device consists of a transformer and spark gap, and the fuel-feeding means is controlled by an electrical discharge tube which is coupled to the spark-gap circuit in such a manner that the fuel and air are fed to the burner only when a spark capable of igniting the mixture is produced. The supply of energy to the ignition device is controlled by a thermostatic switch. Another thermostatic switch heated by the combustion gases controls a time-delay relay which, in turn, controls the fuel-feeding means, so that the supply of fuel is cut off a predetermined time after the thermostatic switch ceases to be heated. A. B. MANNING.

Heat-treating furnaces. H. M. ROBERTSON (B.P. 327,486, 23.1.29).—A tunnel kiln is constructed with a heating zone of the muffle type; the fuel is ignited at the front ends of two side chambers and the flame passes longitudinally through them then up to a common roof chamber where combustion is completed while the gases travel from back to front. The cooling zone is provided with a separate air current through a [metallic] casing forming the roof. A chain-conveyor is used to transport the goods, the return run being through a lower tunnel well insulated from the goods tunnel.

B. M. VENABLES.

Furnaces. J. A. HOPE (B.P. 327,532, 1.3.29).—A form of construction for a fire-grate and -bridge is described. Longitudinally the grate slopes upwards each end from a level centre part. Transversely it is divided into three sections each of which may be convex on the top.

B. M. VENABLES.

[Composite linings for] furnaces. CARBORUNDUM CO., LTD. From CARBORUNDUM CO. (B.P. 327,390, 1.1.29).—A furnace wall or roof is composed of an inner lining of veneer of silicon carbide or similar material supported on a backing of bricks which are comparatively poor heat conductors. The veneer bricks are held in place by

self extensions at the back, several methods of construction of which are given. Forms of suspended arches are also described.

B. M. VENABLES.

Kilns. J. W. and S. JONES (B.P. 327,396, 2.11.28).—In a continuous, multi-compartment kiln, each compartment has a furnace each side, one, with a solid bottom, being higher than the other and having the passage for transfer of air (or gases) underneath it; this passage emerges under the firebars of the lower furnace in the next compartment.

B. M. VENABLES.

Heat-exchange apparatus. SUPERHEATER CO., LTD., Assees. of COMP. DES SURCHAUFFEURS (B.P. 308,966, 26.2.29. Fr., 2.4.28).—Forms of fluid heaters, in which steam or other condensable fluid is blown through short inner tubes into larger blind tubes which are surrounded by the fluid to be heated, are described. The apparatus is wholly or partly inclined to promote discharge of condensate and to permit the outer fluid to make contact with the upper parts of the tubes first.

B. M. VENABLES.

Heat exchangers. MILLIKEN BROS. & BLAW-KNOX, LTD., Assees. of W. DYRSSEN (B.P. 304,755, 10.12.28. U.S., 26.1.28).—An exchanger of the reversing regenerative type is provided with at least three heat-absorbing masses in separate chambers which have metal plates in or surrounding the walls to prevent leakage. Each chamber is provided with two pairs of inlet and outlet valves, which are separate for the two fluids (e.g., gas and air), and these pairs of valves are operated intermittently, slowly, and out of phase (preferably by a common cam-shaft) so that the resistance to flow of either fluid is constant but not necessarily equal for the two fluids, i.e., two pairs of valves partly open must have the same resistance as one pair fully open. To get this result the valves are arranged to move slower when they are near their seats than when they are some distance open. Since in practice it is convenient to have a strong forced draught for the air and a weak induced draught for the gases, it may be arranged that there are more chambers open to the latter than to the former; this can be done with a minimum of four chambers, typical positions in the cycle being as follows: gas—two pairs partly and one fully open; air—one pair fully open. Later, when the cam-shaft has rotated a further 135°, the position would be: gas—two pairs fully open; air—two pairs partly open.

B. M. VENABLES.

Heat exchangers. ETABL. G. DESSON & CIE., and G. DESSON (B.P. 326,981, 5.3.29).—A form of construction of heat recuperator of the cellular type is described, in which fibro-cement sheets are used as the heat-transmitting medium.

B. M. VENABLES.

Heat exchanger. ANC. ETABL. MILLE POURCEL VELUT (B.P. 304,728, 23.1.29. Fr., 25.1.28).—In an exchanger of the concentric-tube type having small tubes in the annular space which by contact with the dividing wall improve the heat transmission, the small tubes are spaced apart so that the liquid can flow easily outside as well as within them, thus rendering both their surfaces effective for transmission of heat.

B. M. VENABLES.

Heat exchanger. C. WAINWRIGHT (U.S.P. 1,743,989, 14.1.30. Appl., 30.1.28).—A multistage exchanger com-

prising a number of bundles of tubes or other units is provided with mixing chambers between each unit.

B. M. VENABLES.

Ball mill. F. O. WILLIAMSON (U.S.P. 1,744,089, 21.1.30. Appl., 21.5.28).—Inclined faces are provided on the ends of the mill, which are stated to make the ball movement more efficient. An intermediate head or diaphragm with inclined faces on each side and apertures for passage of partly-ground material may also be provided.

B. M. VENABLES.

Attrition mills. (A) E. H. HUSSEY, (B) J. MARKLEY and E. M. BRENNAN, Assrs. to BAUER BROS. CO. (U.S.P. 1,744,226 and 1,744,235, 21.1.30. Appl., [A, B] 8.3.28).—Disc grinders suitable for pulping wood or other fibrous material are constructed so that the action is one of pressing and rubbing rather than of cutting, except in the earlier stage. In (A) the discs are divided into zones which are not quite concentric, an arcuate section of any zone is saw-toothed, in the innermost zone the steep edges of the teeth meet, in the subsequent zones the longer edges of the teeth meet, and the pitch of the teeth as also the gap between the discs decrease outwardly. In (B) the discs are formed with concentric wavy grooves and with radial ribs. The space for the material is decreased outwardly by increasing the height of the crests of the waves.

B. M. VENABLES.

Crushing machines. NORDBERG MANUF. CO., Asses. of E. B. SYMONS (B.P. 327,216, 16.1.29. U.S., 8.10.28).—In a gyratory cone crusher the renewable lining of the upper cone or bowl is fixed by means which are not subject to wear. The sections are formed with integral hook-like projections on the back, which extend through the permanent part of the bowl and are secured by U-bolts.

B. M. VENABLES.

Grinding mill. A. J. HAZLE, JUN., Assr. to BLATCHFORD CALF MEAL CO. (U.S.P. 1,745,330, 28.1.30. Appl., 19.3.26).—The screens of a disintegrator can be moved nearer or further from the beaters by placing their lugs in any one of several semi-circular slots, even when the mill is running.

B. M. VENABLES.

Grinding bodies for tube or drum mills. HELIPEBS, LTD., and R. McDUGALL (B.P. 326,656, 5.3.29).—The bodies comprise helices of spring steel or similar material with smaller helices loosely confined within them.

B. M. VENABLES.

Charging regulator for disintegrating and like mills or other apparatus. HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 303,032, 24.12.28. Ger., 24.12.27).—The rate of feed is regulated by the power taken by the driving motor, so that the latter is always fully loaded and the rate is never reduced to zero. A feed shaft is driven from a power shaft by two gear-trains: (a) at a low speed through a free-wheel device, this speed being sufficient to provide say 80% of the feed, and (b) at a high speed through a dog-clutch, and when this gear is in operation the free-wheel over-runs. A cam on the feed shaft moves the dog-clutch into engagement, but it will not stay in unless held by a detent. The detent is pushed out of engagement by another cam, but it is returned by a spring unless held out by an electromagnet, the strength of which is proportional

to the motor current. The result is that as long as the motor remains fully loaded the feeder operates at the lower speed.

B. M. VENABLES.

Utilising waste heat for refrigerating purposes. H. KEMMER (B.P. 301,837, 6.12.28. Ger., 6.12.27).—Waste heat in the form of hot gases at 350–600° from gas works or other industries is utilised in an absorption refrigerator charged with ammonia; the cold is utilised to make ice or for cooling and purifying the gas output.

B. M. VENABLES.

Bearings for the trunnions of drying cylinders. A. BUCHANAN (B.P. 327,424, 4.12.28).—The faced ends of the trunnions which form part of the steam-tight expansion joints are continuously lubricated by means of a chain encircling the trunnion and dipping into an oil-bath.

B. M. VENABLES.

Emulsifiers, mixers, and the like. H. P. JONES (B.P. 327,400, 31.12.28).—Within a container for the ingredients is a cylinder with a hand-operated, double-acting piston. The cylinder is provided with a number of holes either radially through the wall near the ends or axially through the ends themselves, which are 0.045–0.047 in. in diam.

B. M. VENABLES.

Mixing or stirring machines. E. CHRISTIANSEN and C. O. ERICSSON (B.P. 327,465, 10.1.29).—Two concentric shafts are arranged to be rotated in the same or opposite directions at will and to be fitted with a variety of interchangeable stirring tools.

B. M. VENABLES.

Mixing apparatus. C. J. BLEIL (U.S.P. 1,745,291, 28.1.30. Appl., 29.1.27).—An apparatus, suitable for extracting oils from seeds, sewage, etc. by means of a solvent, comprises an elongated chamber divided into sections by partitions extending alternately from the top and bottom nearly to the bottom and top, respectively. A longitudinal shaft drives propellers in each compartment and valved openings are provided in the lower baffles so that the charge may be forced either straight through or in zig-zag fashion.

B. M. VENABLES.

Emulsifiers, mixers, etc. A. C. EATON, and INTERNATIONAL KREEMAKA CO., LTD. (B.P. 326,854, 21.9.28).—Details are given of a hand-operated emulsifier in which the mixture is forced by a piston through a sinuous passage between concentrically grooved plates, and then through fine perforations.

B. M. VENABLES.

Electric mixer. B. GOULD, Assr. to GEM APPLIANCES, INC. (U.S.P. 1,743,271, 14.1.30. Appl., 20.9.28).—The bowl for the material and the unit comprising electric motor, gearing, and stirrer are arranged to be easily and separately detachable from the supporting stand.

B. M. VENABLES.

Presses for damp, or moist, materials. F. KRUPP GRUSONWERK A.-G. (B.P. [A] 306,931, 14.12.28. Ger., 29.2.28, and [B] 326,741, 23.7.29. Ger., 4.5.29).—In a worm-press which may be used for oil-containing fruits or seeds: (A) by the use of a sleeve shaft in one or more portions locked together by a solid shaft, the conveyor-worm is made readily detachable through the inlet end of the press; in (B) the outlet for solid material is throttled by an adjustable hollow cone.

B. M. VENABLES.

Separation process and apparatus. Filtering medium. C. T. CABRERA, Assr. to ELECTRO DIALYZER CORP. (U.S.P. 1,743,524—5, 14.1.30. Appl., [A] 15.11.27, [B] 1.6.28).—(A) Suspended solids are separated from a liquid by means of a number of "filtering" elements in series arranged in tanks of increasing size so that the rate of flow decreases progressively in the direction of flow. The filtering medium may be fragmented metal and the method of construction and operation is similar to the zinc boxes of the cyanide process. (B) Suitable filter media are more fully described, e.g., intimately mixed strands of two metals having different electric potentials and degrees of resiliency, such as zinc and lead. Application to sewage and various factory effluents is indicated. B. M. VENABLES.

Pressure regulator for mechanically pumped liquids. I. G. FARBERIND. A.-G. (B.P. 309,086, 4.4.29. Ger., 4.4.28).—In a process such as the spinning of artificial silk, the pressure may be maintained constant without the use of compressed air by means of a by-pass valve across the pump which comprises a number of small relief valves so adjusted that they lift in a close succession. To prevent ageing because of trapped liquid the valves are arranged in line in one casing, and a small permanent passage or lightly loaded valve is left at the end remote from the inlet and outlet. B. M. VENABLES.

Mixing of liquids. E. W. BUTLER and J. C. MANN (B.P. 326,924, 19.1.29).—Two liquids, such as molten pitch and oil, which may differ largely in temperature but of which the mixture must be maintained between close temperature limits to avoid frothing on the one hand and imperfect mixing on the other, are mixed in stages in a trough-like apparatus divided into compartments, the flow being from the top of one compartment to the bottom of the next or *vice versa*; additions of raw material may be made to successive compartments in proportions suitable to maintain the correct temperature, aided, if necessary, by application of heat to the transfer passages. A longitudinal shaft extends through all compartments and carries stirring arms which extend through the depth of the liquid and well above it so as to beat down any froth produced. B. M. VENABLES.

Apparatus for mixing liquid and gas. K. CHOGO, Assr. to S. KUBOTA (U.S.P. 1,740,441, 24.12.29. Appl., 7.1.28. Jap., 28.1.27).—Liquid is drawn from the upper part of a cylindrical tank by a pump, and returned through the bottom past a baffle. The gas is admitted to the pipe between the outlet of the pump and the tank, and the original liquid may be admitted rather nearer the pump. When it is desired to use the gas again, as in the case of hydrogenation of fats, the circulation is sufficiently rapid to draw the gas back through the pump. B. M. VENABLES.

Foam-producing apparatus. W. FRIEDRICH (B.P. 314,438, 26.6.29. Austr., 27.6.28).—A foam-producing liquid is caused to pass across a finely-perforated metal plate to the back of which gas is supplied under high pressure. Suitable perforated plates may be made from nickel by electrolysis. B. M. VENABLES.

Device for concentration of liquids, especially whey and skim milk. LANDWIRTSCHAFTLICHE ZENTRAL-

GENOSSENSCHAFT R. GES.M.B.H. (Austr.P. 107,317, 16.6.26).—In the upper part of an open vessel which can be heated is placed a bucket-wheel consisting of a number of shovel-like ladles set on a boss at equal angular intervals. The sides of these are closed by discs of greater diameter than that of the boss. The liquid raised in the ladles flows into the vessels formed by adjacent ladles and the lateral discs, and, on further rotation of the wheel, flows back into the container over the edge of the ladle in front. W. J. BOYD.

Dividing substances in the liquid state into drops. STOCKHOLMS BENMJÖLSFABRIKS AKTIEB. (B.P. 313,566, 12.6.29. Swed., 14.6.28).—A number of plungers are reciprocated inside a vessel containing glue or other liquid in such a way that they pass, at the lower end of their stroke, right through a number of discharge nozzles in the bottom of the vessel and are lifted clear of the apertures when at the top of their stroke. B. M. VENABLES.

Centrifugal dust arrestor. F. H. WAGNER, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,743,171, 14.1.30. Appl., 25.4.29).—The external shape of the apparatus is similar to that of a cyclone separator; internally the gas is passed through a spiral conduit, which is considerably smaller in diameter than the outer casing. The outer wall of the spiral is formed with louvre-like outlets, and beyond them is a spiral curtain wall having internal blades which form approximately a continuation of the louvres; consequently the dust cannot whirl directly from the spiral conduit to the outer wall of the apparatus, but is directed downwardly. B. M. VENABLES.

Dust separator. R. RUEMELIN (U.S.P. 1,743,934, 14.1.30. Appl., 26.11.24).—After separation of the coarse dust by inertia the gas passes into filter bags, which are shaken by horizontally moving bars at about the middle of their length. B. M. VENABLES.

Air filter. A. JORDAHL (U.S.P. 1,743,675, 14.1.30. Appl., 10.3.23).—The filter is constructed of corrugated sheets of expanded metal arranged firstly ridge to ridge forming large pockets, then with the sheets parallel at gradually decreasing distances apart to obtain smaller pockets for the complete entrainment of the finest dust. An alternative to the latter method of assembly is to fill the pockets with metal wool, kieselguhr, or the like. B. M. VENABLES.

Gas cleaner. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,743,344, 14.1.30. Appl., 10.3.26).—The casing of the apparatus is cooled externally by water so that its temperature is maintained below the dew point of the gas, as received or after adding moisture from internal water-sprays, though the bulk of the gas may at all times be at a high temperature. The gas enters axially to the casing, and is whirled by fixed or rotating tuyères; the dust, however fine, that reaches the wall of the casing is retained there by the dew and gradually creeps down to the collecting hopper at the bottom. B. M. VENABLES.

Disintegrator for cleaning gases and the like. H. F. J. NOLZE (U.S.P. 1,743,380, 14.1.30. Appl., 19.4.27. Ger., 23.4.26).—The apparatus comprises a disintegrator with radial blades that do not extend

inwards as far as the shaft, the gas (with water also, if desired) being supplied to this central dead space. To regulate the flow, the blades are notched as deeply as possible in a direction parallel to the axis, and in these notches are inserted baffles comprising two perforated, cylindrical plates slidable on each other, so that the effective apertures of the perforations may be varied.
B. M. VENABLES.

Gas-washing apparatus. A. J. BOYNTON, Assr. to H. A. BRASSERT & Co. (U.S.P. 1,744,863, 28.1.30. Appl., 4.6.27).—In a disintegrator type of washer a uniform screen of spray is produced by a device comprising a trough for the liquid, attached to the hub of the beaters, and a number of radial outlet pipes extending to the first ring of beaters. The inlets for gas are sideways among the radial pipes, thus ensuring that the streams are well distributed before coming into contact with the liquid.
B. M. VENABLES.

Treatment of gases with solid substances. METALLGES. A.-G. (B.P. 304,239, 15.1.29. Ger., 17.1.28).—A solid substance which is alternately used to react with a gas and is then reactivated, *e.g.*, in the process of removing hydrogen sulphide from coal-gas, or in many catalytic reactions, is subjected to reactivation before it has lost much activity. Both operations are preferably performed with small quantities moving at considerable speed relative to the reacting and reactivating gas, respectively, and in stages, the general motion of the solid substance being countercurrent to the reacting gas.
B. M. VENABLES.

Forming gases into solid blocks. F. B. DEHN. From SOLID CARBONIC CO., LTD. (B.P. 327,414, 31.12.28).—The snow (or hail) emerging from a spraying chamber where liquefied gas is expanded is forced downwards by a screw-conveyor into a compressing cylinder. The collecting chamber is provided with a jacket through which the vaporised part of the gas passes on its way to waste or re-liquefaction and in which is coiled the pipe supplying the sprays. When the piston of the snow compressor is operated, it, being long, first closes the inlet aperture and then compresses the snow and ejects a solid block of ice through the end of the cylinder, which is opened for the purpose; the feed screw is automatically stopped while the cylinder end is open.
B. M. VENABLES.

Freezing mixtures. L. A. and (MME.) B. GARCHÉY (B.P. 327,038, 30.4.29. Fr., 24.10.28).—The tendency of freezing mixtures composed of powdered ammonium nitrate and sodium carbonate to cake on storage is inhibited by adding talc powder to each of the constituents before they are packed separately. [Stat. ref.]
H. ROYAL-DAWSON.

[Detection of leaks during] manufacture of high-vacuum vessels and apparatus. W. DÄLLENBACH (B.P. 303,512, 1.1.29. Ger., 5.1.28).—The vessel is filled with a chemically reactive gas under pressure and the outside is covered with a suitable solution, conveniently applied by soaked paper or cloth. The recommended reagents are gaseous ammonia and Nessler's solution; globules of mercury, indicating leaks, will appear at much lower pressure than is necessary with air and soapy water.
B. M. VENABLES.

Lubricants. R. S. and C. S. PRENDERGAST, A. SONST-HAGEN, and F. PEARSON (B.P. 327,097, 22.9.28).—For the working parts of machinery subject to friction under rapidly varying local pressure a lubricant is used containing a metal or compound, *e.g.*, powdered zinc or zinc oxide, which is anodic to the metal of the machinery, and which therefore, under the influence of any currents of electricity generated by the friction, would cause a coating of the metal to be deposited on the parts to be protected.
A. B. MANNING.

[Visual] detection of suspended matter in fluids. W. KIDDE & Co., Inc., Assees. of H. C. GRANT, JUN. (B.P. 306,825, 22.2.29. U.S., 25.2.28).—Smoke particles or other matter suspended in air or other fluid render a beam of light visible; in this invention both a direct and a reflected image of the luminous smoke are caused to be seen simultaneously by the observer, thus obtaining nearly double the sensitivity.
B. M. VENABLES.

Industrial furnace. R. WARSITZ (U.S.P. 1,757,492, 6.5.30. Appl., 3.7.28. Ger., 15.6.27).—See B.P. 292,146; B., 1929, 701.

Method and apparatus for burning solid fuel. F. NOVELLI (U.S.P. 1,756,096, 29.4.30. Appl., 21.5.27. It., 27.5.26).—See B.P. 271,889; B., 1928, 506.

Drying plants. N. PANZIREV (B.P. 327,432, 3.10.28).—See U.S.P. 1,736,980; B., 1930, 223.

Purification system for boilers. J. OSTERTAG (Re-issue 17,658, 6.5.30, of U.S.P. 1,700,715, 29.1.29).—See B., 1929, 190.

Gyratory crusher. J. E. KENNEDY (U.S.P. 1,754,895, 15.4.30. Appl., 21.1.28).—See B.P. 322,690; B., 1930, 124.

Apparatus for separation of finely-divided solids from liquids. J. W. WICKES (U.S.P. 1,754,870, 15.4.30. Appl., 4.2.26. U.K., 19.2.25).—See B.P. 256,995; B., 1926, 857.

Separator for treatment of mixtures of mutually insoluble liquids. H. J. HOLFORD, Assr. to HARVEY HOLFORD SEPARATORS, LTD. (U.S.P. 1,756,862, 29.4.30. Appl., 30.1.29. U.K., 4.2.28).—See B.P. 310,996; B., 1929, 543.

Homogenising machine. G. C. HURRELL (U.S.P. 1,756,198, 29.4.30. Appl., 9.10.25).—See B.P. 245,929; B., 1926, 223.

Filtering or like devices. H. A. THOMPSON, Assr. to SWINNEY BROS., LTD. (U.S.P. 1,754,728 and 1,757,153, [A] 15.4.30, [B] 6.5.30. Appl., [A] 14.2.29, [B] 12.1.29. U.K., [A] 17.1.29, [B] 31.7.28).—See B.P. 318,821 and 307,267; B., 1929, 1036, 499.

Furnace walls. AMER. ENG. CO. (B.P. 327,862, 15.4.29. U.S., 22.10.28).

Construction of wagons and rails, applied in ovens for dry distillation, dryers, kilns, and similar ovens working by means of gas and vapour injections or circulations. EESTI PATENDI AKTSIASELTS, and M. KULZINSKI (B.P. 327,717, 7.11.28).

Heat-non-conducting coverings [for domestic cisterns etc.]. W. B. McCULLOCH (B.P. 328,472, 17.5.29).

Absorption refrigerating apparatus. (SIR) C. MARKHAM (B.P. 328,234, 22.1.29).

[Adsorption] refrigerating apparatus. G. H. HASLAM (B.P. 328,301, 25.1.29).

Refrigeration plant of the intermittent absorption or adsorption type. UNIVERSAL REFRIGERATORS, LTD. (B.P. 311,324, 26.4.29. U.S., 9.5.28).

Atomisers [for perfumes etc.]. E. GAGNAN (B.P. 313,532, 13.6.29. Fr., 13.6.28).

II.—FUEL; GAS; TAR; MINERAL OILS.

Origin of coal according to the present position of biological investigation. R. LIESKE (Brennstoff-Chem., 1930, 11, 101—105).—The formation of peat from the original plant materials is essentially a biological process, the earlier stages of which are brought about by aerobic, and the later stages by anaerobic, organisms. In this process the hydrolysable constituents are more easily and rapidly destroyed than the non-hydrolysable; the latter, of which the lignin is the most important, form the principal constituents of the original plant material from which coal has been formed. Taylor's theory (B., 1928, 509), which has received additional support from observations made in the Ruhr coalfield, has thrown light on the biological processes involved in the further transformation of peat into brown or bituminous coal. Purely physical and chemical processes, however, must also have played a part in determining the character of the final product.

A. B. MANNING.

Origin of coal according to the present position of chemical investigation. W. FUCHS (Brennstoff-Chem., 1930, 11, 106—112).—The literature dealing with the constitution of plant materials similar to those from which coal must have been derived, and with the chemical changes associated with the transformation of such material into coal, is critically discussed. It is concluded that the initial changes involve the disappearance of the cellulose with a corresponding increase in the lignin content of the material; the lignin-rich material is then slowly transformed into coal. The character of the final product, i.e., the type of coal produced, will depend to some extent on the chemical constitution of the original lignin, and this may vary considerably with the character of the plant material from which it has been derived.

A. B. MANNING.

Coals and their impurities. J. W. WHITAKER (J. Inst. Fuel, 1930, 3, 175—178).—A lecture. The classification of coals, the effect of moisture, the composition of the ash, distribution of sulphur, occurrence of chlorine, and methods of coal cleaning are discussed.

C. B. MARSON.

The X-ray stereoscopic examination of coal. I. A. N. WILSON (J. Inst. Fuel, 1930, 3, 218—224).—X-Ray apparatus and the application of stereoscopic radiography to the examination of coal are described in detail.

C. B. MARSON.

Composition of coal bitumen and its influence on the coking of coal. H. NOVÁK and J. HUBÁČEK (Paliva a Topeni, 1927, 9, 165—170, 187—196; 1928, 10, 3—9, 22—28; Chem. Zentr., 1929, ii, 3085—3086).

—Bitumen was extracted from a brown coal with tetralin and from a bituminous coal with pyridine; the compositions were compared, the former product being the lighter, and the principal constituents of the latter being asphalts. Brown-coal bitumens in contact with infusible materials give on coking a solid coke. In coke from bituminous coal the "cementing residue" forms one third of the mass.

A. A. ELDRIDGE.

Phosphorus content of coal. FAERBER (Kohle u. Erz, 1929, 26, 755—756; Chem. Zentr., 1929, ii, 3263).—This varies considerably according to the origin of the coal; washing the coal does not remove the phosphorus.

A. A. ELDRIDGE.

Calculation of the net calorific value of strongly bituminous brown coals. W. EISENSCHMIDT and H. KOOP (Chem.-Ztg., 1930, 54, 213—214).—The authors consider that with these coals (lignites) the net calorific value should be calculated only from the value for the hydrogen content as determined by actual analysis, since the Langbein approximation (5.9%) leads to considerable error (of the order of 100 heat units).

E. LEWKOWITSCH.

Future position of coal and carbon [in other forms] as raw materials of the chemical industry. C. MATIGNON (Chim. et Ind., 1930, 23, 543—555).—Coal and petroleum have hitherto been handled by the mechanical engineer rather than the chemist, but this position is changing. The primary by-products of coal carbonisation and petroleum cracking (hydrogen, carbon monoxide, methane, ethylene, etc.) are surveyed and their applications in ammonia synthesis, hydrogenation, and the syntheses of methyl alcohol and formaldehyde are indicated. Methane, in turn, may become a starting point for acetylene, hydrogen cyanide, and methyl chloride, etc., ethylene for ethyl alcohol, and ethylene glycol and butadiene for synthetic rubber.

C. IRWIN.

Comparative costs of fuels for domestic purposes.

A. H. BARKER (Gas J., 1930, 189, 572—573, 643—646, 702—705).—A detached house having entrance hall, 4 living rooms, 7 bedrooms, etc. was equipped with the necessary appliances for heating and cooking by different methods, and tests as to cost were carried out over a period of months. The advantages and disadvantages also of the various methods of heating and cooking are discussed, and it is concluded that a suitable combination of methods is the most efficient; rationalisation of the coke, gas, and electricity undertakings is therefore advocated.

C. B. MARSON.

Agglutination of coals and its relationship to the action of solvents. M. L. CRUSSARD (Rev. de l'Ind. Min., 1929, 551; Fuel, 1930, 9, 177—191).—The work of Lierg (B., 1922, 532 A), Fischer (B., 1925, 233), and Bone (B., 1924, 584) is co-ordinated and discussed in relation to the mechanism of the agglutination of coal. Attention is directed to the part played in this process by the moderately or difficultly fusible extracts which are insoluble in light petroleum, viz., the "asphaltenes" or "solid bitumen."

A. B. MANNING.

Asturian coal. I. Low-temperature distillation of cannel coal from Mieres. B. A. BUYLLA (Anal. Fis. Quím., 1929, 27, [tecn.], 195—218).—Asturian cannel

coal yields concordant results when subjected to low-temperature distillation by the Gray-King and the Fischer-Schrader methods. The rate of heating does not influence the relative yields of gas, tar, and semi-coke, but the last-named becomes more porous and more friable as the rate is increased. At 600°, as compared with 520°, the primary gas becomes richer in hydrogen and carbon monoxide at the expense of the methane and other saturated aliphatic hydrocarbons.

H. F. GILLBE.

Low-temperature distillation of Asturian coal. L. R. PIRE (Anal. Fís. Quím., 1929, 27, [tecn.], 377—382).—The economic aspect of the process as carried out in Spain is discussed.

H. F. GILLBE.

Phenolic solubility of humic acids in coals. F. PODBREZNIK and P. M. SOUM (Proc. II. Int. Conf. Bit. Coal, 1928, 1, 667—669).—Artificial and natural humic acids are soluble in hot phenol. Lignins are more soluble than humic acids in alcohol and in acetone. Phenol depolymerises certain humic substances which are probably derived from lignin.

CHEMICAL ABSTRACTS.

Rôle of humic acids in the transformation through heating of coal and in the production of coke. F. PODBREZNIK (Proc. II. Int. Conf. Bit. Coal, 1928, 1, 662—669).—A discussion. Young coals contain up to 15% of free humic acids, and condensed humic acids probably existing as dilactones which may undergo irreversible change into humic compounds at higher temperatures. Lignites consist of free lactonated humic acids, and disintegrate on carbonisation on account of the rapidity with which steam and carbon dioxide are evolved; humic substances afford gases which dissociate with deposition of carbon in the cavities of the coal.

CHEMICAL ABSTRACTS.

Changes in methoxyl content in the carbonisation of lignite. K. HRDLÍČKA (Paliva a Topeni, 1927, 9, 185—187; Chem. Zentr., 1929, ii, 3200).—Lignite containing 6·12% OMe, when carefully distilled at 400—500°, gave only 0·25% of methyl alcohol and 0·32% of acetic acid. The coke contained no methoxyl groups; the tar contained 0·53%, calculated on the pure organic matter of the lignite. Hence in the carbonisation of lignite greater decomposition of methoxyl groups takes place than in the carbonisation of wood.

A. A. ELDRIDGE.

Hydrogenation of South African coal. P. N. LATEGAN (Third Empire Min. Met. Congr., Apr., 1930, 21 pp.).—Experiments are described on the hydrogenation of coal from the Witbank Central Area, Transvaal, with reference to the use of a vehicle containing a small quantity of ferric oxide, the final temperature, and the rate of heating. The ease of hydrogenation of a coal is found to depend on the readiness with which it dissociates into its molecular complexes, and the composition and stability of these. The mechanism of the process is discussed.

C. W. GIBBY.

Plasticity of coal after treatment by the Bergius process. J. M. PERTIERRA (Anal. Fís. Quím., 1929, 27, [tecn.], 191—194).—Hydrogenation by the Bergius method lowers the temperature at which the material

becomes plastic and raises the temperature of solidification.

H. F. GILLBE.

Combustion of solid fuels and heat-balance of a boiler plant. J. U. STEIGER (Proc. Tech. Sect. Paper-makers' Assoc., 1929, 10, 165—184).—The subject is dealt with mathematically under the following headings: heating power of solid fuels, chemical reactions in combustion, heat in flue gases, and combustion formulæ. Rules for the calculation of efficiency and heat losses are given.

T. T. POTTS.

Disturbing effects in the determination of the decolorising power of active carbons. V. EDELSTEIN (Z. Zuckerind. Czechoslov., 1930, 54, 257—265).—Points made are that the coloured solution (e.g., a 10% solution of beet molasses) should be prefiltered, kieselguhr being used to eliminate suspended matter, and freshly prepared; that the kind of filter-paper used should be specified; and that time and temperature of heating should be constant in all determinations.

J. P. OGILVIE.

Heat economy of coke ovens. (A) O. FEISCHER. (B) K. BAUM (Brennstoff-Chem., 1930, 11, 112—113, 113—114; cf. Baum, B., 1930, 355).—(A) The heat efficiency of an oven will vary with the size, moisture content, and caking properties of the coal carbonised. Although the heat efficiency is a maximum with uniform heating, a vertical temperature gradient is necessary to prevent undue cracking of the tar and gas in the upper part of the oven. (B) Results are quoted by Baum to show that with as uniform a heating of the oven as practicable the maximum efficiency is attained, not only from the point of view of heat economy, but also in respect of the quality of the coke produced.

A. B. MANNING.

Stamping effect in [Japanese] coke manufacture. S. ARAKAWA (J. Fuel Soc. Japan, 1930, 9, 23—27).—Comparison of cokes made from the same coal in compressed and uncompressed charges showed that compressed coke was less spongy and had a higher crushing strength than uncompressed coke, but their shatter indices were the same. The compressed coke produced less breeze, and it is concluded that the quality of certain Japanese foundry cokes can be improved by "stamping."

C. B. MARSON.

Management of gas producers. L. M. WILSON (Fuel, 1930, 9, 152—164).—The choice of coal, the method of starting and maintaining the fire, the regulation of the air: steam ratio, the control of gas output, and other practical considerations in the management of modern producers gasifying raw coal are discussed. The ash in the coal may be any amount up to 20% if it is infusible; the moisture content should not exceed 3%; the volatile matter content, calculated on the ash-free basis, should be high, preferably above 35%; a small-sized non-caking coal is preferable, and the fines should be removed. The depth of ash in the producer above the apex of the blast hood should be maintained at 6—12 in., and the depth of the fuel bed at about 30 in. The gas velocity and temperature in the mains connecting the producer to the furnace, and the length of the mains, are preferably adjusted so that the tar and soot remain in suspension until the gas reaches the

furnace regenerator, wherein the tar is cracked and the soot tends to interact with the carbon dioxide in the gas according to the equation $C + CO_2 = 2CO$. It is desirable to instal a gas governor for controlling gas output.

A. B. MANNING.

[Apparatus for the] determination of the gas yield from coal. H. KOELSCH (Z. angew. Chem., 1930, 43, 310—313).—A laboratory apparatus for testing gas coal is described.

S. K. TWEEDY.

Asphalt emulsions : their nature, manufacture, and practical application. GRÄFE and FLECK (Petroleum, 1930, 26, 357—361).—A general survey of modern practice. It is pointed out that whilst a certain degree of stability is essential, an extremely fine dispersion is not altogether desirable, since the total amount of emulsifier adsorbed by the particles is in this way increased, and the greater the resulting contamination of the separated asphalt surface the more is the tendency to re-emulsification by the combined action of moisture and traffic. The use of asphalt emulsions for road-surface spraying and patching, grouting, and the preparation of mixtures with suitable aggregates are briefly described and discussed. The necessity of very thorough cleansing of the road surface before treatment with emulsion is emphasised.

D. G. MURDOCH.

Removal of phenols from effluents. L. M. HOROVITZ-VLASOVA (Gas- u. Wasserfach, 1930, 73, 275—278).—Intensive aeration of dilute phenol solutions (0.1%) containing soil or charcoal, or passage of such a solution through well aerated filters of the same media, fairly rapidly produces oxidation compounds which give no colour reaction with Millon's reagent and which absorb less chlorine than the original phenol, giving rise to compounds which do not possess an objectionable odour. The oxidation is shown to be independent of bacterial action, no decrease in the amount of phenol being effected by the addition of pure cultures of *B. coli*, *B. implexus*, and *B. megaterium* to a broth containing 0.1% of phenol.

D. G. MURDOCH.

Determination of total cyanogen in ammoniacal liquor. A. TRAVERS and AVENET (Compt. rend., 1930, 190, 1015—1016).—Cyanogen is present both as cyanide and thiocyanate in such liquors. The thiocyanate is oxidised without loss of cyanogen to cyanide and sulphate by nascent hydrogen peroxide in an alkaline medium. This is effected by the addition in small portions at a time of sodium peroxide to a sample cooled below 0°. Oxidation is complete in about 10 min. The solution is then distilled with dilute sulphuric acid and the hydrocyanic acid determined as usual. The phenols present in such liquors have no effect. Results are accurate to 1%.

C. A. SILBERRAD.

Gaseous products of shale retorting : their composition and possible utilisation. A. W. NASH (Third Empire Min. Met. Congr., Apr., 1930, 12 pp.).—The uses of the gaseous products of shale distillation, other than as fuel for the generation of heat, are discussed. The olefine content may be as high as 20%, and these gases could be advantageously used for conversion into higher hydrocarbons, alcohols, and glycols. Brief reference is made to various existing processes.

C. W. GIBBY.

High-pressure hydrogenation of shale oil. I. Y. TANAKA and K. FUJISAWA (J. Soc. Chem. Ind., Japan, 1930, 33, 42—43 B).—Crude shale oil produced in a retort of Japanese design was heated at 280—290° in presence of about 1% of reduced nickel and hydrogen at 140—150 atm. The content of solid paraffins was raised 8% or more, the loss on refining by sulphuric acid and the pitch and distillation loss being at the same time considerably decreased. Apparently the more highly unsaturated hydrocarbons of high b.p. are catalytically hydrogenated to less unsaturated or saturated hydrocarbons without the occurrence of any appreciable cracking.

S. K. TWEEDY.

Lubricating oils : carbon residue estimation. The Ramsbottom method. C. I. KELLY (J. Inst. Petroleum Tech., 1929, 15, 495—515).—The development of the Conradson carbon residue test is reviewed, and two types of apparatus suitable for the Ramsbottom test are described, only one of which, however, is satisfactory in actual use. The satisfactory apparatus consists of (a) a gas ring burner with manometer and needle-valve control; (b) a sheet-metal container for molten-lead bath; (c) a circular lid, to the underside of which are welded the metal sheaths, holes being drilled in the lid to permit the entry of the coking bulbs into the sheaths. An additional hole is made for accommodating the pyrometer and from the underside of the lid is hung a cylindrical skirt of sheet metal lined with asbestos; (d) a circular base-plate with a circular hole and fitted with a cylinder forming an easy sliding fit into the cylindrical skirt; and (e) a vertical rod passing through the upper cylinder and resting in a ferrule. The rod is fitted with a circular eye on which the pyrometer can be rested in an inclined position. Radiation is reduced to a minimum and temperature control is fairly simple, whilst convection currents in the lead are nearly eliminated. Results of several trials with the apparatus show that the highest carbon residue is obtained when the final heating period is least, but the percentage difference is greatest with oils of low carbon residue. The carbon residue was the higher the smaller the weight of oil taken, but variations in size and thickness of bulbs had no effect. The lower the temperature the higher was the result, due probably to the varying time factors.

W. S. E. CLARKE.

Determination of the solidification point of petroleum lubricating oil. M. MIZUTA (J. Soc. Chem. Ind., Japan, 1930, 33, 44—45 B).—The oil is heated (e.g., to 35°) in a test-tube provided with an outer insulating tube, and is then cooled; when the oil has almost set the tube is held in a horizontal position, and that temperature observed at which the oil flows for 5—15 sec. is noted. This gives the solidification point correct to 1°, and agrees with the results obtained by the usual method. Variations in the atmospheric temperature have no effect. The variations in the flow times corresponding to successive 1° lowering of the temperature depend on the nature of the oil.

S. K. TWEEDY.

Sludge of transformer oils. II. Effect of heat on transformer oils in vacuum and in atmospheres of nitrogen, carbon dioxide, and hydrogen. T.

YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 64 B; cf. B., 1930, 272).—Oils which readily deposit sludge on heating in air showed no deposit nor colour change when heated in a vacuum, or in a confined atmosphere of nitrogen, carbon dioxide, or hydrogen, for 100 hrs. at 140°, but they darkened when these gases were bubbled through the heated oils. The darkening under these circumstances is due to traces of oxygen in the gases.

S. K. TWEEDY.

Technical xylene from distillation residues of light oils. I. E. BESPOLOV (Nef. Choz., 1929, 16, 385—386).—The separation of technical xylene in 13% yield is recorded; pure xylene could not be obtained. The separation of higher homologues is as yet impossible commercially.

CHEMICAL ABSTRACTS.

American [mineral oil] refinery technology. B. T. BROOKS (J. Inst. Petroleum Tech., 1930, 16, 125—132).—Important developments during the past 1½ years are reviewed. These include the De Florez cracking still (cf. B., 1929, 668), which produces a motor fuel of high anti-knock rating without high unsaturation and excessive gum formation, and the Gyro high-temperature low-pressure cracking process. Attention is being paid to the production of lubricating oils of very low pour-point for aviation purposes. Aluminium is finding extended use in refineries in order to overcome corrosion trouble, especially in those treating high-sulphur crudes.

H. S. GARLICK.

The Edeleanu process for refining petroleum. R. L. BRANDT (Ind. Eng. Chem., 1930, 22, 218—223).—Flow sheets and a description of a commercial plant for operating this process, using liquid sulphur dioxide as the treating reagent, are given. The process is shown to give excellent results, especially in the removal of sulphur and nitrogenous compounds, and to have overcome many of the disadvantages inherent in the sulphuric acid method of refining. Analyses of the products show them to be superior to those obtained by the usual methods. Pressure distillate can be treated by this process, so that the anti-knock properties of the finished gasoline are greatly improved. Cost data are given for a plant treating kerosene and light oils and also for a plant treating lubricating oil.

H. S. GARLICK.

Cracking. G. EGLOFF, C. D. LOWRY, JUN., and R. E. SCHAAD (J. Inst. Petroleum Tech., 1930, 16, 133—246).—A comprehensive review of papers and patents published during 1928 and 1929 dealing with the application and technology of cracking.

H. S. GARLICK.

Catalytic oxidation of paraffin and mineral oil. E. ZERNER (Chem.-Ztg., 1930, 54, 257—259, 279—281).—Treatment of paraffin by air or oxygen at 110—150° in the presence of catalysts, such as manganese or zinc stearate, results in considerable absorption of oxidation and the formation of a white pasty product with a cocoa-like odour, a small quantity of an oily distillate having a strongly acid odour, and a strongly acid aqueous distillate. The pasty product contains a large proportion of fatty acids, chiefly hydroxy-acids, as well as much unsaponifiable matter the nature of which has not yet been elucidated. The fatty acids yield hard

soaps which give a good lather, but have a most unpleasant odour and cannot be salted out from solution. The oily distillate contains about 30% of oily fatty acids which also yield unpleasant soaps, and 60—70% of hydrocarbons, alcohols, and carbonyl compounds; those of the last-named compounds which have a low b.p. have a fruity odour, and those of high m.p. crystallise well. The aqueous distillate contains formic, acetic, and other volatile fatty acids. Similar results are obtained by the catalytic oxidation of mineral oil; this oil after purification with liquid sulphur dioxide can also be catalytically oxidised with air in the presence of 1% of slaked lime and a manganese or zinc catalyst, but the products obtained all have an unpleasant odour, and so far it has not been found possible to remove the cause of this odour, consequently the oxidation products of these oils are unsuitable for the manufacture of soap.

A. R. POWELL.

Distillation of naphthas containing mercaptans by steam, with and without reagents. P. BORGSTROM, R. ROSEMAN, and E. E. REID (Ind. Eng. Chem., 1930, 22, 248—249).—The mercaptans studied were all volatile in steam, and the mercaptan content of all naphthas but one remained constant after distillation from water or from alkali. On steam-distillation from copper sulphate or copper acetate solutions the mercaptan content in the distillate decreased with increasing amounts of copper salts. Caustic soda did not aid in removing the mercaptan in these solutions.

H. S. GARLICK.

Loss of mercaptan-sulphur in naphtha by use of inorganic salts and caustic soda. P. BORGSTROM, V. DIETZ, and E. E. REID (Ind. Eng. Chem., 1930, 22, 245—247).—The efficiency of removal of low-boiling mercaptans from various naphthas by a number of inorganic salts and sodium hydroxide has been studied. Copper acetate, mercuric acetate, and ammoniacal copper sulphate gave a sweet naphtha, although with the last-named there was oxidation to disulphide instead of removal as with the other two salts. With reagents such as mercuric oxide, copper oxide, and copper sulphate, where a precipitate could be formed, variable results were obtained. Acetates of sodium, calcium, barium, cobalt, zinc, and iron show only slight removal of the lower mercaptans and practically none of the *sec*-amyl compound. Bismuth nitrate has a greater effect, but ferrous and ferric salts are not so efficient. Cadmium chloride, neutral and acidified, give a slight decrease which appears dependent on the solvent for the mercaptans. A naphtha solution treated with a solution of sodium zincate made up similarly to that of sodium plumbite shows that more of the higher mercaptans remained than of the lower. *N*-Caustic soda removes more of the lower mercaptans than of the higher. With variations in the naphtha used, mercaptan content, and concentration of alkali it was found that solutions of ethyl mercaptan are "doctor-sweet" after treatment with all concentrations of alkali used. *N*-Propyl and *isopropyl* mercaptans show only a trace of mercaptan remaining after treatment with all except the lowest concentrations of alkali. With increasing mol. wt. of the same type of mercaptan there is increase in the percentage remaining in the naphtha, the secondary

straight-chain mercaptan being less removed than the corresponding primary compound, independent of the naphtha used. Increase in concentration of alkali shows no corresponding decrease in mercaptan remaining in the naphtha, whilst there appears to be a minimum with 2·18*N*-sodium hydroxide.

H. S. GARLICK.

Petrol-water emulsions. C. VARADHAN and H. E. WATSON (*J. Indian Inst. Sci.*, 1930, 13A, 21—30).—The emulsions were prepared by dissolving or suspending a stabiliser in a definite volume of water and mixing this with a known volume of petrol, then passing the mixture many times through a Premier colloid mill. Consistent emulsions were difficult to obtain, and varied from a thin fluid, which separated into its constituents in a few seconds, to a butter-like mass apparently indefinitely stable. Emulsions of two types were obtained with every stabiliser used, depending on the relative quantities of the two constituents. Each stabiliser gave a fairly definite inversion point, in the neighbourhood of which emulsions of both types having the same concentration could be obtained. Ammonium soaps gave an inversion point around 30% of petrol, sodium and potassium soaps of unsaturated acids about 65%, potassium stearate 50%, and potassium palmitate 30%, although with the last-named petrol-in-water emulsions were obtained with 52% of petrol, and one emulsion prepared with 0·5% of potassium stearate was of the water-in-petrol type, although containing only 33% of petrol. The inversion point did not appear to depend on the concentration of the stabiliser. Ammonium soaps generally gave the more stable emulsions, particularly as regards the water-in-petrol type. Emulsions using potassium palmitate or stearate tended to set to a jelly and were more stable than those containing salts of the unsaturated acids, although except for these two cases the degree of unsaturation of the acid appeared to have little effect. Comparison of the stability of emulsions with the drop numbers for the different stabilisers shows that the most stable emulsions are obtained with stabilisers having the lowest drop numbers, *i.e.*, ammonium oleate and ammonium soaps from olive oil fatty acids. Measurements were made of the viscosity of the emulsions after varying periods of time.

H. S. GARLICK.

Benzol. W. H. COLEMAN (*J. Inst. Petroleum Tech.*, 1930, 16, 252—254).—Work published during 1928 and 1929 chiefly concerns methods of treating benzol so that easily polymerisable substances may be removed without destroying the valuable unsaturated hydrocarbons which will not polymerise, thus avoiding the loss of a large proportion of aromatic hydrocarbons following treatment with drastic refining agents. Methods of determining the content of aromatic compounds in mixtures have received attention, but up to the present these still fall short of what might be desired.

H. S. GARLICK.

Production of benzine by cracking. F. KOUDÉLÁK (*Paliva a Topeni*, 1928, 10, 36—37, 51—56, 67—75, 89—95, 109—113, 119—126; *Chem. Zentr.*, 1929, ii, 3265).—Descriptive.

A. A. ELDRIDGE.

Raoult's law and the equilibrium vaporisation of hydrocarbon mixtures. M. C. ROGERS and G. G.

BROWN (*Ind. Eng. Chem.*, 1930, 22, 258—264).—Mixtures of three natural gasolines, a synthetic mixture, three mixtures of natural gasoline with a mid-Continent absorption oil, a mixture of a naphtha with a natural gasoline, and a mixture of tetralin with natural gasoline were studied in order to determine the extent to which Raoult's law holds in calculations involving the vaporisation of hydrocarbon mixtures. After analysis of the mixtures by a special fractional-distillation procedure and mol. wt. determinations on the various residues, vaporisations were made on these mixtures and the results compared with the curves calculated by Raoult's law. The results show that deviations from Raoult's law when applied to complex mixtures are dependent on the relative composition of the mixtures, and that the vapour pressure of natural gasolines and similar mixtures cannot be calculated by this law when a precision greater than 5—15% is desired.

H. S. GARLICK.

Motor-fuel volatility. I. Equilibrium volatility.

G. G. BROWN and E. M. SKINNER (*Ind. Eng. Chem.*, 1930, 22, 278—288).—Methods previously proposed for estimating the equilibrium volatility, which is defined as the percentage by wt. vaporised under equilibrium conditions at specified temperature, pressure, and air-fuel ratio, from the A.S.T.M. distillation are shown to be inaccurate, although convenient, and it has been found impossible to determine accurately the partial equilibrium volatility of motor fuels in the presence of air in any form of apparatus that has been described. Equilibrium volatility can be computed accurately from the continuous equilibrium vaporisation or "flash distillation" data by making proper allowance for the mol. wt. of the vaporised part of the fuel. This relationship is incorporated in a chart which is applicable to all types and sources of motor fuel.

H. S. GARLICK.

Natural gas. S. C. CARNEY (*J. Inst. Petroleum Tech.*, 1930, 16, 118—124).—Progress in the utilisation and distribution of natural gas in the U.S.A. is reviewed. Natural-gas gasoline is the chief by-product, with carbon black second in importance, most of the production of this latter product being still by the old method of burning gas jets against slowly moving steel surfaces. Various chemical products derived from natural gas are rapidly increasing in importance, but they use relatively small amounts of natural gas, gas from cracking installations being preferred. Of liquefied gas fuels, propane is sold chiefly for domestic use and butane for industrial purposes, *e.g.*, gas enrichment and as a high-grade fuel for ceramic and metallurgical operations. Helium is being separated in increasing quantities in certain localities.

H. S. GARLICK.

Solubility of gas benzines and gases in paraffin oil. F. FISCHER and P. DILTHEY (*Ges. Abh. Kennt. Kohle*, 1928, 8, 305—306; *Chem. Zentr.*, 1929, ii, 3265).—Methane, pentane, and carbon dioxide, but not hydrogen, carbon monoxide, air, or water-gas, are distinctly soluble in paraffin oil. Similar experiments with gas produced with the aid of a benzine catalyst indicated no correlation with the contraction of the catalyst or the composition of the gas.

A. A. ELDRIDGE.

Anti-detonators. W. H. COLEMAN (J. Inst. Petroleum Tech., 1930, 16, 247—251).—A summary of papers and patents published during 1928 and 1929 dealing with the action of anti-detonators and causes of detonation.

H. S. GARLICK.

Interpretation of results of chemical analyses for gas works. W. SCOTT (Gas J., 1930, 190, 198).

Pulverised-fuel-fired furnaces. HOLLANDS and LOWNDES.—See I. Separation of phenols from tar. SCHULZ and MENGELE.—See III. Road tar. NICHOLSON. Tar roads. MÜLLER.—See IX. Alcohol for motor fuel. GUINOT.—See XVIII.

See also A., May, 546, **Inflammability of hydrogen** (TANAKA and NAGAI). 551, **Catalytic reduction of carbon monoxide at ordinary pressure** (KODAMA). 561, **Determination of water [in tars and oils]** (NOTEVARP). 570 and 571, **Lignin theory of origin of coal** (BODE ; LIESKE). 571, **Origin of bituminous coal** (STADNIKOV). 589, **Separation of xylenes** (NAKATSUCHI).

PATENTS.

Separation of coal from dirt and like foreign substances. W. H. BERRISFORD (B.P. 326,961, 19.2.29).—The material is passed through several stages each comprising a declining plate and a gap, the apparatus being so adjusted that in at least one stage substantially clean coal is separated, and in at least one other stage coal-free dirt is separated; the middlings from the last stage are returned to the first for re-treatment.

A. B. MANNING.

Coal-washing apparatus. F. H. BLATCH, H. L. McLEAN, and W. C. MENZIES (B.P. 327,042, 3.5.29).—The coal is separated from its impurities by means of a current of water which passes up through a perforated plate forming the bottom of the compartment wherein the washing is effected; the coal is carried over the upper edge of a breast-plate into a chute and is thence discharged from the apparatus. The impure coal is delivered into the compartment through a pivoted control gate, which is connected to the gate controlling the opening through which the impurities are discharged, the two gates co-operating to maintain the required relation between the rate of delivery of impure coal and the rate of discharge of the impurities. Means are provided for agitating the material in the washing compartment and for preventing clogging of the passage through which the impurities are discharged.

A. B. MANNING.

Heat-treatment of carbonaceous material. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 307,918, 26.2.29. U.S., 17.3.28).—The finely-divided material is fed continuously into a vertical retort, through which it passes by gravity, and wherein it is subjected to progressively higher temperatures in such a manner that the volatile products are first removed, and the residue is then carbonised, without substantial agglomeration of the material. Heating is effected by the circulation of the hot gases from suitably placed combustion chambers through tubes extending across the retort. The hot residue is withdrawn through a water-seal at the bottom of the retort. The steam thereby

evolved reacts with the hot carbonised particles to produce water-gas. The latter may be withdrawn from the retort mixed with the distillation gases, or, by the provision of separate sets of eduction tubes, the two gases may be withdrawn separately. Provision is made for the complete gasification of the material in the lower part of the retort, if desired, by the introduction of a controlled amount of air.

A. B. MANNING.

Low-temperature carbonisation of coal and other suitable fuels. J. E. HACKFORD (B.P. 326,813, 19.9. and 10.11.28).—Adhesion of the fuel to the container, conveyor, or other surfaces with which it is brought into contact during the heat treatment, is prevented by interposing a thin layer of paper, pulped paper, sawdust pulp, etc. between the fuel and the surfaces in question. The interposed material must be one which does not pass through an intermediate plastic or liquid phase during carbonisation. The coal may be carbonised in cylindrical metal containers, the interior surfaces of which have been coated with a thin layer of the material, in a liquid or semi-liquid form, by a spraying operation.

A. B. MANNING.

Production of carbon black. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 326,913, 9.1.29).—A particularly finely-divided carbon black is obtained by subjecting suitable carbon compounds, *e.g.*, olefines, diolefines, acetylenes, carbon monoxide, to heat treatment in the presence of catalysts, the treatment being carried out in a non-sparking electric field in which no substantial discharge takes place.

A. B. MANNING.

Manufacture of carbon black. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 327,374, 19.12.28. Cf. B.P. 324,959; B., 1930, 405).—Unsaturated hydrocarbons are treated in the gaseous phase with a dehydrogenating catalyst, at temperatures between 100° and 600°, either under reduced pressure in the absence or presence of other gases, or under atmospheric pressure in the presence of other gases. The catalyst comprises a metal or compound of a metal of the iron group.

A. B. MANNING.

Operation of retort [coal]-gas plants, and particularly the stand-pipes therefor. BARRETT Co., Assees. of S. P. MILLER (B.P. 303,166, 27.12.28. U.S., 29.12.27).—In a horizontal retort setting the vertical stand-pipes leading from the retorts to the main are insulated so that the temperature of the gases therein is maintained sufficiently high to prevent any condensation of tar or pitch in the pipes. The sensible heat of the gases is utilised in the distillation of tar which is supplied to a section of the main or to a special distillation box. The tar is brought into intimate contact with the gas by spraying or other means. The pitch residue is withdrawn and the enriched gases are passed to a condensing system.

A. B. MANNING.

Water-gas plants. T. SCHLATTER (B.P. 326,998, 16.3.29).—A water-gas plant is provided with a steam superheater, the tubes of which are built into the outer walls of the igniting chamber, which is interposed between the producer and the waste-heat boiler.

A. B. MANNING.

Removal of ammonia and hydrogen sulphide from coal-distillation and like gases. C. J. HANSEN, Assee. of H. KOPPERS A.-G. (B.P. 307,903 and 309,116, [A] 15.11.28, [B] 24.9.28. Ger., [A] 17.3.28, [B] 5.4.28. Cf. B.P. 299,765 and 301,061; B., 1930, 449).—(A) The gases are scrubbed with solutions containing thiosulphates, polythionates, or other suitable oxy-sulphur compounds, and the spent liquors are regenerated with sulphurous acid obtained by the oxidation of some or all of the hydrogen sulphide in the gas. The oxidation is carried out by passing a part or the whole of the gas, together with air or oxygen, over a catalyst, *e.g.*, finely-divided iron oxide, the treated gases being subsequently worked up by a wet process. (B) The gases are scrubbed with a single liquor containing iron or manganese thiosulphates or polythionates in solution, and the corresponding hydroxide in suspension, the proportion of hydroxide being sufficient to eliminate all hydrogen sulphide in excess of the ratio $2\text{NH}_3 : 1\text{H}_2\text{S}$. The spent liquor is divided into two parts, one of which is regenerated by oxidation with air and the other by treatment with sulphurous acid, in such a manner as to produce hydroxide and thiosulphate, respectively, in the required ratio. A. B. MANNING.

Destructive hydrogenation. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,896, 31.12.28).—Tars, oils, suspensions of coal in oil, etc. are passed together with hydrogen through an externally heated spiral coil arranged within a high-pressure vessel. The coil may be heated by means of a gas under pressure, *e.g.*, hydrogen, which is itself used subsequently in the reaction, or nitrogen, which may also flow in a spiral coil enveloping the first coil. The coils are preferably arranged vertically and the space within them is occupied by a vessel wherein the liquid and gaseous products are separated.

A. B. MANNING.

Carrying out catalytic hydrogenation [of hydrocarbons]. GES. F. TEERVERWERTUNG M.B.H., and H. KAFFER (B.P. 302,912, 18.12.28. Ger., 23.12.27).—Naphthalene, low-temperature tar benzines, etc. are hydrogenated, without cracking, by the action of hydrogen under pressure and at elevated temperatures, using as catalyst the residual coal which remains as the insoluble and undistillable portion of the original coal after hydrogenation according to the Bergius process.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials, especially for the production of motor fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,194, 3.12.28).—The desulphurisation of crude benzol by treatment with hydrogen under pressure and at elevated temperatures is combined in one operation with the destructive hydrogenation of other carbonaceous materials, *e.g.*, coal, oil, etc.

A. B. MANNING.

Distillation of tar. BARRETT Co., Assees. of S. P. MILLER (B.P. 306,093, 14.2.29. U.S., 15.2.28).—The hot gases from a number of coking chambers or retorts are passed through a common collecting main through which also a stream of thinly fluid tar is circulated and is thereby distilled by surface contact with the hot gases. The gases which leave the main are scrubbed

while hot to remove suspended particles of pitch, and are then passed to a condensing system wherein relatively clean oils are separated. A. B. MANNING.

Production of (A) high-, (B) low-carbon pitches and pitch compositions. (c) **Production of high- and low-carbon pitches.** BARRETT Co., Assees. of S. P. MILLER (B.P. 303,163—5, 27.12.28. U.S., 29.12.27).—(A) The gases and vapours evolved during the later stages of coal distillation in coke ovens, horizontal retorts, etc. are withdrawn separately and cooled, in stages if desired, and the tars obtained are subsequently distilled to give pitches of high free-carbon content. The distillation may be effected by bringing the tar into intimate contact with hot coal-distillation gases (cf. B.P. 282,826; B., 1929, 633). By suitably blending the pitches and tars produced, pitches of any desired m.p. and of high free-carbon content are obtained. (B) By separately withdrawing and cooling the gases evolved during the earlier stages of coal distillation, and subsequently distilling the tars thus recovered, pitches containing a relatively low proportion of free carbon are obtained. The distillation may be carried sufficiently far to give pitches of high m.p., which may then be blended with tars or softer pitches to form products of any desired characteristics. (c) The coke ovens or retorts are provided with three collector mains in which the tars evolved during the initial, intermediate, and final stages of carbonisation, respectively, are separately collected. The duration of the initial and final stages, during which low-carbon and high-carbon tars, respectively, are collected, can thus be independently regulated. The tars are subsequently distilled to produce pitches of different free-carbon content. A. B. MANNING.

Fractional distillation [of lubricating oil distillate]. M. B. COOKE, Assr. to ATLANTIC REFINING Co. (U.S.P. 1,744,261, 21.1.30. Appl., 27.8.26).—A lubricating oil distillate having a narrow b.p. range and from which wax is readily removable is produced by passing the appropriate petroleum oil vapours through a series of fractionating zones one above another in countercurrent to and in actual contact with a continuous stream of reflux liquid, while withdrawing and cooling liquid from a zone intermediate the uppermost and lowermost zones and returning the cooled liquid to the zone from which it was withdrawn. H. S. GARLICK.

Recovery of gasoline. H. B. BERNARD, Assr. to SINCLAIR OIL & GAS Co. (U.S.P. 1,743,185, 14.1.30. Appl., 17.4.25).—A liquid absorbing medium used for extracting condensable hydrocarbons from gas mixtures is heated by heat exchange with an external heating medium and the volatile products are dephlegmated by heat exchange with the separated absorbing medium after cooling. The absorbing medium from the dephlegmator is cooled and part returned for treating fresh gas mixtures, the remainder being re-cycled and mixed with the absorbing medium circulating to the dephlegmator. H. S. GARLICK.

Production of stabilised dispersions. N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 301,805, 3.12.28. Holl., 5.12.27).—Aqueous dispersions of asphalt, pitch, tar, mineral oils, rubber, etc. are stabilised by reversing the charge of the particles from

negative to positive by the addition of suitable agents, *e.g.*, aluminium chloride, the reversal being effected in the presence of added protective substances (*e.g.*, casein, gelatin), and of finely-divided solid substances preferably colloidal in character, *e.g.*, iron or aluminium hydroxide, clay, barium sulphate. A. B. MANNING.

Bone-black of great decolorising power. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,755,156, 15.4.30. Appl., 3.7.26. Fr., 22.7.25).—See B.P. 255,816; B., 1926, 1003.

Products [acetylene] from coal, tars, mineral oils, and the like. O. EISENHUT, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,757,454, 6.5.30. Appl., 18.1.27. Ger., 19.1.26).—See B.P. 286,825; B., 1928, 356.

Recovery of gas tars from their emulsions with water. R. R. RIPLEY and S. C. SCHWARZ (B.P. 328,516, 29.7.29).—See U.S.P. 1,724,222; B., 1929, 842.

Manufacture of oil-gas mixtures. C. CHILOWSKY (U.S.P. 1,757,855, 6.5.30. Appl., 18.7.22. Fr., 27.7.21).—See B.P. 183,828; B., 1924, 46.

Production of products from montan wax. W. FUNGS and M. JAHRSTORFER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,755,189, 22.4.30. Appl., 10.9.27. Ger., 13.9.26).—See B.P. 292,298; B., 1928, 633.

Automatic means for regulating the calorific value of coal gas. C. M. CROFT, R. H. BUXTON, and WANDSWORTH, WIMBLEDON, AND EPSOM DISTRICT GAS CO. (B.P. 328,007, 15.1.29).

Oil-cleaning apparatus [for waste lubricating oil etc.]. A. R. BELLAMY, and RUSTON & HORNSBY, LTD. (B.P. 327,386, 1.1.29).

Fuel-burning system (B.P. 314,757). Utilising waste heat (B.P. 301,837). Treatment of gases with solids (B.P. 304,239).—See I. **Fibres from peat (B.P. 325,904).**—See V. **Asphalt- and bituminous-masses (B.P. 307,465).**—See IX.

III.—ORGANIC INTERMEDIATES.

Formation of butadiene by cracking of hydrocarbons. P. K. FROLICH, R. SIMARD, and A. WHITE (Ind. Eng. Chem., 1930, 22, 240—241).—Experiments were made on the production of butadiene by simultaneous cracking and polymerisation of propane, butane, ethylene, propylene, and cyclohexane by passing the gases or vapours through a cracking chamber consisting of a 2-ft. length of 1-in. quartz tubing wound with resistance wire, and leading the exit gases first through an ice-cooled and then through a carbon dioxide-cooled condenser. The former condensed most of the high-boiling products, *e.g.*, tar, benzene, and naphthalene, whilst the butadiene condensed along with the lower-boiling materials in the carbon dioxide snow cooler. This product was collected and distilled, the fraction boiling above -30° being passed through a train of absorbers containing a concentrated solution of bromine in carbon tetrachloride. Brominated olefines and diolefines remained in solution, which on evaporation to dryness left a solid which was almost pure butadiene tetrabromide. In the case of propane the highest yield obtained corresponds to about 1 mol. of butadiene per

100 mols. of propane, and it appears that this is close to the maximum conversion obtainable by simple cracking. With propylene and butane the highest yields were observed when slightly more than 3 mols. of the diolefine per 100 mols. of hydrocarbon were employed, but in view of the incomplete survey of the experimental conditions a possibility exists of increasing these conversion figures. A single experiment on the cracking of cyclohexane gave 20.6 mols. of butadiene per 100 mols. of cyclohexane reacting. In all experiments with propane, butane, ethylene, and propylene some high-boiling material was formed consisting largely of benzene with smaller amounts of naphthalene and unidentified tarry compounds. The results throw some light on the mechanism of production of aromatic compounds from paraffins, in that they point to the formation of diolefines as intermediates in the process. II. S. GARLICK.

Triethanolamine [tri- β -hydroxyethylamine] emulsions. A. L. WILSON (Ind. Eng. Chem., 1930, 22, 143—146).—The properties and many uses of tri- β -hydroxyethylamine and its fatty acid soaps for emulsification are discussed. The oleate is completely miscible with practically all organic liquids, including hydrocarbons, and like the linoleate is valuable for the emulsification of vegetable (and boiled) oils, animal and mineral oils for all purposes. In general, emulsions may be formed by adding an oil solution of a fatty acid (6—20%) to an aqueous solution (2—8%) of tri- β -hydroxyethylamine. The stearate is a hard soap of special value for the emulsification of waxes, and for polishes, cosmetics, etc. E. LEWKOWITSCH.

Separation of phenol mixtures, especially those obtained from brown-coal tar. G. SCHULZ and H. MENGELE (Z. angew. Chem., 1930, 43, 277—279).—The phenol mixture is fractionally distilled, the fractions being collected separately over every 5° range from 185° to 225° . Every fraction is then converted by means of dimethyl sulphate into a mixture of ethers; the b.p. of these from every 5° fraction are 30 — 35° below those of the original phenols and extend over a range of 17 — 30° . The ethers are fractionated four times, the receiver being changed at every 5° rise in temperature, and finally the ethers are converted into tribromoderivatives for identification. In this way the following phenols have been detected in the phenol mixture obtained from brown-coal tar: phenol, *m*-cresol, *o*- and *m*-ethylphenol, *o*-4-, *m*-2- and -4-, and *p*-2-xenols, *o*-isopropylphenol, and pyrocatechol. A. R. POWELL.

Absorption of cresol by active carbon. L. PIATTI and O. SPRECKELSEN (Z. angew. Chem., 1930, 43, 308—310).—Cresol cannot be quantitatively removed from active carbon in which it is absorbed by steam-blowing or by thermal distillation, although such treatment does not affect the adsorptive powers of the carbon. It may be completely withdrawn, however, by thoroughly extracting the carbon with organic solvents, *e.g.*, ethyl alcohol, in a Soxhlet extractor. S. K. TWEEDY.

Coal and carbon as raw materials. MATIGNON. **Products from shale retorting.** NASH.—See II. **Sulphonated oils.** RIESS; NISHIZAWA and WINOKUTI.—See XII. **Guaiacol and phenacetin.** SCHWYZER.—See XX.

See also A., May, 551, Catalytic reduction of carbon monoxide at ordinary pressure (KODAMA). 552, Hydrogenation over metallic caesium (HILL and KISTIAKOWSKY). Copper-zinc oxide catalysts (ABORN and DAVIDSON). 552, Catalysts for synthesis of methyl alcohol (NATTA). 553, Preparation of salicylaldehyde electrolytically (KAWADA and YOSIDA). 580, Electrolytic reduction of crotonaldehyde (SHIMA). 588, Preparation and application of higher esters of *p*-toluenesulphonic acid (SLOTTA and FRANKE). 593, 1-Methylnaphthalene and derivatives (STEIGER; VESELY and others). 607, Derivatives of hydroxyaminomethylantraquinones and dihydroxydianthraquinonylethylenes (DE DIESBACH and others). Reduction products of hydroxyanthraquinones (CROSS and PERKIN). 608, Perylene and its derivatives (ZINKE and WENGER). 616, Preparation of quinoline (DARZENS and others).

PATENTS.

Manufacture of butyl alcohol. E. NEUMANN (B.P. 326,812, 18.9. and 9.11.28).—Alcohol vapour, or a mixture of alcohol vapours, is led over a mixed catalyst consisting of a metal (for hydrogenation and dehydrogenation) and a metal oxide (for dehydration), *e.g.*, copper and alumina at 250°; copper and thoria at 200°; silver and alumina at 250°; copper, nickel, and thoria at 200°; copper and kaolin at 250°. Ethyl alcohol gives acetaldehyde, butyraldehyde, and butyl alcohol; propyl, isopropyl, or a mixture of ethyl and isopropyl alcohols similarly yield alcohols *etc.* containing C₅ and C₆.

C. HOLLINS.

Manufacture of condensation products containing nitrogen and sulphur [from acetylene, ammonia, and inorganic sulphides]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,795, 17.12.28).—Acetylene and ammonia are passed over iron pyrites at 350–450°. The products consist of nitriles, thiophen, pyridine bases, ethyl mercaptan, carbon disulphide, *etc.* and are useful as denaturants, flotation agents, *etc.*

C. HOLLINS.

Manufacture of nuclear iodination products of oxindole-3-propionic acid. SCHERING-KAHLBAUM A.-G., Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 459,361, 7.2.26. Addn. to G.P. 436,518; B., 1927, 378).—Iodinated oxindole-3-aldehydes are melted with malonic acid and the resulting acrylic acids are reduced. 5-Iodo-oxindole-3-aldehyde, decomp. above 250°, and the 5:7-di-iodo-compound, decomp. 204°, are thus converted into acrylic acids, which are reduced with sodium amalgam.

C. HOLLINS.

Preparation of fatty sulphonic acids and wetting, cleaning, and emulsifying agents therefrom. H. T. BÖHME A.-G. (B.P. 298,559, 21.9.28. Ger., 11.10.27. Addn. to B.P. 261,385; B., 1928, 418).—Oleic acid, or other unsaturated, non-hydroxylated, fatty acid, is sulphonated with sulphuric acid in presence of 1 mol. of acetic anhydride (or other organic acid anhydride or chloride).

C. HOLLINS.

Manufacture of sulphonic acids derived from non-aromatic carboxylic acids [wetting and emulsifying agents]. J. Y. JOHNSON. From I. G. FARBENIND.

A.-G. (B.P. 326,815, 12.12.28).—A non-aromatic acid or fat over C₈ (*e.g.*, stearic acid, oleic acid, coconut oil) is treated below 35° with not more than 3 mols. of a strong sulphonating agent (sulphur trioxide) in carbon tetrachloride, nitrobenzene, or other inert organic diluent to give wetting or emulsifying agents.

C. HOLLINS.

Manufacture of sulphonic derivatives of hydrogenated aromatic hydrocarbons [wetting agents *etc.*]. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER and G. BALLE (G.P. 459,045, 23.7.24. Addn. to G.P. 449,113; B., 1928, 846).—In the process of the prior patent the benzylated naphthalene is replaced by hydrogenated or partly hydrogenated aromatic hydrocarbons. *E.g.*, tetrahydronaphthalene and *n*-butyl alcohol are treated with chlorosulphonic acid at 30–50°. The products may subsequently be benzylated.

C. HOLLINS.

Propylation of *o*- and *p*-cresols. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 325,855, 30.8.28. Ger. 12.10.27. Cf. B.P. 298,600; B., 1930, 352).—Cresol vapour is passed with excess of propylene over a dehydrating catalyst (alumina, thoria, aluminium phosphate, kaolin, *etc.*) at 250–350°. *o*-Cresol yields mainly 3- and 5-isopropyl-*o*-cresols and a little carvacrol; *p*-cresol gives as main product 4-hydroxy-*m*-cymene, m.p. 36°, b.p. 228–229°, and a little 4-hydroxy-*o*-cymene, m.p. 37°, b.p. 248°.

C. HOLLINS.

Manufacture of arylcarboxyamido-*o*-thioglycolic acids [*o*-carbamylarylthioglycolic acids]. I. G. FARBENIND. A.-G. (B.P. 305,140, 30.1.29. Ger., 31.1.28).—The *o*-thiocyano-nitriles of B.P. 306,575 (B., 1929, 316) are hydrolysed, sometimes by way of the carbamylaryl thiocarbamates, NH₂·CO·Ar·S·CO·NH₂, to *o*-thiolarylcarboxylamides by sulphuric acid followed by alkali and hyposulphite. 5-Chloro-3-thiocyano-*o*-tolunitrile is converted by 90% sulphuric acid at 20–25° into 5-chloro-2-carbamyl-*m*-tolyl thiocarbamate, m.p. 182–183°, from which by action of alkali and hyposulphite, followed by condensation with chloroacetic acid the 5-chloro-2-carbamyl-*m*-tolylthioglycolic acid is obtained. 4-Chloro-2-thiocyanobenzonitrile gives with sulphuric acid at 20–25° 5-chloro-2-thiolbenzamide, m.p. 305°, which is condensed with chloroacetic acid to form 5-chloro-2-carbamyl-phenylthioglycolic acid, m.p. 206°. From 4-ethoxy-2-thiocyanobenzonitrile are obtained 4-ethoxy-2-thiolbenzamide, m.p. 285°, and a thioglycolic acid of m.p. 208–210°.

C. HOLLINS.

Manufacture of 2-mercaptoarylenethiazole compounds [vulcanisation accelerators]. I. G. FARBENIND. A.-G. (B.P. 306,558 and 306,842, [A] 23.2.29. Ger., 23.2.28; [B] 25.2.29. Ger., 24.2.28).—(A) A benz- or naphtha-thiazthionium chloride or hydroxide, such as is obtainable by action of sulphur chloride on arylamine, is treated with carbon disulphide and aqueous sodium sulphide, or with a di- or tri-thiocarbonate; the *o*-aminothiophenol presumably first formed is converted at once into the mercaptobenzthiazole. The thiazthionium chloride from *p*-phenetidine gives 6-ethoxy-2-thiolbenzthiazole, m.p. 198°. The 6-chloro-4-methyl- (m.p. 252°, from *o*-toluidine), 6-chloro-4-methoxy- (m.p. 233°, from *o*-anisidine), and 6-phenylamino-4-carboxy- [m.p. 300–305° (decomp.), from anthranilic

acid after an aniline treatment] derivatives are described. 2-Thiol- α -naphthathiazole, m.p. 235°, and 5-chloro-2-thiol- β -naphthathiazole, m.p. 304°, are obtained respectively from β - and α -naphthylamines. Benzidine gives 2:2'-dithiol-5:5'-dibenzthiazolyl. (B) 2-Thiolbenzthiazoles are similarly obtained by refluxing an *o*-thiocyanarylamine with aqueous sodium sulphide and carbon disulphide (etc.). The preparation of 6-chloro-2-thiol-4-methylbenzthiazole, m.p. 252°, 2-thiol-4:6-dimethylbenzthiazole, m.p. 263—265°, 2:6-dithiolbenzthiazole, m.p. 275°, and 2-thiol- α -naphthathiazole is described.

C. HOLLINS.

Manufacture of condensation products from aldehydes and amines and the application thereof in the manufacture of vulcanised rubber. CLAYTON ANILINE CO., LTD., and H. FRITZSCHE (B.P. 326,525, 12.11.28).—Vulcanisation accelerators are made by condensing an aldehyde (more than 1 mol.) with the product from an amine and an aldehyde-bisulphite compound; e.g., formaldehyde is condensed with sodium anilinomethanesulphonate, acetaldehyde or crotonaldehyde with sodium α -anilinoethanesulphonate.

C. HOLLINS.

Manufacture of compounds having hydrogenated ring-systems. I. G. FARBENIND. A.-G., Assees. of O. DIELS and K. ALDER (B.P. 300,130, 5.11.28. Ger., 5.11.27).—Maleic, itaconic, and citraconic anhydrides, acrylic acid, acraldehyde, and other non-quinonoid compounds containing the grouping C:C:C:O react exothermically in absence of a condensing agent with butadiene, cyclopentadiene, phellandrene, myrcene, and other not purely aromatic compounds containing conjugated carbon double linkings, with the production of hydrogenated cyclic or polycyclic compounds. From maleic anhydride and cyclopentadiene in cooled benzene solution there is obtained 3:6-endomethylene- Δ^4 -tetrahydrophthalic anhydride, m.p. 164—165° (acid, m.p. 177—179°; reducible to the hexahydro-acid, m.p. 160—161°). Butadiene gives *cis*- Δ^4 -tetrahydrophthalic acid, m.p. 166° (anhydride, m.p. 103—104°), and dihydrobenzene yields 3:6-endoethylene- Δ^4 -tetrahydrophthalic anhydride, m.p. 147°. 3:6-endoMethylene- Δ^4 -tetrahydrobenzaldehyde, b.p. 70—72°/20 mm. (bisulphite compound; semicarbazone, m.p. 160°; cyclohexanone compound, m.p. 104—105°), and the corresponding benzoic acid, b.p. 128—130°/15 mm., are obtained from cyclopentadiene and acraldehyde and acrylic acid, respectively.

C. HOLLINS.

Manufacture of compounds of the general formula

$R \cdot CH < \begin{smallmatrix} \text{C} \\ \text{C} \end{smallmatrix} > Ar$, especially derivatives of 1:3-

diketohydrindene [indan-1:3-dione]. T. K. and L. M. WALKER, H. SHAW, and R. BLACK (B.P. 326,467, 12.11.28).—A monoalkylmalonyl halide is condensed with a phenol, or a phenol ether or ester, in presence or absence of a solvent (nitrobenzene, carbon tetrachloride, etc.) and in presence of aluminium chloride or similar condensing agent. *n*-Butylmalonyl chloride and *p*-cresol (or its methyl ether or acetate) give 7-hydroxy-4-methyl-2-(or its methyl ether or acetate) 1:3-dione, m.p. 165°; the 2-*n*-propyl-*n*-butylindan-1:3-dione, m.p. 187°. Ethylmalonyl chloride and resorcinol dimethyl ether yield, with loss of a methyl

group, 5:7- or 7:5-hydroxymethoxy-2-ethylindan-1:3-dione, m.p. 192·5°. β -Naphthyl methyl ether condenses with *n*-butylmalonyl chloride in the 1:8-position to give a product, m.p. 182°. The indandiones may be reduced to indanes, e.g., 7-hydroxy-1-methyl-2-*n*-butyl- and 2-*n*-propyl-indanes, b.p. 207—210°/21 mm. and 190°/18 mm., respectively.

C. HOLLINS.

Manufacture of α -tetrahydronaphthols and their esters and ethers. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,762, 16.11. and 10.12.28).—A naphthol ester or ether is hydrogenated in a solvent in presence of nickel, nickel-cerium, etc. The following compounds are described; α -tetrahydro- α -naphthol, m.p. 68° (acetate, m.p. 73—75°, b.p. 142—144°/10 mm.; benzoate, m.p. 46°; carbonate, m.p. 114°; methyl ether, b.p. 113°/7 mm.; ethyl ether, b.p. 122°/9 mm.); α -tetrahydro- β -naphthol (acetate; carbonate, m.p. 106°; methyl ether, b.p. 118°/9 mm.; ethyl ether, b.p. 123°/8 mm.; *n*-propyl ether, b.p. 140°/10 mm.; isopropyl ether, b.p. 135—136°/9 mm.; isobutyl ether, b.p. 146—148°/12 mm.); 2-hydroxy-5:6:7:8-tetrahydro-3-naphthoic acid, m.p. 176° (methyl ester O-acetate, m.p. 94°, b.p. 193°/5 mm.; ethyl ester O-acetate); 2-hydroxy-1-methyl-5:6:7:8-tetrahydronaphthalene acetate, b.p. 156°/9 mm.; 1:4-dimethoxy-5:6:7:8-tetrahydronaphthalene, b.p. 138—140°/5 mm.

C. HOLLINS.

Manufacture of halogen-naphthalene ketones.

I. G. FARBENIND. A.-G. (B.P. 301,311, 23.11.28. Ger., 26.11.27).—Aromatic acid halides or anhydrides react with di- and poly-halogenated naphthalenes in presence of aluminium chloride etc. to give naphthyl ketones. The preparation of phenyl 5:8-dichloro- α -naphthyl ketone, m.p. 104—105°; phenyl 4:8-dichloro- α -naphthyl ketone, m.p. 112°, b.p. 270—275°; *o*- and *p*-chlorophenyl 5:8-dichloro- α -naphthyl ketones, m.p. 122—123° and 118°, respectively; 5:8-dichloro- α -naphthyl ketone, m.p. 163—164°; 5:8-dichloro- α -naphthyl methyl ketone, b.p. 250°/13 mm.; and of a ketone from 1:4-dichloronaphthalene and phthalic anhydride is described.

C. HOLLINS.

Manufacture of arylated quinones and hydroxy-arylquinones. R. PUMMERER (G.P. 459,739, 22.3.22; cf. Pummerer and Prell, A., 1922, i, 1164).—Benzo- and naphtha-quinones are condensed in presence of hydrogen chloride and aluminium chloride or zinc chloride with aromatic hydrocarbons, or aromatic compounds containing no free hydroxy-, thiol, or primary or secondary amino-groups; or with monohydric phenols in a diluent.

C. HOLLINS.

Manufacture of reduction products of indoxyl, naphthindoxyl, their homologues and acyl derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,523, 6.11.28).—Indoxyls are reduced by hydrogen at suitable pressure and temperature in presence of a catalyst (nickel-copper, copper-cobalt or -nickel, on kieselguhr) to dihydroindoxyls and dihydroindoles; the former may be dehydrated to form indoles. The preparation of 2:3-dihydroindoxyl, m.p. 92—93° (N-acetyl derivative, m.p. 158°), indole, 7-methylindoxyl (ON-diacyl derivative, m.p. 118—120°), 7-methyldihydroindoxyl, 7-methylindole, m.p. 84—85°, N-acetyl- $\beta\beta$ -naphthindoxyl, m.p. 182° (dihydro-compound, m.p.

175—180°), *N*-acetyldihydro- $\beta\beta$ -naphthindole, m.p. 118—120°, and $\beta\beta$ -naphthindole, m.p. 62—63°, is described.

C. HOLLINS.

Manufacture of [olefine - polynuclear hydrocarbon] condensation products. R. MICHEL, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,741,473, 31.12.29. Appl., 3.12.28. Ger., 8.12.27).—See B.P. 323,100; B., 1930, 363.

Preparation of condensation products of ethyl alcohol. W. O. HERRMANN and E. BAUM, ASSRS. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,755,692, 22.4.30. Appl., 2.12.27. Ger., 18.12.26).—See B.P. 282,448; B., 1929, 426.

Production of acetone. L. SCHLECHT, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,755,193, 22.4.30. Appl., 23.11.28. Ger., 16.12.27).—See B.P. 313,897; B., 1929, 708.

Preparation of dihydroxyacetone. W. LENZ, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,755,656, 22.4.30. Appl., 20.4.27. Ger., 26.4.26).—See B.P. 269,950; B., 1928, 313.

Manufacture of esters of the borneols. K. STEPHAN and F. ULFFERS, ASSRS. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,755,750 and 1,755,752, 22.4.30. Appl., 30.3.26. Ger., 11.4.25).—See B.P. 250,555; B., 1926, 995.

Manufacture of the dehydration product of sorbitol. J. MÜLLER and U. HOFFMANN, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,757,468. 7.1.28. Ger., 7.6.27).—See B.P. 301,655; B., 1929, 123.

Sulphurised compounds of phenols. A. THAUSS, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,757,400, 6.5.30. Appl., 7.7.27. Ger., 15.7.26).—See B.P. 298,280; B., 1928, 886.

Substitution of alkyl groups in the aromatic nucleus. A. VERLEY (U.S.P. 1,756,575, 29.4.30. Appl., 7.7.27. Fr., 31.3.27).—See B.P. 288,122; B., 1929, 47.

[Production of] 6-chloro-2-amino-1-methylbenzene-4-sulphonic acid. F. HENLE and B. VOSSEN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,755,648, 22.4.30. Appl., 6.6.28. Ger., 1.9.24).—See G.P. 434,402; B., 1927, 276.

Manufacture of a compound of isonaphthyridin. C. RÄTH (U.S.P. 1,755,515, 22.4.30. Appl., 10.11.26. Austr., 17.10.25).—See B.P. 259,973; B., 1928, 244.

Manufacture of cyclic ketones of the aromatic series [indanones]. F. MAYER, K. BILLIG, K. HORST, and K. SCHIRMACHER, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,031, 8.4.30. Appl., 15.3.27. Ger., 16.3.26).—See B.P. 288,441; B., 1928, 441.

Hydrogenation of hydrocarbons (B.P. 302,912).—See II.

IV.—DYESTUFFS.

See also A., May, 595, Azomethine azo dyes (SEN and SEN). 605, Sulphur-containing dyes (BETRABET and CHAKRAVATI). 606, Colouring matters of fungi (KÖGL and ERNLEBEN). 614, Reactions of magnesium pyrrol and indolyl compounds (NENITZESCU). 616,

Action of amines on benzylquinoline chloride (MICHAILENKO and MINOFIEV).

PATENTS.

Manufacture of derivatives of anthanthrone [vat dyes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,874, 22.12.28).—Alkoxyanthanthrones are halogenated, preferably in a diluent (chlorosulphonic acid or nitrobenzene) in presence of iodine etc. to give vat dyes. Methoxyanthanthrone gives on bromination a red, on chlorination a yellowish-red; a bluish-red is obtained by brominating ethoxyanthanthrone.

C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 302,173, 10.12.28. Ger., 10.12.27).—Imides of 4-amino-1:8-naphthalic acid are diazotised and coupled in substance or on the fibre with 2:3-hydroxynaphthoic arylamides. The methylimide, e.g., gives with the 2:5-dimethoxyanilide a bluish-claret, with the α -naphthylamide a bluish-bordeaux, with the dianisidide violet-brown: the imide is coupled with the 2:5-dimethoxyanilide (claret-red), anilide (violet), α -naphthylamide (violet), *p*-anisidide (violet), or dianisidide (chocolate-brown).

C. HOLLINS.

Production of monoazo dyes from aminobenzyl- ω -sulphonic acid. W. DUISBERG, W. HENTRICH, and L. ZEH, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,755,871 and 1,757,419, [A] 22.4.30, [B] 6.5.30. Appl., [A, B] 12.2.26. Ger., [A] 19.2.25, [B] 13.2.25).—See (A) B.P. 265,767, (B) F.P. 611,004; B., 1927, 325, 404.

Production of azo dyes [pigments and ice colours]. O. SOHST, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,754,652, 15.4.30. Appl., 26.6.26. Ger., 7.7.25).—See B.P. 255,072; B., 1927, 743.

Manufacture of (A) azo, (B—D) secondary disazo, dyes. BRIT. DYESTUFFS CORP., LTD., ASSEES. OF (A, B) J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN, and (C, D) J. BADDILEY (U.S.P. 1,757,496, 1,757,499, 1,757,500, and 1,757,506. Appl., [A] 2.3.27, [B, C] 21.5.27, [D] 3.5.29. U.K., [A] 25.6.26, [B] 8.9.26, [C] 10.12.26, [D] 13.12.26).—See B.P. 277,756, 281,767, 287,609, 287,214; B., 1927, 869; 1928, 118, 402, 402.

Preparation of dyes derived from indoline bases. O. WAHL, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,755,678, 22.4.30. Appl., 26.4.27. Ger., 3.5.26).—See B.P. 291,888; B., 1928, 599.

Manufacture of indigoid dyes. R. STOCKER, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,755,972, 22.4.30. Appl., 7.7.26. Switz., 29.7.25).—See B.P. 236,332; B., 1925, 703.

Colloidally dispersed pigments (B.P. 326,516).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of fat in washed wool. P. KRAIS and K. BILTZ (Chem. Umschau, 1930, 37, 81—82).—If washed wool be boiled with very dilute sulphuric acid after the usual extractions of fatty matter etc., further small ether and alcohol extracts (substances in the wool unconnected with the washing process?) can

be obtained. Further, the ordinary alcohol extract contains substances other than soaps or fat, and for an accurate knowledge of the state of the washed wool the alcohol extract should be diluted with water, acidified, and extracted with ether, the fatty matter obtained titrated with alkali, and the result calculated to oleic acid. This figure added to that for the ether extract of the wool ("wool fat") gives total fatty matter, or multiplied by 1.08 expresses the anhydrous soap content.

E. LEWKOWITSCH.

Wood-pulp viscose. II. A. LOTTERMOSER and H. RADESTOCK (Z. angew. Chem., 1929, 42, 1151–1154; cf. B., 1928, 189).—The influence of pulp origin, concentration of caustic soda, duration of mercerising, maturing of alkali crumbs, and ripening of viscose has been investigated. Great variations of initial viscosity are in part dependent on pulping and more so on bleaching; excessive bleaching greatly diminishes viscosity. Mercerising with 17½% caustic soda for 2 hrs. gives good dispersions for all pulps; below 17½% good dispersion is not obtainable with certainty, but a 30% solution behaves as does a 17½% solution. Insufficient mercerising cannot be rectified by modifying sulphiding. Increasing the mercerising period diminishes viscosity up to a period of 24 hrs., after which it increases until 90 hrs.' mercerising gives the same viscosity as 2 hrs. More viscous pulps suffer higher percentage diminution of viscosity than do pulps of lower viscosity when over-mercerised. Atmospheric oxygen has a lowering influence on viscosity. Maturing the crumbs also diminishes the viscosity, the effect being most marked during the first 24 hrs. and depending on the concentration of caustic soda used. All viscosities were measured on dispersions of 1 g. of pulp per 200 c.c. of viscose.

C. J. J. Fox.

Viscose. III. Influence of oxygen on the ripening of viscose. A. LOTTERMOSER and F. SCHWARZ (Z. angew. Chem., 1930, 43, 16–19; cf. preceding abstract).—During the preliminary ripening of alkali-cellulose in air a reaction takes place with the oxygen of the air which causes the resulting viscose solution to become more fluid. Alkali-cellulose, removed from the sodium hydroxide solution in which it is prepared and allowed to ripen in nitrogen or hydrogen, yields, on xanthation, a viscose solution of higher initial viscosity than that yielded by similar material ripened in oxygen. Subsequent ripening of the viscose solution in nitrogen results in a rapid decrease of viscosity for 24–30 hrs., followed by a steady increase until gelatinisation ensues; at the same time the liquid becomes gradually darker in colour owing to the formation of sodium trithiocarbonate. Viscose solutions made from alkali-cellulose pre-ripened in air or oxygen have, when ripened in oxygen, a low viscosity which rapidly decreases nearly to that of sodium hydroxide solution, and remains constant until gelatinisation occurs; in this case the solution gradually becomes almost colourless and, on acidification, yields sulphur dioxide and carbon dioxide but no hydrogen sulphide. Ripening in air follows an intermediate course between these two extremes. These results indicate that oxygen causes considerable oxidation of cellulose in both ripening stages in the manufacture of viscose silk, and is thus probably the cause of the lower

tensile strength of this type of silk as compared with that of cuprammonium silk, the preparation of which is carried out in the absence of air. A. R. POWELL.

Viscose. IV. Reason for the dependence of the cross-section of viscose filaments on the composition of the coagulating bath. A. LOTTERMOSER and C. SCHIEL (Z. angew. Chem., 1930, 43, 80–81; cf. preceding abstract).—Investigation of osmotic pressure differences between viscose solutions and coagulating-bath liquids shows that when a viscose solution is spun into a coagulating bath a skin of regenerated cellulose is first formed which surrounds the viscose solution. This skin acts as a semi-permeable membrane, and since the osmotic pressure of the coagulating liquid is much higher than that of the viscose solution, water passes from the latter to the former. This causes a partial collapse of the skin and produces a filament of irregular cross-section when coagulation is complete. The more concentrated in sodium bisulphate is the bath, the higher is its osmotic pressure and the more pronounced the collapse of the skin, and hence the more irregular is the shape of the cross-section. This effect is further promoted by reason of the fact that the porosity of the skin increases with increasing salt concentration of the bath.

B. P. RIDGE.

Ripening of alkali-cellulose and the viscosity of the viscose. Y. KAMI, K. KITAZAWA, and T. YAMASHITA (J. Cellulose Inst., Tokyo, 1930, 6, 55–66).—At normal temperature the curve expressing the relation between time of ripening and the viscosity of the viscose is hyperbolic in shape, but the lower the temperature the less is its curvature, so that with decreasing temperature the curves approach nearer and nearer to a straight line parallel with the time axis. The viscosity shows a minimum at about 9% soda content of the viscose, and the clearness of the liquid increases with soda content, whilst its colour changes from dark to reddish-brown. In the case of viscoses of about 9% soda content, the influence of the soda content of the alkali-cellulose (degree of pressing) is very small, but for higher or lower contents it is very marked, because homogeneous viscose is then obtained only with difficulty. Optimum conditions for homogeneity of the alkali-cellulose are obtained by pressing the latter to about 3.2 times the weight of cellulose used. If the viscosity-time of ripening curves are drawn for different temperatures of ripening, and if the angle of inclination of the tangents to these curves is Σ , the points of contact of the tangents satisfied by the equation $\tan \Sigma = \frac{1}{2}$ lie on a straight line. This line makes an angle ϕ with the time axis which is characteristic of the cellulose concerned, and the determination of this line furnishes a good method of testing the cellulose.

B. P. RIDGE.

Treatment of alkaline drain from [a Japanese] viscose factory. K. TANEMURA and T. KOHNO (J. Cellulose Inst., Tokyo, 1930, 6, 67–75).—The sulphide wash, drain from the viscose cellar, and the alkaline drain liquors have been analysed and conditions of treatment of the effluents under which viscose and sulphides may be removed without neutralisation or acidification have been studied. Satisfactory results are obtained by the

addition of zinc and/or ferrous sulphate, provided that the p_H of the liquor is controlled on the alkaline side during precipitation. After settling, the supernatant liquid is colourless and contains neither sulphide and viscose nor the precipitant. When zinc sulphate is used as an ingredient of the spinning bath, the excess liquid from these baths may be used for this treatment.

B. P. RIDGE.

Preparation of acetate artificial silk. F. OHL (Chem.-Ztg., 1930, 54, 202).—A brief review of the above industry.

H. F. HARWOOD.

Beating of [paper] pulp. VII. **Beating degree and copper number.** M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 75—76; cf. B., 1930, 412).—As a result of an investigation of the relation between copper number and degree of beating it is shown that (1) the copper number of the beaten stuff is generally higher than that of the original pulp, (2) the rate of increase of the degree of beating of the stuff does not depend on the initial copper number of the unbeaten pulp, and (3) there is no relation between the degree of beating and the copper number of the beaten stuff.

B. P. RIDGE.

Swelling of cellulose esters. A. EICHENGRÜN (Z. angew. Chem., 1930, 43, 236—237).—By treatment with suitable swelling media, cellulose esters undergo a considerable increase of volume, without essential change of structure, and are easily worked in this state. The author's results differ from those of Hess (B., 1930, 412).

J. A. V. BUTLER.

Interim report of the Pulp Evaluation Committee (Proc. Tech. Sect. Papermakers' Assoc., 1929, 10, 4—42).—An apparatus has been designed, and is described, for the purpose of making standard sheets for testing the initial (unbeaten) strength of pulps. The apparatus consists essentially of a disintegrator, a sheet-making machine comprising a cylindrical deckle-box, 6½ in. in diam. with a 150-mesh wire, and constant-head drainage system, a couch-roll, a sheet press, stainless-steel drying plates, and a device for clamping the sheets at the edges during drying. A sample of pulp equivalent to 24 g. of bone-dry fibre is thoroughly wetted out, made up to 2000 c.c., and disintegrated for 75,000 revolutions at 3000 r.p.m. Mechanical pulps are diluted to 8 litres; chemical pulps to 16 litres. 800 C.c. of chemical pulp stock, or 420 c.c. of mechanical, are poured into the container of the sheet-machine, water being run in to a height of 35 cm. above the wire. After gentle agitation to distribute the fibres, the drain-cock is opened, the sheet allowed to drain for ½ min. after the bulk of water has disappeared, and the container removed. The sheet is couched off the wire by means of blotters, and is transferred to a stainless-steel plate. Seven sheets are made in this way, the drying plates being alternated with blotters and the stack pressed at 50 lb./in.² for 5 min. The order of the sheets is then reversed and the pressing repeated. The sheets are air-dried, being clamped at the edges to prevent shrinkage. An "M.G." finish is imparted to the sheets. After conditioning at 65% R.H. and 70° F., tests are made for thickness, basis weight, tensile strength, tearing strength, and bursting strength.

The strengths are reported as factors independent of the basis weight. In dealing with mechanical pulps, the Canadian freeness tester is recommended for checking the fineness. The method has been adopted as standard by the Papermakers' Association. The experimental work carried out by the Committee is described in a separate publication. A full bibliography is appended.

T. T. PORTS.

Drying of solids. III. **Mechanism of the drying of pulp and paper.** T. K. SHERWOOD (Ind. Eng. Chem., 1930, 22, 132—136; cf. B., 1929, 1035).—The drying operation is divisible, in general, into two periods—a constant-rate period and a falling-rate period. During the former the surface of the solid is uniformly wetted and the rate of drying is independent of the water content and the thickness of the slab. The falling-rate period is usually further divisible into two phases, either or both of which in turn may control during this period. In the first phase the reduced rate of drying is due to a decrease in the area of wetted surface (unsaturated surface drying), and is independent of the thickness of the material being dried. In the second the rate of drying is controlled by the rate of internal liquid diffusion, and is then a function of the thickness of the slab. Experiments are cited which show that for thick pulp slabs internal liquid diffusion is the controlling factor during the falling-rate period of drying, whilst for thin pulp sheets and paper the mechanism during the falling-rate period is that of unsaturated surface-drying. An empirical equation is given showing the relationship between water content and time for the drying of pulp and fibre boards.

D. J. NORMAN.

Performance of [heat] economisers in the drying of pulp and paper. F. W. ADAMS and C. M. COOPER (Ind. Eng. Chem., 1930, 22, 127—129).—The results of a series of tests on a Briner-type economiser indicate that when heat is recovered solely for preheating air for the dryer, the maximum heat recoverable is less than one third of the total heat available in the outgoing air, and of this amount an actual recovery of 50—60% may be expected. When, however, there is a demand outside the dryer system for large quantities of warm air for ventilating purposes, the efficiency of heat recovery is greatly improved. A hypothetical case is worked out to show the financial advantages of using economisers.

D. J. NORMAN.

Cooking process. I. **Rôle of water in the cooking of wood.** S. I. ARONOVSKY and R. A. GORTNER (Ind. Eng. Chem., 1930, 22, 264—274).—The effect of digestion under varying conditions of a number of wood dusts is examined. Changes in the amounts of sugars, pentoses, pentosans, lignin, furfuraldehyde, and volatile acids obtained in the cooking liquor at different temperatures are recorded, together with the corresponding proportions of cellulose, α -cellulose, pentosans, and lignin in the wood residue. During digestion for 2 hrs. at temperatures up to 186°, changes occurring in the wood result from the direct action of water, and are not secondary effects of acids formed in the process.

A. G. POLLARD.

Carbonyl groups of lignin and its sulphonates and sulphate pulp digestion. E. HÄGGLUND, T.

JOHNSON, and L. H. TRYGG (Cellulosechem., 1930, 11, 30—38).—Investigations have been made to establish the presence of active carbonyl groups in lignin hydrochloride prepared by the authors' method (B., 1929, 241). The presence of these groups is masked, but treatment with acid in presence of phenols and aromatic amines discloses their presence. Phenol-lignin does not behave thus, and appears to be a condensed ring product formed by interaction of phenolic hydroxyl and ligninaldehyde to form an acetal compound. Sulphonation of lignin in the pulping process proceeds directly and carbonyl groups are masked in consequence; if lignin is not fully sulphonated before the buffer action of sulphite ceases, owing to its interaction, "black cook" is unavoidable. The acidity of the cooking solution remains constant until the buffer action fails; until then the cooking solution and pulp are almost colourless. Bisulphited carbonyl groups act protectively, but later diminution of bisulphite concentration and intramolecular changes lead to decomposition of bisulphited carbonyl groups, darkening of liquor, and corresponding changes in pulp colour. These facts are confirmed by colour changes obtained with fuchsin reagent. C. J. J. FOX.

Corrosion problems in the rayon industry. P. C. KINGSBURY (Ind. Eng. Chem., 1930, 22, 130—132).—The advantages of stoneware plant for the storage of corrosive liquids are outlined. Vessels of 2000-litre capacity are the largest that can be satisfactorily manufactured in quantity, but a number of these may be connected up by a siphoning system to resemble in operation one large storage tank. D. J. NORMAN.

Action of liquids on paper. J. STRACHAN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 59—70).—A discussion of a paper already abstracted (B., 1930, 97).

China clay. AITKEN.—See VIII. **Air-conditioning in the press room.** GRAHAM.—See XXIII.

See also A., May, 538, **Adsorption of salts by artificial silk** (HELLAT).

PATENTS.

Manufacture of textile fibres from peat. I. WEGMAN (B.P. 325,904, 8.12.28).—The tensile strength, appearance, and spinning properties of peat fibre as ordinarily prepared are improved by a treatment for 20—30 days in wooden vats with dilute solutions of plant extracts and/or resin emulsions with addition of small quantities of metal salts or mixtures thereof. After rinsing, the fibres are finally treated with oxygen or solutions of oxidising agents. The plant extract is preferably prepared from plants of the *Labiata*, *Aristolochiaceae*, or like family. D. J. NORMAN.

Production of artificial (A) filaments and threads, (B) textile yarns or threads. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 326,462 and 325,823, [A] 28.9.28, [B] 21.11.28).—(A) A "unitary" filament of relatively large cross-section is obtained from twisted or untwisted bundles of filaments by moistening them with a swelling agent so that the individual filaments stick together. The "unitary" filament may contain different types of artificial filaments or yarns or even metallic wires.

Any tackiness remaining after adhesion of the filaments is removed by the application of lubricants. Suitable apparatus is described. (B) The bundles of filaments are treated at intervals with a swelling agent instead of continuously. D. J. NORMAN.

Extraction of cellulose. A. THIRIET (B.P. 325,570, 20.11.28).—Cellulosic material is subjected to successive treatments under atmospheric pressure with hot caustic alkali solution of gradually increasing concentration, the caustic alkali solution circulating in a more or less continuous flow through a series of vats so that the final purification of the cellulose is effected with soda solutions that are free from organic matter. Suitable apparatus is described. D. J. NORMAN.

Production of pulp. EUROMERICAN CELLULOSE PRODUCTS CORP., Assecs. of E. R. DARLING (B.P. 314,061, 6.6.29. U.S., 22.6.28).—Cellulose pulp is prepared from straw, oat hulls, and similar material containing pentosans, by digesting the shredded and washed material first with water, then with dilute acid, e.g., 1% sulphuric acid to hydrolyse the pentosans, and finally with dilute alkali solution. Each or all of these operations may be conducted under pressure. D. J. NORMAN.

Machine for dissolving [cellulose] xanthate. R. THURM and E. SCHMIERER, Assrs. to BAKER PERKINS Co., INC. (U.S.P. 1,742,226, 7.1.30. Appl., 10.9.27).—A receptacle contains an open-ended drum fixed therein above a series of radially-disposed bars; this, in turn, is mounted above a co-axial, rotatable, bladed impeller. The material being dissolved passes through the impeller and bars and thence through the drum, and is impeded from flowing away laterally from the impeller by means of a grille arranged as a circular wall outside, and close to, the tips of the impeller blades, which tips may be constructed as shear edges for better disintegration of the material. A second, finer-meshed, removable grille may also be provided. S. K. TWEEDY.

Preparation of plastic composition articles and method of preparing the same. BRIT. CELANESE, LTD. (B.P. 303,516, 4.1.29. U.S., 6.1.28. Addn. to B.P. 282,723; B., 1928, 853).—The mixtures of finely-powdered cellulose esters and plasticisers prepared according to the prior process are moulded under heat and pressure. Temperatures of 50—80°, preferably 70°, and pressures of 300—500 lb./in.² are suitable for a period of 2—3 hrs. D. J. NORMAN.

Production of paper. L. MELLERSH-JACKSON. From MATHIESON ALKALI WORKS (B.P. 326,956—8, 15.2.29).—(B) Chlorine is used for the sterilisation of the stock and for this purpose is conveniently introduced continuously into the mixing boxes ahead of the paper machine in sufficient quantity to leave 0.5—1 p.p.m. of free chlorine. (A) A coagulating agent, e.g., alum, may be added at the same time to effect coagulation of the colloidal material. This chlorination treatment prevents slime and pitch formation, increases the hydration of the stock, and at the same time causes it to run freer on the machine. (C) Chlorine is added at or near the beginning of the beating operation to sterilise the stock and thereby prevent the formation of pitch

and beater gum during the beating operation (cf. B.P. 324,547; B., 1930, 368).

D. J. NORMAN.

Production of paper-, artificial leather-material, and the like from leather waste. M. M. SEREBRIANY (B.P. 326,936, 30.1.29).—Leather waste is swollen and degreased in hot water containing about 5% of salt or turpentine, and is then moulded in hot or cold presses. Alternatively, the swollen mass may be pressed into log-shaped pieces, which are then converted into fibrous material and run on a paper or board machine optionally after the addition of vegetable or animal fibrous material or fillers.

D. J. NORMAN.

Manufacture of composite sheet material. WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 309,019, 9.1.29. Ger., 3.4.28. Addn. to B.P. 304,722; B., 1930, 368).—Improved results are obtained by using as the agglutinant a protein, *e.g.*, gelatin, dissolved in a solvent such as glacial acetic acid which is also a solvent for the cellulose derivative which does not swell in water. If desired, cellulose derivatives which swell in water may be used instead of the regenerated cellulose specified in the prior patent.

D. J. NORMAN.

Manufacture of artificial threads of cellulose derivatives. K. HOFFMANN (U.S.P. 1,754,427, 15.4.30. Appl., 11.3.26. Ger., 12.3.25).—See B.P. 249,141; B., 1927, 184.

Production of porous paper. G. L. SCHWARTZ, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,757,757, 6.5.30. Appl., 6.6.28).—See B.P. 313,085; B., 1930, 414.

[Apparatus for] conversion of vegetable fibres into fibres resembling wool. TEXTILES (NEW PROCESS), LTD., Assees. of J. VIALLET (B.P. 309,020, 15.1.29. Fr., 3.4.28).

Composite [cotton-jute heat-]insulating material. UPSON Co., Assees. of C. A. UPSON and H. McC. SPENCER (B.P. 317,363, 23.2.29. U.S., 14.8.28).

Production of artificial [silk] ribbons, tapes, straws, etc. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 328,312, 21.11.28).

Devices for winding artificial silk threads. O. (FRH.) VON KOHORN (O. KOHORN & Co.), and A. PERL (B.P. 316,252, 17.5.29. Austr., 26.7.28).

[Apparatus for] washing of pulp [for use as filtering-medium]. BLAIR, CAMPBELL, & McLEAN, LTD., and S. HUTCHIN (B.P. 328,090, 20.3.29).

Making paper of definite thickness. A. E. O'DELL. From FREIBERGER PAPIERFABR. ZU WEISSEN-BORN (B.P. 327,655, 25.7.29).

Protection against textile pests (B.P. 326,451 and 326,567).—See VI. Waste alkali liquors (B.P. 314,812).—See VII. Impregnating insulating material (B.P. 297,035).—See XI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Protection of fibres. ORANIENBURGER CHEM. FABR. A.-G., Assees. of CHEM. FABR. MILCH A.-G. (B.P. 289,898, 5.5.28. Ger., 6.5.27).—Textile fibres are protected

during treatment with acids or alkalis as in bleaching, scouring, carbonisation, degumming, and tanning, by addition to the treatment liquors of water-soluble, halogen-substituted sulphonic acids or their salts prepared from aliphatic or hydroaromatic substances of high mol. wt. containing at least 10 carbon atoms in the molecule. [Stat. ref.]

A. J. HALL.

Dyeing of animal fibres with vat dyes. I. G. FARBENIND. A.-G., Assees. of F. JUST, K. DAIMLER, and G. BALLE (G.P. 457,802, 20.12.23).—Wool is dyed from a weakly alkaline, neutral, or weakly acid bath with a leuco-vat dye maintained in solution by means of benzylnaphthalenesulphonic acid resins etc.

C. HOLLINS.

Dyeing with the aid of diazo compounds [ice colours]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,866, 19.12.28).—The stability of diazo solutions in presence of metallic iron is improved by addition of nitrous acid (sodium nitrite) so that ferrous ions are oxidised to ferric.

C. HOLLINS.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assees. of R. METZGER (G.P. 457,957, 23.4.22).—Acetate silk etc. is dyed with carboxylated azo dyes (other than derivatives of salicylic acids). Examples are: *p*-aminophenoxyacetic acid \rightarrow β -naphthol (red-orange); *p*-aminobenzoic acid \rightarrow *m*-phenylenediamine (orange); α -naphthylamine \rightarrow *p*-hydroxybenzoic acid (gold-yellow); *p*-nitroaniline \rightarrow 4-hydroxy- α -naphthoic acid (brown-red).

C. HOLLINS.

Decorative treatment of textile fabrics. CALICO PRINTERS' ASSOC., LTD., and J. D. WEBSTER (B.P. 326,823, 20.12.28).—The fabric is prepared on one side by means of an engraved padding or printing roller with a coupling component, dried, and prepared on the other side with a different coupling component, and, after drying, a single application of a diazo compound is made, which penetrates the fabric and produces the same pattern on both sides in different shades. An example is Naphthol AS and AS/G with three-colour pattern of Fast black salt K, 5-nitro-*o*-anisidine, and *m*-nitroaniline. A printing roller of exceptional depth ("bang-through" engraving) is used.

C. HOLLINS.

Protection of materials from the attack of moth and other pests. Protection of wool, fur, hair, etc. against textile pests. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. [A] 326,451, 10.12.28, and [B] 326,567, 20.12.28).—(A) The hydroxylated condensation products obtained from phenols of the benzene series and hydroaromatic cyclic ketones are used for moth-proofing wool, fur, etc. The example is the product from phenol and cyclohexanone. (B) Symmetrical disubstituted thiocarbamides, *e.g.*, valerylphenyl- and hexoylphenyl-thiocarbamides are similarly employed.

C. HOLLINS.

Dyeing of cellulose esters or ethers. E. FISCHER, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,755,640, 22.4.30. Appl., 9.9.26. Ger., 17.9.25).—See B.P. 258,611; B., 1927, 874.

Dyeing of regenerated cellulose materials. BRIT. DYESTUFFS CORP., LTD., Assees. of (A, D, E, F, H) J. BADDILEY, and (B, C, G) J. BADDILEY, P. CHORLEY,

and R. BRIGHTMAN (U.S.P. 1,757,158, 1,757,497—8, 1,757,501—5, 6.5.30. Appl., [A] 8.3.28, [B, C] 16.3.27, [D] 21.5.27, [E] 26.5.27, [F] 3.12.27, [G] 6.7.28, [H] 11.12.28. U.K., [A, F] 10.12.26, [B] 16.4.26, [C] 7.9.26, [D] 13.12.26, [E, H] 7.9.26, [G] Ger., 2.5.27).—See B.P. 283,319, 270,883, 281,410, 287,214, 280,320, 281,767; B., 1928, 189; 1927, 520; 1928, 121, 402, 49, 118.

[Carroting] treatment of animal hair and wool with chlorine. E. BÖHM (U.S.P. 1,756,723, 29.4.30. Appl., 22.11.27. Austr., 1.12.26).—See B.P. 281,646; B., 1928, 520.

Printing colours. K. H. MEYER, J. MÜLLER, and W. HOFFMANN, ASSS. to I. G. FARBENIND. A.-G. (U.S.P. 1,756,951, 6.5.30. Appl., 7.1.28. Ger., 7.1.27).—See B.P. 292,655; B., 1928, 681.

Treatment of materials made of or containing cellulose derivatives. G. H. ELLIS, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,757,519, 6.5.30. Appl., 25.3.27. U.K., 30.10.26).—See B.P. 280,698; B., 1928, 86.

[Apparatus for] washing, desulphurising, bleaching, and similar treatment of artificial silk. C. LANDESKROENER (B.P. 305,980, 7.1.29. Ger., 13.2.28).

Machines for dyeing, washing, mercerising, or otherwise treating skeins of textile threads. C., G., and F. BONNET (SOC. VEUVE BONNET AINÉ ET SES FILS) (B.P. 327,399, 29.12.28).

Dye vats for dyeing. F. A. BERNHARDT A.-G., and T. KUHNEL (B.P. 327,782, 25.1.29).

Dye jigs. S. WALKER & SONS, LTD., G. E. WALKER, and J. MACE (B.P. 327,781, 25.1.29).

Means for dyeing yarn. J. LEVY (B.P. 328,099, 2.4.29).

Method and apparatus for dyeing or other liquid treatment of silks and other fabrics. C. P. COLE, SEN. (B.P. 306,462, 20.2.29. U.S., 20.2.28).

Treatment of textile yarns and threads in wound form with fluids. Treatment with air or gases of textile yarns. J. BRANDWOOD (B.P. 328,013—4, 16.1.29).

[Means for] coating of fabrics with cellulose derivatives. G. S. WALLIS (B.P. 328,431, 22.4.29).

Wetting agents (B.P. 298,559 and 326,815).—See III.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Synthesis of ammonia by the high-pressure method and the production of the necessary hydrogen. G. CLAUDES (Oesterr. Chem.-Ztg., 1930, 33, 64—65).—A review of the development of the Claude process. A. R. POWELL.

Applications of hydrogen peroxide in industry. G. ADOLPH (Z. Electrochem., 1930, 36, 146—149).—A general survey: the preparation and properties of the compound are also described. H. F. GILLBE.

Rapid differentiation of precipitated calcium carbonate and pulverised chalk or limestone. J. RANEDO (Anal. Fis. Quím., 1929, 27, [tecn.], 168—170).

—The sample (0.5 g.) is mixed with 10—15 c.c. of water and centrifuged for a few minutes. The precipitated carbonate may be distinguished from the pulverised mineral by the appearance of the supernatant liquid and by the smaller volume of the precipitate.

H. F. GILLBE.

Barium aluminates. E. MARTIN (Chim. et Ind., 1930, 23, 564—572).—Aluminium hydroxide, when calcined at a controlled rate and then hydrated afresh, differs in its properties from the original compound, as the process of calcination complicates the alumina molecule in the same way as it affects ferric oxide or silica. This behaviour offers an explanation of the complexity of barium aluminates prepared by heating mixtures of barium carbonate and aluminium hydroxide. Solutions of barium aluminates are unstable, yielding a precipitate containing a less proportion of barium than the solution. The author has in this way isolated a crystalline compound of composition $10\text{Al}_2\text{O}_3 \cdot 11\text{BaO}$. Precipitation with excess of calcium chloride has indicated the existence of a series of compounds of the formula $n\text{Al}_2\text{O}_3 \cdot (n+1)\text{BaO}$. These are simplified by hydrolysis, the final product being $\text{Al}_2\text{O}_3 \cdot 2\text{BaO}$. The author has, however, prepared at the temperature of the electric arc the compound $\text{Al}_2\text{O}_3 \cdot 3\text{BaO}$, which is not obtainable at temperatures below 1500° . Its solutions are much more stable, and it is likely to be the most satisfactory form for industrial purposes.

C. IRWIN.

Analysis of crude phosphates. A. SUCHIER (Z. angew. Chem., 1930, 43, 313—314).—Precise details are given for a method of determining the phosphoric acid in crude phosphate containing 25—38% P_2O_5 , in which the acid is precipitated with ammonium molybdate, the precipitate is taken up in ammonia, and then the acid is reprecipitated from the solution by addition of a magnesia solution. S. K. TWEEDY.

Coal and carbon as raw materials. MATIGNON.—See II. Rarer constituents of clays. FIOLETTOV.—See VIII. "Kalkammonsalpeter." NEHRING.—See XVI.

See also A., May, 552, Copper-zinc oxide catalysts (ABORN and DAVIDSON). Catalysts for synthesis of methyl alcohol (NATTA). 553, Synthesis of nitrogen dioxide (WESTHAVER and BREWER). 555, Concentration of hydrogen peroxide solutions (HURD and PUTERBAUGH). Production of hydrogen peroxide (ASKENASY and ROSE). 556, Production of barium oxide (ASKENASY and ROSE). 561, Determination of halogen in insoluble halides (KLEIN). Direct determination of bromide in presence of chloride (LONGINESCU and PIRTEA). Determination of selenium, tellurium, and gold (SOMEYA). 562, Determination of phosphoric acids (HINSBERG and LASZLO; BRESTAK and DAFERT). 564, Determination of aluminium in presence of manganese (LUKAS and JILEK). Determination and separation of rare earths (MOSER and SIEGMANN).

PATENTS.

Contact process for manufacturing sulphuric acid. H. T. MERRIAM, ASSR. to GEN. CHEM. CO. (U.S.P. 1,737,320, 26.11.29. Appl., 17.12.27).—The gases

obtained by roasting pyrites or smelting sulphide ores, after leaving the purifiers, are caused to mix with the hot gases produced by combustion of Louisiana brimstone, the amounts of the two gas streams being regulated to give the proper temperature for conversion and the required concentration of sulphur dioxide. Preheating the purified gas or disposing of the surplus heat of the brimstone gas is thus avoided, and the relatively low sulphur dioxide content of the purified gas is increased.

W. J. WRIGHT.

Manufacture of alkali hydroxides. I. G. FARBENIND. A.-G. (B.P. 303,366, 29.12.28. Ger., 31.12.27).—An alkali salt soluble in water and in liquid ammonia, *e.g.*, sodium chloride, is treated under pressure with aqueous ammonia of 50% concentration or above. Liquid ammonia may be added to an aqueous solution of the salt, or water to a solution of the salt in liquid ammonia, the alkali hydroxide being precipitated.

F. G. CLARKE.

Production of alkali carbonates and ammonia by saponification of calcium cyanamide. M. BUCHNER (U.S.P. 1,737,297, 26.11.29. Appl., 27.7.26. Ger., 21.7.24).—Calcium cyanamide is "saponified" under pressure in presence of an alkali fluoride, the calcium fluoride is removed by filtration, and the filtrate heated to liberate ammonia and yield an alkali carbonate. The calcium fluoride may be caused to react with silicon fluoride and an alkali salt in presence of an acid, thereby producing an alkali silicofluoride, which on heating forms alkali fluoride and silicon fluoride. The calcium chloride formed may be converted into calcium carbonate and ammonium chloride.

W. J. WRIGHT.

Manufacture of ammonium chloride crystals. IMPERIAL CHEM. INDUSTRIES, LTD., and C. W. BUNN (B.P. 326,642, 23.2.29).—Before a solution of ammonium chloride is crystallised, with agitation, a small proportion (0.1%) of a substance which forms mixed crystals or a double salt with ammonium chloride, *e.g.*, manganous or iron chloride, is added. Small spherical crystals are formed which pour well and do not cake.

F. G. CLARKE.

Purification of waste alkali liquors. I. G. FARBENIND. A.-G. (B.P. 314,812, 1.7.29. Ger., 2.7.28).—The liquor (from artificial silk manufacture etc.) is treated with an alkali manganate or permanganate. Manganese dioxide is precipitated and the organic impurities are oxidised to carbon dioxide which reacts with the sodium hydroxide formed to produce sodium carbonate. The latter may be separated out almost completely by cooling, and may be fused, after addition of a further quantity, with the manganese dioxide formed to produce suitable fresh manganate or permanganate; sodium hydroxide may also be employed for this purpose, but when only carbonate is used sodium hydroxide is formed in the alkali liquor, and so the process becomes continuous.

S. K. TWEEDY.

Extraction of borax [from rasorite etc.]. T. M. CRAMER (B.P. 326,116, 3.7.29. U.S., 19.12.28).—Minerals containing prismatic borax are heated above 100° and under pressure. The borax dissolves in its own water of crystallisation and the solution is separated

off from the gangue, which does not slime. Suitable apparatus is described for a continuous process.

S. K. TWEEDY.

Manufacture of sodium formate. R. KOEPP & Co., and T. BADENHAUSEN (G.P. 457,112, 17.1.25).—Carbon monoxide is led into a suspension of calcium sulphate in 10% sodium formate solution at 160–170° under pressure, and hourly or continuous additions of a suspension of lime in 20% sodium sulphate (or formate) are made, sodium formate and calcium sulphate being removed from time to time. The formate is obtained in high concentration after filtration.

C. HOLLINS.

Manufacture of alkaline-earth cyanides. I. G. FARBENIND. A.-G. (B.P. 303,115, 27.12.28. Ger., 28.12.27).—A mixture of equivalent amounts of an alkali cyanide and an alkaline-earth salt, the acid residue of which yields with the alkali used a salt soluble in liquid ammonia, is treated with liquid ammonia either below its b.p. or at a higher temperature under a pressure dependent on the relative solubilities of the individual components.

H. ROYAL-DAWSON.

Treatment of thiocyanates. C. J. HANSEN, Assee. of H. KOPPERS A.-G. (B.P. 311,725, 12.11.28. Ger., 15.5.28).—The thiocyanates, *e.g.*, ammonium thiocyanate, are decomposed by heating in aqueous solution at 220–350° under pressure in autoclaves, or by treatment at the ordinary pressure with steam superheated above 200°. The products are cooled and treated for the recovery of ammonia, or, after removal of carbon dioxide, of ammonium sulphide solution, or they may be worked up together with the spent liquors from the scrubbing operation of a thionate process, *e.g.*, that described in B.P. 309,116 (B., 1930, 546).

L. A. COLES.

Decomposition of raw phosphates. A. MESSERSCHMITT (B.P. 306,086, 6.2.29. Ger., 15.2.28. Addn. to B.P. 300,961; B., 1930, 57).—The reducing agents employed in the prior process are replaced, wholly or partly, by calcium sulphide, *e.g.*, alkali waste, or Leblanc soda or potash contaminated with calcium sulphide.

S. K. TWEEDY.

Production of dried [calcium] superphosphate. CHEM. IND. A.-G. (CHEM. IND. U. PAPIERFABR. A.-G.), and H. MEYER (B.P. 304,697, 23.1.29. Ger., 24.1.28).—Superphosphate is stored until the calcium sulphate present is hydrated (1–4 days), and is then dried at a temperature insufficient to convert the phosphoric acid into insoluble form.

F. G. CLARKE.

Reduction of gypsum. (SIR) G. C. MARKS. From G. POLYSIUS A.-G. (B.P. 326,612, 23.1.29).—In the production of sulphur oxides for the manufacture of sulphuric acid from gypsum, the charge of gypsum, aluminous material, and an excess of reducing agent is burned in the form of granules in a rotary furnace. The charge does not cake on the furnace walls.

F. G. CLARKE.

Manufacture of organo-metallic [iron] compounds. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 304,731, 24.1.29).—Oxygen carriers for oxidations below 40° are obtained by reducing hæmin with hyposulphite or cysteine, treating the resulting ferrous complex compound with bases (nicotine), and

adsorbing the product on starch, animal charcoal, silica gel, etc. C. HOLLINS.

Production of phosphoric acid and hydrogen. M. LARSSON, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,756,429, 29.4.30. Appl., 31.3.25. Swed., 15.4.24).—See F.P. 595,987; B., 1926, 320.

Catalytic oxidation of ammonia. O. BALZ and F. REUSCHER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,345, 15.4.30. Appl., 3.8.25. Ger., 22.12.24).—See G.P. 431,506; B., 1926, 915.

Manufacture of basic calcium hypochlorites. S. URANO and S. IMAI (U.S.P. 1,755,677, 22.4.30. Appl., 15.3.26).—See B.P. 266,180; B., 1927, 299.

Manufacture of coated [calcium] phosphide. F. HEBLER (U.S.P. 1,757,713, 6.5.30. Appl., 23.7.27. Ger., 14.8.25).—See B.P. 279,751; B., 1928, 14.

Manufacture of pure lead hydroxide from impure lead sulphate. R. DALOZE (U.S.P. 1,757,902, 6.5.30. Appl., 8.11.26. Fr., 12.10.26).—See F.P. 636,163; B., 1930, 324.

Liquid-air oxygen-producing plant. O. SIMONIS (U.S.P. 1,757,022, 6.5.30. Appl., 9.4.26. U.K., 21.1.26).—See B.P. 269,661; B., 1927, 482.

Device for mechanically emptying superphosphate chambers. J. ZATHEY, and TOWARZYSTWO ZAKŁADÓW CHEMICZNYCH "STREM" SPÓŁKA AKCYJNA (B.P. 328,486, 4.6.29).

Gases as solid blocks (B.P. 327,414). Freezing mixtures (B.P. 327,038).—See I. **Ammonia and hydrogen sulphide from gases (B.P. 307,903 and 309,116).**—See II. **Bricks etc. (B.P. 327,247).**—See IX. **Conductivity of solutions (B.P. 309,025).**—See XI.

VIII.—GLASS; CERAMICS.

Theory of the strength of thermally toughened glass. J. T. LITTLETON, JUN., and F. W. PRESTON (J. Soc. Glass Tech., 1929, 13, 336—349 τ).—It is shown that the optimum desirable heat treatment for an ordinary soda-lime glass rod is one which introduces 3500 lb./in.² of axial tension, the strength being thereby increased 50% with respect to plain tension. If the modulus of rupture of annealed glass were t , then, considering bending, for optimum heat treatment the central tension, f , should theoretically be $1.2t$, and the modulus of rupture, q , of the toughened glass $3.42t$. With less than optimum central tension, $q = t + 2f_0$; with more, $q^2 = 12f_0 \times (2t - f_0)$. In practice the case is much more complicated, though the results deduced were of the right order. Thus it was generally easily possible to raise the modulus of rupture to 2.5 times its value for annealed glass. Theoretical curves showing the values q/t against f_0/t and also modulus of rupture against strain at centre of specimen (measured in wave-lengths of birefringence per in.) had some experimentally determined points plotted on them which were in very fair agreement. M. PARKIN.

Strength of glass containing cracks. L. H. MILLIGAN (J. Soc. Glass Tech., 1929, 13, 351—360 τ).—The rate at which cracks in glass extended and the forces required to cause complete rupture of such glass were considerably influenced by external factors, such as presence of

water. Small glass plates with a transverse, wheel-cut scratch across the middle of one face were broken by loading the centre of the plate with the scratch facing downwards. The mean values for the breaking loads of bars treated under the following conditions, viz., (a) dry, (b) wetted, (c) wetted then dried, (d) freshly oil-treated (paraffin), (e) sodium silicate treated, were, in the same order: 960, 780, 1050, 1124 (check 1223 against 1012 dry), and 1148 g. The mechanism by which liquids thus affected the strength was not apparent, and though the effect of the sodium silicate was to cement the crack, the low rise in strength was somewhat surprising. M. PARKIN.

Analysis of glasses containing phosphate. J. D. CAUWOOD, J. H. DAVIDSON, and V. DIMBLEBY (J. Soc. Glass Tech., 1929, 13, 270—279 τ).—The method for removing phosphorus pentoxide from glass by means of silver carbonate and nitrate already described (B., 1928, 404) was found to be unsatisfactory in the case of a complex glass containing more than 5% P_2O_5 . The methods finally recommended are the following. For simple glasses with less than 6—7% ($Fe_2O_3 + Al_2O_3$) the phosphate solution is treated with 30 c.c. of ammonium nitrate solution (340 g. per litre) and 5 c.c. of concentrated nitric acid, the solution is heated just to boiling, and the required amount of hot ammonium molybdate solution added to precipitate the phosphorus pentoxide as phosphomolybdate. After settling, filtering, and washing with ammonium nitrate solution, the filtrate, after neutralising with ammonia solution and acidifying with acetic acid, is treated with 4% lead acetate solution to precipitate excess molybdate as lead salt, and, after boiling, filtering, and washing, the excess lead is removed by hydrogen sulphide. After boiling off excess gas the bases are determined as usual, aluminium and zinc being separated by the basic acetate method. For glasses in which ($Fe_2O_3 + Al_2O_3$) is above 6—7%, or for those containing also lead and barium, hydrochloric acid should be used in place of acetic acid. A rapid method for determination of calcium in presence of phosphorus is to add ammonium carbonate to a hydrochloric acid solution until slight turbidity is produced, followed by ferric chloride solution, a few drops at a time, till the liquid above the ferric phosphate is brown. The bulk is then made up to 400 c.c., the solution boiled for 1 min., filtered, etc., and calcium determined as usual in the filtrate. M. PARKIN.

Absorption of glasses in the ultra-violet region. P. GILARD and P. SWINGS (Bull. Acad. roy. Belg., 1929, [v], 15, 749—755).—Details are given of a simple method of determining the percentage transmission of ultra-violet light by glass. An examination of a number of glasses indicates that the transmission is increased by the presence of nickel oxide and that barium oxide is more effective than calcium oxide in producing transparency in the region 2700—3300 Å. F. G. TRYHORN.

Conversion of quartz of different grain-size into silica-stone in presence of various fluxes. H. SALMANG and B. WENTZ (Keram. Rundsch., 1929, 37, 711—716; Chem. Zentr., 1929, ii, 3175).—Successful products obtained by heating mixtures of sand, lime, sulphite liquor, ferric oxide, and sodium and potassium

carbonates at 1410° are attributed to the fine grain and to the presence of sodium ferrite. A. A. ELDRIDGE.

China clay. J. E. AITKEN (Proc. Tech. Sect. Paper-makers' Assoc., 1929, 10, 213—220).—A popular account of the occurrence, mining, purification, and use of China clay for papermaking. T. T. POTTS.

Rarer constituents of clays. A. FIOLETOV (Keram. Rund., 1929, 37, 659—663; Chem. Zentr., 1929, ii, 3246).—The clays examined contained 1—1.5% TiO₂, probably as rutile, and 0.1% V, probably finely divided as sulphide. Zirconium is often absent, but sometimes is present up to 0.1%. A. A. ELDRIDGE.

Treatment of certain Western [Canadian] clays to overcome drying defects. H. FRÉCHETTE and J. G. PHILLIPS (Canada Dept. Mines, Invest. Ceramics and Road Materials, 1929, No. 697, 4—16).—The serious drying defects due to the high shrinkage of a laminated plastic clay known as "gumbo," and of "Redcliff" clay, both from the Winnipeg district of Canada, may be overcome either by preheating the material to 450—550° or by the use of grog together with the addition of chemical coagulants, *e.g.*, ferric or sodium chloride. The former process would have to be preceded by a drying operation to reduce the moisture from about 33% to 5%, and thus involves considerable capital expenditure. The addition of grog or of coagulants separately was completely unsuccessful, but additions of 25—50% of grog and of up to 5% of a chemical coagulant gave satisfactory results; ferric chloride or a mixture of ferric and sodium chlorides was the most effective. C. A. KING.

See also A., May, 569, **Fireclays of southern Saskatchewan** (HUTT). **Kaolin minerals** (Ross and KERR). 570, **Kaolinite** (SETO).

PATENTS.

Manufacture of strengthened glass. H. D. MURRAY and D. A. SPENCER (B.P. 327,121, 24.12.28).—The shock-absorbing layer of a composite glass comprises layers of regenerated cellulose united by means of gelatin or a soluble gum, preferably in the presence of a mounting medium (acetic acid) comprising a solvent for the substratum and the binder. F. G. CLARKE.

Manufacture of composite materials and the like [non-splintering glass]. H. DREYFUS (B.P. 326,520, 12.10.28 and 18.7.29).—Cellulose ether and ester films are made to adhere to glass by incorporation of a synthetic resin, *e.g.*, the resin from phenol and furfuraldehyde, aniline and furfuraldehyde, formaldehyde, and *p*-toluenesulphonamide, or acaroid resin. C. HOLLINS.

Manufacture of transparent fused silica. H. GEORGE, Assr. to Soc. QUARTZ & SILICE (U.S.P. 1,755,953, 22.4.30. Appl., 9.3.28. Fr., 23.3.27).—See B.P. 287,522; B., 1928, 642.

Production of ceramic material and binding agent therefor. V. M. GOLDSCHMIDT and R. KNUDSEN, Assrs. to BORGSTAD FABRIKKER (U.S.P. 1,756,786, 29.4.30. Appl., 18.10.26. Nor., 24.10.25).—See B.P. 260,298; B., 1928, 232.

Manufacture of glazed non-vitreous pottery. J. W. MELLOR (Re-issue 17,656, 6.5.30, of U.S.P. 1,666,828, 17.4.28).—See B., 1928, 368.

Apparatus for handling and emptying glass-melting pots. N.V. MAATS. TOT BEHEER EN EXPLOIT. VAN OCTROOIEN (B.P. 317,860, 23.8.29. Ger., 25.8.28. Addn. to B.P. 212,250).

Making laminated glass [containing layers of celluloid]. L. J. KOLB, Assees. of R. A. GIBBS (B.P. 315,834, 28.6.29. U.S., 19.7.28).

Furnace linings (B.P. 327,390).—See I.

IX.—BUILDING MATERIALS.

Trass and sand as additions to cement. R. GRÜN (Chem. Erde, 1930, 5, 113—145).—Tests were made on the addition of trass and sand to Portland cement and slag-cements, the materials being either ground together or simply mixed. The products were tested after exposure to air, water, or magnesium sulphate solution for various periods of time (3 days to 24 months). It was found that the addition, by simple mixing, of up to 40% of trass increases the strength and resistance to wear of the product. L. J. SPENCER.

Sand-cement [mixtures]. A. MADINAVEITIA and A. BOOTELLA (Anal. Fis. Quím., 1929, 27, [tecn.], 164—167).—It is suggested that the peculiar properties of sand-cement mixtures are due to the colloidal nature of the finely-divided cement particles which in the wet mixture carry a positive charge while the sand particles are negatively charged; the mixture thus forms a more compact mass than is obtained by the use of cement alone, in which case all the particles are similarly charged. The hypothesis is supported by measurement of the densities of cement and of sand-cement mixtures; a contraction in the latter case, greater than that due to the quantity of cement present, is attributable to electrostatic attraction of the particles. H. F. GILLBE.

Hot cement. N. DAVEY (Cement, 1930, 3, 413—418).—Doubt has been expressed as to the quality of cement which arrives at its destination, for use, in a heated condition. Physical tests made on new cement which had a temperature of 120° showed that no reduction in the strength of concrete need be expected on account of the higher temperature at which it was mixed. C. A. KING.

Road tar. P. B. NICHOLSON (Gas J., 1930, 190, 197—198).—It is suggested that lack of uniformity in tar supplied for road dressing is responsible for poor demands for this material. R. H. GRIFFITH.

Tar roads for average and heavy traffic. MÜLLER (Gas- u. Wasserfach, 1930, 73, 279—284).—A summary of methods and costs of road construction and maintenance involving the use of tar in conjunction with various aggregates. Tar macadam manufacture receives special attention, and reference is made to the advantage of laying this material hot, whereby a far more viscous tar may be utilised; the addition of asphalt to the tar is regarded as lacking justification. The grading of the aggregate and the physical properties of the tar used in the preparation of tar concrete are of great importance, and the standard specification for this type of road construction is quoted. The preparation and application of Dammann (Essen) asphalt is described, and another

type of material claimed to give a very satisfactory surface consists of calcareous limestone previously impregnated with hot tar under vacuum. Brief reference is made to the use of tar emulsions for patching and grouting.

D. G. MURDOCH.

Asphalt emulsions. GRÄFE and FLECK.—See II.

PATENTS.

Means for measuring the permeability of sands and other aggregates. BRIT. CAST IRON RES. ASSOC., W. J. REES, and J. G. A. SKERL (B.P. 327,306, 19.4.29).—The aggregate (*e.g.*, foundry sand) is packed into a cylindrical container having apertures at both ends, the whole being then secured over an opening in a gas chamber provided with a manometer. Coal gas is introduced into the chamber at the lower end and the time taken for gas to escape through the sand in sufficient quantity to be ignited by a pilot flame is noted.

F. G. CLARKE.

Manufacture of bricks, blocks, slabs, tiles, pipes, etc. F. P. JONES (B.P. 327,247, 25.2.29).—A plastic mass is prepared from a waste lime-containing sludge, *e.g.*, alkali waste, and clay, shale, marl, ground slate, or mixtures thereof, and the articles formed therefrom are burned in a closed kiln. By using appropriate proportions and temperatures, products having different colours, porosity, hardness, etc. are obtained.

F. G. CLARKE.

Composition capable of being moulded. MICHELIN & C^{IE}. (B.P. 317,073, 11.7.29. Fr., 11.8.28. Addn. to B.P. 263,138; B., 1927, 367).—Moulded compositions made as described in the prior patent are rendered less porous by plunging them into molten sulphur.

H. ROYAL-DAWSON.

Preservation of wood. I. G. FARBENIND. A.-G. (B.P. 302,698 and 303,168, [A] 20. and [B] 27.12.28. Ger., [A] 20. and [B] 29.12.27).—Wood is impregnated (A) with a higher fatty ester, ether-ester, or mixed ester of a carbohydrate (*e.g.*, cellulose triaurate, laurylated starch), or (B) with a diazo compound, preferably stabilised as zinc chloride double salt or naphthalene-sulphonate, *e.g.*, the diazonium sulphate from 5-nitro-*o*-anisidine.

C. HOLLINS.

Improvement of asphalt and bituminous masses. K. WINKLER (B.P. 307,465, 8.3.29. Ger., 8.3.28).—Mixtures obtained by boiling drying oils with metal oxides until they thicken are incorporated with natural or artificial asphalts, pitches, etc., and the whole mixture is again boiled for a short time. About 5% of the oil mixture, containing 2–5% of metal oxide, may be used. The metal oxides may be partly replaced by sulphur or sulphur compounds. Asbestos or cellulose, and sand, lime, or other fillers, may be added also. The products are impervious to water, tough, and highly adhesive.

A. B. MANNING.

Charging apparatus for vertical kilns for burning cement, lime, and the like. E. SCHWENK and A. HAUENSCHILD (B.P. 327,679, 31.12.28).

Manufacture of faced articles of cement, such as slabs, panels, cornices, mouldings, etc. H. G. C. FAIRWEATHER. From O. L. McDERMOTT (B.P. 327,584, 16.4.29).

Building blocks. A. INGRAM (B.P. 327,766, 17.1.29).

Composite plaster boards or slabs intended particularly for building purposes. A. HARLEY (B.P. 328,326, 26.1.29).

Production of flooring and paving. C. GARTENMANN and K. RINGOLD (C. GARTENMANN & C^{IE}.) (B.P. 304,199, 12.1.29. Ger., 16.1.28).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Decomposition of carbon monoxide in the iron blast furnace. F. WÜST (Z. anorg. Chem., 1930, 188, 143–151).—The conditions under which carbon monoxide is decomposed in the blast furnace with separation of carbon are discussed on the basis of numerous analyses of gas drawn from different levels. At any particular temperature a certain degree of reduction of the ore is required for the separation of carbon, since the local concentration of carbon dioxide at the surface of the unreduced ore, due to its high rate of accumulation and slowness of diffusion, displaces the carbon monoxide-carbon dioxide equilibrium in the direction of the monoxide. The decomposition of the monoxide occurs chiefly at 600–700°, not at 500° as would be expected from laboratory experiments.

F. L. USHER.

Decomposition of blast-furnace slag. F. HARTMANN and A. LARGE (Arch. Eisenhüttenw., 1929–1930, 3, 615–625; Stahl u. Eisen, 1930, 50, 517–518).—Blast-furnace slags containing 41–54% CaO were fused, allowed to cool slowly to various temperatures, quenched, and examined for the presence of γ -dicalcium silicate which causes them to disintegrate. All slags with more than 51% CaO disintegrated immediately after quenching from below 1300°; those with 47–51% CaO disintegrated immediately when quenched from 1100–900°, according to the lime content, but only slowly when quenched from lower temperatures. Slags with less than 47% CaO were stable after quenching from below 1300°, but those with 46–47% CaO had a slight tendency to disintegrate after quenching from 1000–1100°. Increase in the manganese content reduces the maximum amount of lime present in the stable slags and also renders them brittle; alumina, on the other hand, increases the stability of the slags with a high lime content, whilst magnesia still further increases the stability and strength of the slags. Ferrous oxide, above 10%, causes the so-called “iron disintegration” to take place; these slags must be quenched from 800°. The yellow glow observed on exposing slags containing γ -dicalcium silicate to ultra-violet light is apparently due to the presence of impurities and is not a property of the γ -form of the silicate.

A. R. POWELL.

Reduction of Anshan iron ore. S. MITA (J. Ferrous Met. Anshan, Japan, 1929, 31, 1067–1102).—A study of the relation between temperature and reduction velocity, using hydrogen and dried Anshan haematite.

CHEMICAL ABSTRACTS.

Growth of grey cast iron by repeated heating. T. TERAU (Suiyokaishi, 1929, 5, 814–815).—Heating at 200° for 2 hrs. and cooling were repeated 40 times, the volume change being measured; for constant silicon

content the effect of the carbon content was small, whilst the increase was the greater the greater was the silicon content.

CHEMICAL ABSTRACTS.

Cause of graphite formation in cast iron. H. NISHIMURA (Suiyokaishi, 1929, 5, 763—769).—A discussion in which the view that graphite is formed solely by decomposition of Fe_3C is attacked. In a ternary diagram it is assumed that the primary surface of Fe_3C exists only in the range where the silicon content is small, and the primary surface of graphite exists over the range where the silicon content is large; sulphur causes supercooling and increase in the stability of Fe_3C .

CHEMICAL ABSTRACTS.

Effect of alloying elements on the iron carbide in cast iron. F. ROLL (Giesserei, 1929, 16, 933—936; Chem. Zentr., 1929, ii, 2930).—The iron carbide is the more stable the more vanadium, chromium, or manganese is present; cobalt, nickel, copper, and zinc decompose the carbide. Tungsten, uranium, and lead promote the formation of graphite; tin produces grey cast iron, and molybdenum, boron, aluminium, silicon, germanium, phosphorus, oxygen, and sulphur partly preserve and partly decompose the carbide.

A. A. ELDRIDGE.

Galvanic corrosion on cast-iron pipes. R. J. KUHN (Ind. Eng. Chem., 1930, 22, 335—341).—A study of the extensive corrosion of cast-iron service pipes laid in the soil of the New Orleans district showed that the metal was distinctly electropositive *in situ*, and that the rate of corrosion was proportional to the difference of potential of pure iron in the electromotive series, and the actual potential of the pipes in the earth. This difference is not attributed to direct leakage from current-carrying cables, but is thought to be due to the presence of oxide films acting similarly to mill-scale on steel. The most efficient cure up to the present has been a system of electrolysis drainage, by connecting electrically pipes liable to be affected to the negative system of public electric plants.

C. A. KING.

Formation of graphite during solidification of cast iron. T. KASÉ (Sci. Rep. Tôhoku, 1930, 19, 17—35).—The determination of the change of electrical resistance in the vicinity of the eutectic temperature, thermal analyses, and microscopical examination of cast iron when cooled under various conditions have been made. It is concluded that the adoption of the double diagram which gives two horizontal lines corresponding to the cementite-austenite and graphite-austenite eutectic is unreasonable.

W. E. DOWNEY.

Mechanism of nitriding of pure iron. B. TAZAWA (J. Study Ferrous Met., Japan, 1929, 106, 375—401).—When iron is heated in ammonia at 600°, 670°, or 760°, the nitrogen content of the outer layer (8.1—11.1% N) varied sinusoidally with time; the interior layer was composed of the eutectic mixture and the core of ferrite.

CHEMICAL ABSTRACTS.

Experiments with coal-fired pot-annealing furnaces. H. STÄBLER (Stahl u. Eisen, 1930, 50, 381—391).—An attempt has been made to elucidate some questions regarding annealing time, fuel consumption,

choice of pot, etc. arising from an analysis of the details of operation of annealing furnaces in a number of works. The maximum and minimum temperatures within the heating stock, in a semi-gas-fired furnace capable of accommodating five pots, have been determined as a function of the time. The annealing time, *i.e.*, the time required for the minimum temperature within the material to reach the arbitrarily chosen value of 700°, is a linear function of the weight of the charge (W), and is given by the equation $T = T_0 + KW$, where T_0 is the time required for the inner wall of the empty pot to reach 700°. The temperature difference, $t_{\text{max.}} - t_{\text{min.}}$, when $t_{\text{min.}} = 700^\circ$, increases linearly with the weight of the charge until the latter reaches about 1500 kg., when it passes through a maximum and thereafter decreases. By suitably regulating the heating conditions the characteristic temperature curves ($t_{\text{max.}}$ and $t_{\text{min.}}$ as functions of the time) can be varied to correspond with any desired heat-treatment of the material. The calculation of the efficiency of the plant from the annealing time is discussed and illustrated by one or two examples.

A. B. MANNING.

Cold-working and annealing of metals and alloys. L. GUILLET and J. COURNOT (Compt. rend., 1930, 190, 905—908).—Some of the inferences of Guichard, Clausmann, and Billon (B., 1930, 331, 337) are called in question, but without production of any fresh data. The measurement of deformation, D , as $100(S - s)/S$, where s is the sectional area before, and S after, deformation, is preferred. The non-dependence of hardness on method of deformation, and the alleged effects of annealing, are disputed. It is pointed out that hardness is not the only desideratum in metal for coins.

C. A. SILBERRAD.

Effect of various alloying elements on the critical points of carbon steels. A. MERZ (Arch. Eisenhüttenw., 1929—1930, 3, 587—596; Stahl u. Eisen, 1930, 50, 518—519).—Dilatometric investigations of nickel steels show that the temperature of the critical points falls with increasing carbon content. Chromium lowers the temperature of the A3 point of iron to a minimum with 5—7% Cr; with increasing carbon content the various critical temperatures are lowered in an irregular manner. Tungsten raises the temperature of the A3 points and silicon and nickel-chromium cause a broadening of the γ -field with increasing carbon content. Manganese has a relatively small effect on the critical temperature, only a slight fall taking place with increasing manganese content.

A. R. POWELL.

Apparatus for the determination of sulphur in iron and steel. ANON. (Chem.-Ztg., 1930, 54, 260).—The apparatus comprises a conical flask with two concentric long necks into which fits a double, glass, bell-shaped vessel carrying a thistle funnel for admitting acid to the flask and provided with a side tube at the top of the outer bell for connexion with the usual cadmium acetate absorption vessel. A water-seal in the space between the two necks serves to prevent ingress of air, and a second seal between the two walls of the bell-jar enables hydrochloric acid to be removed from the gas stream.

A. R. POWELL.

Determination of titanium in alloy steels. F. SPINDECK (Chem.-Ztg., 1930, 54, 260).—The sample (5–10 g.) is dissolved in hydrochloric acid, the ferrous chloride oxidised with nitric acid, and the solution evaporated to dryness to separate silica. The residue is extracted with 1 : 1 hydrochloric acid, the silica collected, ignited, and volatilised with hydrofluoric acid, and any residue dissolved by fusion with bisulphate. The resulting solution is added to the main filtrate after removal of the iron therefrom by extraction with ether. The solution is neutralised with sodium hydroxide, treated with 5 c.c. of 1 : 4 sulphuric acid and 80 c.c. of saturated sulphur dioxide solution, diluted to 600 c.c., and boiled for 2–3 hrs. to precipitate metatitanic acid. The precipitate is collected, washed with 15% acetic acid, ignited, and weighed as TiO_2 .

A. R. POWELL.

Volumetric determination of cobalt : application to special steels. L. MALAPRADE (Bull. Soc. chim., 1930, [iv], 423, 405–420).—The method proposed by Job (B., 1898, 4) has been modified. The cobalt salt is oxidised with precipitation of hydrated cobaltic oxide by boiling in a large excess of hydrogen peroxide in a caustic alkaline medium. The mixture is treated with sulphuric or hydrochloric acid and potassium iodide at 40–50° and the iodine liberated is titrated with sodium thiosulphate. Alternatively, the hydrated cobaltic oxide may be reduced with ferrous sulphate, excess of the latter being determined by permanganate titration. Only the latter method is applicable when determining cobalt in presence of not more than four times its weight of iron. Manganese and cobalt may be determined together by using hydrogen peroxide and potassium hydroxide as the oxidising agent and proceeding iodometrically. The caustic alkali must be replaced by sodium hydrogen carbonate when determining cobalt in presence of nickel, the amount of which latter must not very much exceed the amount of cobalt. In presence of zinc and aluminium the oxidation is effected in caustic alkaline medium. The application of the method to the analysis of cobalt-chromium steel is described.

S. K. TWEEDY.

Rapid determination of corrodibility. K. KONOPICKY (Z. Elektrochem., 1930, 36, 244–248).—The corrodibility of a sample of iron or steel by a given solution may be determined by adding to the solution ferrous and ferric ions in such proportions as to give a suitable oxidation potential, then measuring the potential of the metal in contact with the solution. Owing to reduction of ferric ions in the layer of solution in the metal surface by local currents, the measured potential is less than the calculated oxidation-reduction potential for the solution by an amount which is a measure of the corrodibility of the metal. In alkaline solutions a mixture of stannous and stannic ions may be used to give the necessary oxidation potential. Experiments made by this method on the corrosion of acid-resisting steels show that the rate of attack decreases logarithmically with the time as corrosion proceeds, and clearly reveal the effect of heat treatment and of surface defects, such as the presence of rolling-scale, on corrosion. The method is particularly suitable for detecting local defects in large pieces of metal.

R. CUTHILL.

Structure of hard brass (58% Cu). R. HINZMANN

and H. FLÖSSNER (Z. Metallk., 1930, 22, 115–118).—Extruded rods of brass with 58% Cu and 2.3% Pb usually have a needle-like structure of ($\alpha + \beta$), the α needles being finely dispersed throughout the β -needles and thus offsetting their brittleness. The structure of the end of the rods, however, shows rounded α irregularly embedded in a ground-mass of β . Heat-treatment experiments on this type of brass have proved that the needle structure is formed directly from pure β by rapid air-cooling, whereas the granular or rounded ($\alpha + \beta$) structure is obtained by slow cooling of β or by prolonged annealing at a temperature just below the $\beta \rightarrow (\alpha + \beta)$ transformation point. The latter treatment produces large α -grains free from twinning. Deformation of the alloy with a granular ($\alpha + \beta$) structure followed by annealing above the recrystallisation point produces a finer-grained, granular ($\alpha + \beta$) structure in which the α -grains are relatively small and twinning is common. Either of the first two structures may be obtained by annealing the metal above the transformation point until the α has completely disappeared; slow cooling then produces the granular, and rapid cooling the needle, structure.

A. R. POWELL.

Structure of hard brass (58% Cu) after various heat treatments. P. SIEBE and G. ELSNER (Z. Metallk., 1930, 22, 109–114).—The end portions of extruded brass rods rapidly develop a coarsely crystalline macro-structure on annealing at 800°. When the rods are heated slowly to the annealing temperature, cooled rapidly, and re-annealed, the middle portions also become coarsely crystalline irrespective of the rate of heating during the second anneal. With a rapid rate of heating in the first anneal, however, a second anneal fails to produce large crystals. The thicker the rods the smaller is the tendency to form coarse β -crystals on annealing, but the structure is always coarser than that of thinner rods. The degree of decomposition of the β -crystals after cooling slowly is always greater in the end portions of the rods than in the middle, and in thick rods than in thin; the orientation of the precipitated α varies with the heat treatment and depend on the fine structure of the β from which it has separated. In the manufacture of extruded brass rod it is recommended that the ingot used should be as short as possible so that the temperature at which the last part leaves the press is not greatly different from that at which the first part was extruded; if possible, not more than one anneal should be made.

A. R. POWELL.

Ancient bronzes. G. ZENGHELIS (Chim. et Ind., 1930, 23, 556–563).—The “patina,” or scale, on ancient Greek bronzes was in some cases deliberately produced by heating and exposure to vapours containing hydrogen sulphide. The corrosive scale formerly called “bronze disease” consists of a mixture of oxychlorides and carbonates and is of electrolytic origin. It cannot exist in bronzes beneath the sea owing to the absence of oxygen and carbon dioxide. Newly recovered bronzes should be tested by being kept for 2 days in an atmosphere saturated with water vapour. If a green scale develops, the bronze is treated by the Rozenberg method, in which chlorine is removed with a paste containing aluminium.

C. IRWIN.

Air-hardening copper-cobalt alloy. C. S. SMITH (Min. and Met., 1930, 11, 213—215).—An alloy of copper with 3.58% Co has a homogeneous α -structure at 950°, but this changes to a duplex structure at the ordinary temperature. As the initial rate of formation of nuclei is high and the growth of the particles slow, the alloy undergoes air-hardening when allowed to cool from 900—950° in the air and age-hardening when quenched and reheated at 475—550°. When quenched in ice-water the Rockwell B hardness is 14.7, which rises to 41.7 at 475° and to 54.5 at 550°. The hardness of the air-cooled alloy is 48.7, which rises to 58.6 on reheating at 475° and to 61.4 at 550°. Slower cooling in "Sil-o-cel" produces a hardness of 50, which rises to 59 on reheating at 550°. After cooling in the furnace the hardness is 22.2. The tensile strength of the hardened alloy is 51,000 lb./in.² and the elongation 20%. When the alloy is annealed in air there is formed below the usual scale of copper oxide a layer of tightly adherent, metallic "sub-scale" which appears to consist of polygonal grains of the underlying metal surrounded by films and impregnated by grains of cobalt oxide.

A. R. POWELL.

Quenching velocities [of various liquids for metals]. I. OBINATA (Mem. Ryojun Coll. Eng., 1930, 2, 315—330).—The rates of cooling of brass and cupronickel from 200—900° after plunging into water, toluene, liquid air, a mineral oil, and a vegetable oil have been determined. Water proved to be the most powerful quenching medium, whilst liquid air and toluene were very poor. In all cases a maximum rate of cooling occurred at a definite temperature, which was highest for water and lowest for toluene. This temperature was lowered and the initial cooling rate decreased by raising the temperature of the cooling medium. The initial cooling rate was increased, however, by raising the temperature of the metal before quenching, except in the case of water, where 650° gave the maximum cooling rate.

A. R. POWELL.

Rapid method of analysis of anti-friction metals. J. BEATO (Anal. Fis. Quim., 1929, 27, [teen.], 171—190).—The method of Oesterheld and Honegger (A., 1919, ii, 478) yields results for tin which are too low. The following method is recommended. The alloy (1 g.) is dissolved in 20 c.c. of boiling concentrated sulphuric acid and, after cooling and diluting with 100 c.c. of water, 5—15 c.c. of concentrated hydrochloric acid are added and the antimony is titrated in the hot solution with 0.1N-potassium bromate, using methyl-orange as indicator. Lead is next determined directly by weighing the precipitated lead sulphate, which may be contaminated with sulphur if there is much tin in the alloy. To the filtrate are added 50 c.c. of concentrated hydrochloric acid and 1.5 g. of aluminium in three equal portions, and the solution, which should be contained in a flask fitted with a Bunsen valve, is boiled until dissolution of the tin which first separates is complete; after cooling, 2 g. of potassium bicarbonate and 5 c.c. of 2N-potassium iodide are added, and the solution is titrated with 0.1N-potassium bromate, using starch as indicator. Iron in moderate quantity does not influence the determination, whereas copper, and to a less extent antimony when present in con-

siderable quantity, tends to produce low results for the tin, but this error may be reduced by prolonging the boiling during the reduction with aluminium.

H. F. GILLBE.

"Improvement" or "age-hardening" of alloys. W. GUERTLER (Z. Metallk., 1930, 22, 78—84).—The exact meaning of the terms "improvement" or "age-hardening" ("Vergütung" or "Veredelung") in connexion with the heat-treatment of alloys is discussed. It is suggested that these terms should be applied to that heat-treatment which causes an improvement of the mechanical properties of an alloy due to changes in its internal heterogeneous state unaccompanied by change of composition or by visible change in the structure and texture of the alloy. Hence, age-hardening alloys must necessarily be capable of existing in different states of equilibrium with variations in the temperature, the reactions which take place in the solid state must be reversible, and it must be possible to restrain these reactions from going to completion by suitable heat-treatment and to cause them to proceed by another heat-treatment. The three principal types of age-hardening alloys are those in which (a) a compound is precipitated from solid solution, (b) the solubility of one constituent in the other increases with rise of temperature, and (c) a crystal phase is formed from or decomposed into two crystal phases during cooling. The characteristics of these types of alloys are discussed from the theoretical point of view. A. R. POWELL.

Age-hardening aluminium alloys. W. FRAENKEL (Z. Metallk., 1930, 22, 84—89).—The mechanism of the age-hardening process in copper-aluminium alloys with and without small quantities of other constituents is discussed in the light of recent research on these alloys, and the author concludes that no satisfactory explanation has been advanced to account for all the observed phenomena, especially for the fact that, in many cases, the hardness increases during ageing at the ordinary temperature and then decreases sharply before commencing to increase again on subsequent ageing at high temperatures.

A. R. POWELL.

Age-hardening heavy-metal alloys. G. MASING (Z. Metallk., 1930, 22, 90—94).—A review of recent work on the development of age-hardening alloys of copper (Heusler alloys, Corson alloys, and beryllium-copper alloys) and of iron, with a brief description of the properties of the alloys and the effect of heat-treatment thereon. The mechanism of the hardening process is discussed.

A. R. POWELL.

Age-hardening precious-metal alloys. L. NOWACK (Z. Metallk., 1930, 22, 94—103).—Recent work on alloys containing silver, gold, and platinum which are subject to age-hardening is reviewed, and a brief account given of various new systems in which the phenomena occurs. Alloys of gold with 20—25% Pt exhibit age-hardening after quenching from 1000° and annealing at 550°; addition of a small amount of zinc increases the hardening effect appreciably. Alloys of gold with 10% Pt or Pd and 1.5—3% Zn harden rapidly at 550° to a maximum hardness of 170. Iron-gold alloys with 15—20% Fe harden rapidly at 400°, the original hardness of the quenched alloy (100) increasing to 280 in 1 hr. Nickel-

gold alloys with more than 10% Ni harden, due to the slow decomposition of the solid solution, and copper-gold alloys with equimolecular proportions of the elements become almost twice as hard after annealing at 200°, owing to the transformation of the solid solution into the compound CuAu; corresponding with this change the d increases and the electrical resistance falls. Palladium-copper and platinum-copper alloys behave similarly. A. R. POWELL.

Flowability of aluminium and alpac under constant pressure. A. COURTY (Compt. rend., 1930, 190, 936–938).—By the method previously described (cf. Saito and Hayashi, B., 1920, 159 A; Guillet and Portevin, B., 1926, 983), with addition of a device whereby the metal enters the mould under uniform pressure, thereby ensuring much more uniform results, the effect on the flowability of aluminium (99.8%) and of alpac (Al 87%, Si 13%) of (i) temperature of pouring (575–890°), (ii) temperature of the mould (15–560°), and (iii) superheating and the period of heating before pouring has been examined. It is shown that as regards (i) flowability increases in approximately direct ratio with increase of temperature of pouring; (ii) has little effect below 200°, above which flowability is markedly increased; (iii) has little or no effect. Under similar conditions (save at the lowest temperatures, when the difference is greater) the flowability of alpac is about 30% greater than that of aluminium. C. A. SILBERRAD.

Experience with aluminium alloys in sea-water. H. BAUERMEISTER (Z. Metallk., 1930, 22, 119–128).—The behaviour of machinery parts made of cast and worked aluminium alloys in Baltic and North Sea water has been examined. None of the commercial cast alloys behaved very badly when not in contact with other metals, but all rapidly corroded in contact with brass, copper, or iron except “KS-sea-water” alloy containing 3% Mg, 3% Mn, 0.93% Sb, 0.56% Fe, 0.26% Si, and 92.3% Al. This alloy has a tensile strength of 18 kg./mm.², a yield point of 9.5 kg./mm.², an elongation of 3–8%, and a Brinell hardness of 60; tensile strength and elongation remain unchanged after prolonged immersion in sea-water, whereas a slow reduction occurs in these values for lual and duralumin and a much more rapid reduction with silumin. Corrosion of aged alloys usually commences around rivet holes and at places where the metal has been bent, but the KS-alloy can be riveted or welded without deteriorating its resistance to corrosion. Tests with artificially produced coatings of oxide showed that these serve only to prolong the life of the metal in sea-water and do not entirely prevent corrosion. A. R. POWELL.

Texture of cold-deformed metals. F. WEVER and W. E. SCHMID (Z. Metallk., 22, 133–140).—The changes in the crystal texture of iron and aluminium after a parallelepiped plane deformation have been followed by X-ray examination and the pole figures on the crystals determined at the maximum deformation (48% and 73%, respectively) obtained. The mechanism of deformation is then explained on the basis of known laws of plastic deformation of cubic, face-centred, single crystals. The results obtained are in agreement with the assumption that deformation takes place in the

(111) direction along the (011) plane as a slip plane. As the deformation to which the metals were subjected is very similar to that undergone during ordinary rolling operations, it follows that the results are directly applicable to the study of the structure of rolled metals.

A. R. POWELL.

Analysis of the platinum metals. W. GRAULICH (Oesterr. Chem.-Ztg., 1930, 33, 2–4).—A scheme for the analysis of commercial platinum for palladium, gold, iridium, rhodium, and platinum is outlined, and details are given of a method for the analysis of rhodium sponge for impurities based on the insolubility of anhydrous rhodium trichloride in dilute aqua regia and the solubility of palladium, platinum, and gold in that solvent.

A. R. POWELL.

Stress-strain relation in the impact test. T. SUTOKI (Sci. Rep. Tôhoku, 1930, 19, 1–15).—Stress-strain diagrams at high temperatures have been obtained with a Charpy machine, using the method of Körber and Storp. Carbon steels (0.3 and 0.5% C) in the form of Izod test-pieces were used. The highest temperatures at which perfect breaks were obtained were 600° and 650°, respectively. A piece of brittle material breaks abruptly at a maximum stress, whilst a tough test-piece fails in several steps. The bending of a test-piece increases at first almost linearly with rise of temperature, but from about 300° it begins to increase, and at 550° falls almost to the value at ordinary temperatures, and then rapidly increases. Honda's view that thermal brittleness is the combined effect of work-hardening and of the softening due to temperature is confirmed.

W. E. DOWNEY.

Effect of dissolved substances on gravity concentration [of ores]. F. J. TROMP and E. BEYERS (J. Chem. Met. Min. Soc. S. Afr., 1930, 30, 226–232).—Experiments on the settling and gravity concentration of a clayey tin ore tailing after treatment with dilute acids and alkalis are recorded. Sulphuric acid retarded settling of the heavier constituents and gave a low recovery on passing the acidulated pulp along an inclined rubber-lined trough, probably owing to its flocculating action on the clay present. Lime, sodium hydroxide, and, especially, sodium carbonate deflocculated the clay and allowed the heavier tinstone to settle out freely, a recovery of over 70% of the tin being obtained from a pulp containing sodium carbonate. Settling tests on this pulp showed a large increase in the rate at which the non-clayey particles separated.

A. R. POWELL.

Addition agents in electrodeposition. III. Application of the complex cation theory to baser metals. G. FUSEYA, K. MURATA, and R. YUMOTO (Tech. Rep. Tôhoku, 1930, 9, 33–56; cf. B., 1927, 632).—Complex cation formation was detected in solutions of lead fluosilicate and nitrate, ferric sulphate, chromic sulphate, copper sulphate, zinc sulphate, and possibly of nickel and cobalt salts, all containing glycine, by spectrographic, E.M.F., and migration experiments. In the case of zinc and lead the degree of complex cation formation is exceedingly high. Electrodeposition from zinc sulphate, lead nitrate, and lead fluosilicate solutions containing glycine was investigated. The glycine

does not enter the deposit, nor does it diminish the crystal size of the latter, contrary to the case when silver and copper are deposited (cf. *loc. cit.*). Apparently, during the electrolysis of a salt of a metal more electropositive than hydrogen in the electrochemical series, the solution becomes deficient in hydrogen ions in the vicinity of the cathode. The complex cation consequently loses its charge in this region and becomes incapable of electrodeposition.

S. K. TWEEDY.

Electrolytic iron from sulphide ores. T. D. YEN-SEN (Min. and Met., 1930, 11, 212—213).—Electrolytic iron produced from sulphide ores contains 0.005% C, 0.007% S, and probably up to 0.01% O. The removal of this oxygen can be effected only by melting *in vacuo* with a powerful deoxidising agent. The chief use of electrolytic iron will therefore probably be for the production of silicon-iron and cobalt-iron alloys, for which, however, it does not appear to have any advantage over Armco iron. With power at 0.3 cent/kw.-hr. and scrap iron at \$15 per ton, electrolytic iron could be produced at \$80—85 per ton with a daily output of 25 tons.

A. R. POWELL.

Effect of superposing alternating currents on the electrolytic oxidation of aluminium. S. SETOH and A. MIYATA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1930, 12, 268—274; cf. B., 1930, 16).—By superposing a suitable amount of alternating current in the electrolytic oxidation of an aluminium surface the electric energy required to obtain a certain thickness of oxide film can be reduced, and the film is more resistant to corrosion by hydrochloric acid than that obtained with direct current only. Apparatus for investigating these effects is described. If the electrolyte contains a reducing agent such as oxalic acid the film is rendered waterproof by a short steam-treatment, the length of which varies inversely with the concentration of the reducing agent. Steam-treatment applied to films first prepared in other electrolytes and then soaked in oxalic acid increases their chemical resistance. Best results are obtained when using a continuous voltage of 60 volts and an alternating current of 1.5 amp.

H. I. DOWNES.

"Stopping-off" materials for use in the electro-deposition of nickel. D. J. MACNAUGHTON and A. W. HOTHERSALL (Trans. Faraday Soc., 1930, 26, 163—172).—Stopping-off materials are necessary in electro-deposition to enable the deposit to be confined to certain areas, and a number of materials have been examined as to their suitability for use in nickel-plating. Oil varnishes and varnishes made by dissolution or suspension of india-rubber, gutta-percha, ebonite, bitumen, or shellac were unsatisfactory owing to pinholing or poor adhesion. The best material is a wax mixture containing 75% of paraffin wax and 28% of gutta-percha. By coating the areas to be plated with a paste of chalk and water the wax is prevented from adhering to these parts. The disadvantage of this mixture lies in its contaminating effect on the bath, which after several months gives brittle deposits. This is overcome by oxidising any organic materials in the bath by boiling with potassium permanganate (1—2 lb./

1000 gals.) followed by removal of any manganese in solution.

C. J. SMITHELLS.

Modern electrolytic methods for the protection of metals from corrosion. W. BIRETT (Z. angew. Chem., 1930, 43, 274—277).—A review of the preparation, properties, and resistance to corrosion of electrolytic deposits of chromium, nickel-chromium, and cadmium.

A. R. POWELL.

Pulverised-fuel-fired furnaces. HOLLANDS and LOWNDES.—See I.

See also A., May, 530, **Melting of rhodium** (SWAN-GER). **Recrystallisation of metals** (TAMMANN and CRONE; KARNOP and SACHS). 536, **Diffusion of zinc in copper crystals** (ELAM). **System silver-aluminium-zinc** (UENO). **Iron-vanadium alloys** (OSAWA and ŌYA). 537, **Composition of eutectics** (STOCKDALE). 546, **Deposition of zinc and cadmium from ammoniacal solutions** (DOBRYSZYCKI). 552, **Platinum-black catalysts** (TAYLOR and others). 565, **Determination of tungsten in presence of vanadium** (JÍLEK and LUKAS). 566, **Apparatus for making single-crystal wire** (SUNAGA). 567, **Continuous automatic purification of mercury** (CORBIÈRE).

PATENTS.

Smelting of ferro-alloys in blast furnaces. F. W. DAVIS, Assr. to S. G. ALLEN (U.S.P. 1,744,213, 21.1.30. Appl., 2.6.24).—The furnace is blown with a blast containing a higher proportion of oxygen than air and the fuel is supplied in powdered or liquid form at the tuyère zone. The falling charge in the shaft is preheated by admitting quantities of oxygen-enrichened air at various points to effect combustion of the carbon dioxide produced in the tuyère zone.

A. R. POWELL.

Carburising process [for case-hardening iron and steel]. A. W. MACHLET (U.S.P. 1,745,104, 28.1.30. Appl., 6.5.27).—The articles are heated in a chamber in an atmosphere consisting of 1 vol. of propane and 3 vols. of air under pressure.

A. R. POWELL.

Cementation and hardening of iron, iron alloys, and steel. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 304,209 and Addn. B.P. 308,963, [A] 15.1.29, [B] 4.2.29. Austr., [A] 16.1.28, and Ger., [B] 2.4.28).—(A) Metals are case-hardened by the action of finely-divided carbon suspended in molten sodium chloride or in a low-melting mixture of salts, e.g., sodium and potassium chlorides, at above 850°, preferably at 900—950°. (B) Caustic alkalis or alkaline carbonates may be added to the bath either separately or together.

C. A. KING.

Cementation and hardening of iron, iron alloys, and steel. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 310,837, 4.2.29. Ger., 1.5.28. Addn. to B.P. 304,209; preceding abstract).—The articles are case-hardened in a bath of fused alkali chloride to which is added 4—12% of a fused or granulated mass made by stirring 250—300 g. of powdered wood charcoal with 1 kg. of fused sodium hydroxide or carbonate.

A. R. POWELL.

Blackening, by oxidation, of iron and steel, and articles thereof. L. TARICCO (B.P. 327,615, 14.5.29).

—The iron or steel articles are dipped at 125° into a concentrated solution of potassium hydroxide containing potassium cyanate and prepared by the addition of 2.12 g. of potassium cyanide to a hot solution of 22.21 g. of litharge in a solution of 35–85 g. of potassium hydroxide in 39.82 g. of water. During use the cyanate becomes reduced to cyanide and may be regenerated by boiling the bath with litharge. A. R. POWELL.

Coating of metal articles [iron pipes] with lead. S. OTIS and W. T. HERREN, Assrs. to NAT. BOILER WASHING Co. of ILLINOIS (U.S.P. 1,745,185, 28.1.30. Appl., 31.5.24).—The pipes are thoroughly cleaned, coated with lead by chemical or electrochemical deposition from a solution of lead salt, dipped in a concentrated acid zinc chloride solution and then into molten lead, and finally quenched in oil floating on water. A. R. POWELL.

Treatment of [aluminium-iron] alloys. N. B. PILLING, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,744,242, 21.1.30. Appl., 23.8.26).—Aluminium-iron alloys with 4–10% Al which are relatively brittle at the ordinary temperature may be rolled, stamped, or forged at 30–200° according to the aluminium content. A. R. POWELL.

Treatment of [zinc] ores. A. FOLLIET and N. SAINDERICHIN (B.P. 312,667, 28.5.29. Fr., 30.5.28).—Metals, e.g., zinc, are volatilised by the action of a current of air at 650–800° on the surface of a mixture of powdered ore, e.g., calamine, and coal to which is added a trace of an alkali chloride. The process is conducted conveniently in a rotary furnace, the hot air being directed on to the charge near the outlet end after the distillation of the fuel has been completed. C. A. KING.

Manufacture of carbonised metallic wire or ribbon. WESTINGHOUSE ELECTRIC & MANUF. Co., Assees. of C. B. UPP and L. SUTHERLIN (B.P. 305,467, 28.1.29. U.S., 4.2.28).—The wire or ribbon is passed through a tube furnace through which a current of hydrocarbon gas is passed and in which the wire is heated electrically to the carburising temperature. For example, nickel wire for the manufacture of grids for radio valves is carburised at 800° in an atmosphere of acetylene. A. R. POWELL.

Casting of magnesium or magnesium alloys. I. G. FARBENIND. A.-G., and A. L. MOND (B.P. 326,820, 19.12.28. Addn. to B.P. 187,943; B., 1923, 1137 A).—The moulding sand used is previously mixed with ammonium fluosilicate and stored for some time or it may be mixed with ammonium fluoride or ammonium hydrogen fluoride and oxalic acid just prior to use. Castings made in moulds prepared from sand so treated have a corrosion-resistant coating of magnesium fluoride and are free from oxide inclusions. (Cf. B.P. 219,753; B., 1924, 794.) A. R. POWELL.

Production of dark oxidic coatings on magnesium and its alloys. W. W. TRIGGS. From SPRENGER CORP. M.B.H. (B.P. 326,693, 23.4.29).—Articles made of these metals are immersed in a neutral bath containing one or more soluble salts of bivalent manganese and sodium chromate or dichromate, and gently heated. H. ROYAL-DAWSON.

Soldering process [for galvanised articles] J. H. NEAD, E. R. WEHR, and C. C. MAHLIE, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,743,615, 14.1.30. Appl., 25.2.27).—To solder galvanised articles, made by dipping in a zinc bath containing more than 0.5% Al, the surface is cleaned with 20% sodium hydroxide solution, washed, and soldered in the usual way with a zinc chloride flux. A. R. POWELL.

Internal cooling of metal vessels containing acid liquids. BERNDORFER METALLWARENFABR. A. KRUPP A.-G. (Austr.P. 109,381, 14.5.25).—To diminish or completely to prevent electrical potential differences between the cooling coils and the walls of the container, the cooling tubes are coated with materials the electrical potential of which approximates as nearly as possible to that of the metal of the container. Enamels, metals, and metallic oxides are the most suitable for coatings. C. RANKEN.

Sintering apparatus [for ores]. A. HOLMBERG (U.S.P. 1,757,863, 6.5.30. Appl., 28.1.28).—See B.P. 310,089; B., 1929, 522.

Furnace for treating zinc ores and other zinciferous material. A. ROITZHEIM and W. REMY (U.S.P. 1,755,076, 15.4.30. Appl., 23.4.26. Ger., 13.11.25).—See B.P. 261,344; B., 1927, 658.

Metallurgy of metals. [Refining of copper.] H. H. ALEXANDER (U.S.P. 1,756,967, 6.5.30. Appl., 21.7.26).—See B.P. 311,812; B., 1929, 562.

Manufacture of heat-treated copper-nickel-aluminium alloys. W. A. MUDGE, Assr. to INTERNAT. NICKEL Co., INC. (U.S.P. 1,755,554—7, 22.4.30. Appl., [A] 10.6.24, [B] 31.3.25, [C, D] 13.7.25).—See B.P. 250,194; B., 1927, 302.

Refining of nickel-copper matte. F. E. LATHE (U.S.P. 1,756,092, 29.4.30. Appl., 13.12.28. Can., 27.12.27).—See B.P. 303,066; B., 1930, 64.

Protecting easily oxidisable metals such as those having a base of magnesium, aluminium, calcium, etc. G. MICHEL (U.S.P. 1,754,481, 15.4.30. Appl., 8.3.28. Fr., 12.3.27).—See B.P. 287,046; B., 1929, 562.

Pickling of metals. V. BERTLEFF (U.S.P. 1,757,829, 6.5.30. Appl., 27.6.28. Austr., 9.7.27).—See B.P. 293,701; B., 1929, 176.

Electrolytic refining of copper or copper alloys. M. HOSENFELD and G. HÄNSEL, Assrs. to SIEMENS & HALSKE A.-G. (U.S.P. 1,757,047, 6.5.30. Appl., 24.7.26. Ger., 6.1.26).—See B.P. 264,116; B., 1927, 633.

Permeability of [foundry] sand (B.P. 327,306).—See IX. Electric furnaces (B.P. 306,969 and 326,517).—
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— XI.

XL—ELECTROTECHNICS.

Copper oxide rectifiers. H. PÉLABON (Compt. rend., 1930, 190, 630–632).—Copper oxide rectifiers are obtained by heating copper discs in air, just below the m.p. of copper, and removing the black cupric oxide till a surface blood-red in colour appears. Microscopical examination revealed (1) a grey powdery, semi-conducting layer of cupric oxide produced during cooling in air; (2) a well-defined, pale green, heterogeneous phase

composed of the mixture ($2\text{CuO} + \text{Cu}_2\text{O}$), the lower layers being the richer in cupric oxide; (3) a sharp, homogeneous, green layer of pure cupric oxide; (4) a granular, highly-conducting, eutectic layer of copper containing 3.5% Cu_2O . The seat of the rectifying properties is in the condenser effect due to the action as an insulator of the cuprous oxide in the second layer. Strong currents pass most easily from the bad to the good conductor, i.e., from the oxide to the copper.

J. GRANT.

Electrolysis of water under pressure. H. CASSEL and F. TÖDT (Z. Elektrochem., 1930, 36, 241—242; cf. Schnurmann, B., 1929, 945).—It is suggested that the apparent decrease in resistance of a cell for the electrolysis of water when pressure is applied is due actually to an increase in the residual current, primarily as a result of the increased solubility of the oxygen increasing the depolarisation of the cathode.

R. CUTHILL.

Breakdown of liquid insulators. F. KOPPELMANN (Naturwiss., 1930, 18, 333).—Experiments were made with pure paraffin oil and with hexane, using point discharges and also discharges from blunt surfaces. For oil which has not previously had the air removed there is an evolution of gas at the electrodes at potentials much below that necessary for breakdown. The bubbles were regular and came from isolated spots on the electrodes. After some time the evolution of gas ceased. It seems probable that, under the influence of the potential at the electrodes, charge-bearing layers are formed and as the potential is increased they are propagated through the dielectric.

A. J. MEE.

Addition agents in electrodeposition. FUSEYA and others. **Electrolytic iron.** YENSEN. **Electrolytic oxidation of aluminium.** SETOH and MIYATA. **Electrodeposition of nickel.** MACNAUGHTON and HOTHERSALL. **Protecting metals from corrosion.** BIRETT.—See X. **White lead.** OKUNO.—See XIII. **pH of soils.** DE CONNICK; SMOLIK.—See XVI. **Determination of silver in photographic layers.** LÜHR.—See XXI.

See also A., May, 544, **Measurement of resistance of electrolytes** (SCARPA). 546, **Polarographic studies with the dropping mercury cathode** (DOBRYSZYCKI). 553, **Preparation of salicylaldehyde** (KAWADA and YOSIDA). **Synthesis of nitrogen dioxide** (WESTHAVER and BREWER). 562, **Potentiometric determination of barium, lead, and sulphate** (MUKAI). 565, **Comparison of hydrogen-ion indicator electrodes in presence of ferric iron** (ELDER). 580, **Reduction of aldehydes** (SHIMA).

PATENTS.

Electric furnace. P. L. J. MIGUET and M. P. PERRON (B.P. 313,121, 10.5.29. Fr., 7.6.28).—Current is supplied through a casing in electrical contact with an electrode suspended above the hearth, and through an inner cylinder insulated from the casing and connected with electrodes embedded in the furnace hearth.

J. S. G. THOMAS.

Electric furnaces [heated by direct current]. T. D. KELLY and G. E. LEAVEY (B.P. 326,321, 22.2.29).—Furnace electrodes are connected with the terminals of a

differentially wound generator, giving a decreased voltage immediately a large short-circuit occurs. [Stat. ref.]

J. S. G. THOMAS.

[Two-deck] electric furnace. A. SADLER. From G. B. SHIPLEY and H. ALINDER (B.P. 326,517, 15.9.28).—A treatment tunnel having bottom charging and discharging apertures at its opposite ends is superimposed on a handling deck, and elevators operating between the two decks are arranged at the charging and discharging ends, one for raising work-carriers into the charging end of the tunnel and the other for lowering work-carriers from the discharging end. Means are provided for moving a train of work-carriers in the tunnel towards the discharging end when both elevators are raised, and for moving another train of carriers from the discharging to the charging end when both elevators are lowered. Electric-resistance heating elements are arranged in both sides of the tunnel and extend substantially from the top to the bottom thereof. A stream of reducing or deoxidising gas can be introduced into the treatment zone.

J. S. G. THOMAS.

Electric annealing or like furnace. P. MENE (B.P. 306,969, 28.2.29. Fr., 29.2.28).—The heating chamber comprises vertical heating resistances arranged on the lateral walls of the chamber and horizontal heating resistances arranged beneath the top face of the hearth. The vertical heating resistances are formed by extensions of the horizontal resistances, and the whole heating system is suspended from the side walls of the heating chamber.

J. S. G. THOMAS.

[Filaments for] electron-discharge tubes or thermionic valves. MULLARD RADIO VALVE CO., LTD., and B. KROL (B.P. 326,334, 28.2.29).—Fine, parallel, tungsten filaments are bound together with fine tungsten wire, and the whole is coated with the oxide of an alkaline-earth metal.

J. S. G. THOMAS.

[Getter for] incandescence electric lamps. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. FORCE (B.P. 313,010, 4.6.29. U.S., 4.6.28).—Refractory metal powder having an affinity for oxygen, e.g., tungsten or tantalum, is placed in the lamp near or at the junction of the lamp filament and leading-in conductors.

J. S. G. THOMAS.

Manufacture of Wehnelt cathodes for electric-discharge devices. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 309,581, 25.3.29. Holl., 13.4.28).—A core is coated with an alkaline-earth manganate or chromate, e.g., the barium salt, and heated, preferably at about 1200°.

J. S. G. THOMAS.

Manufacture of piezo-electric, pyro-electric, and the like dielectrics. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 302,726, 14.12.28. Ger., 21.12.27).—Finely-divided material, e.g., quartz, together with, if desired, a binding material, e.g., paraffin wax, is subjected to the action of an electric field, and preferably is heated and/or subjected to high pressure.

J. S. G. THOMAS.

Manufacture of electric insulations, high-tension storage batteries, and condensers. A. JOFFE (B.P. 302,686, 15.12.28. Ger., 20.12.27).—Two substances of different electrical conductivity, e.g., phenol-formaldehyde condensation products and cellulose acetate,

are dispersed in one another by emulsification, so that small particles of the material of higher conductivity are separated by thin walls, e.g., up to 0.005 mm. thick, of the material of lower conductivity.

J. S. G. THOMAS.

Treating [impregnating] electrically-insulating material. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF F. W. HARRISON (B.P. 297,035, 11.9.28. U.S., 12.9.27).—Electrically-insulating material, e.g., dry cellulose pulp, cotton fibre, is compressed and immersed in oil, e.g., a mineral oil of the paraffin series, at 150–200° until evolution of bubbles ceases. J. S. G. THOMAS.

Devices for measuring the electrical conductivity and concentration of acid or salt solutions. ATELIERS J. CARPENTIER (B.P. 309,025, 8.3.29. Fr., 3.4.28).—The coils of a logometer, described in B.P. 5961 of 1911, are introduced into a Wheatstone bridge, one arm of which comprises a variable resistance in series with the solution the resistance of which is to be determined, in such manner that according to the conductivity of that solution the coils of the logometer turn through angles which can be calibrated to give directly the conductivity or concentration of the solution. Any change of conductivity of the solution due to change of temperature is compensated.

J. S. G. THOMAS.

[Assembly of parts in Leclanché-type] electric batteries. F. S. S. WATES (B.P. 327,760, 9.4.29).

Manufacture [encasing the agglomerates] of dry batteries [in braid]. SOC. ANON. ELECTROCHIM. PHÆBUS, and S. DE PLOËG (B.P. 326,704, 8.5.29).

Electric accumulator. H. ELSNER (B.P. 307,927, 13.3.29. Ger., 17.3.28).

Production of galvanic elements or batteries. SIEMENS & HALSKE A.-G., ASSEES. OF K. SCHENKEL (B.P. 301,934, 8.12.28. Ger., 9.12.27).

Incandescence electric lamps [with internal reflectors]. G. MAIN (B.P. 310,929, 29.4.29. Fr., 3.5.28).

Manufacture [sealing-off] of [leaky] electric incandescence lamps or the like. GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 317,723, 13.8.29. Addn. to B.P. 242,303).

Electric-discharge tubes [for purposes of illumination]. A. E. CHAPMAN and J. VIELLE (B.P. 326,771, 18.12.28).

Fuel-burning system (B.P. 314,757).—See I. Carbon black (B.P. 326,913).—See II. Electro-paint (U.S.P. 1,744,469). Metal coat for condenser plates (U.S.P. 1,744,281).—See XIII.

XII.—FATS; OILS; WAXES.

Recovery of vegetable oils and fats by a bacterial process. J. W. BECKMAN (Ind. Eng. Chem., 1930, 22, 117–118).—Crushed copra is mixed with ground limestone and sufficient water to form a mush and is incubated at 50°, with exclusion of air, with a culture of *B. Delbrücki*, obtained from brewer's malt. The bacteria convert the sugars into lactic acid, with simultaneous

production of an enzyme which converts the protein material into soluble amino-acids: the mixture gradually becomes more liquid, and after 6 days the oil can be recovered from the residue by filtration. The high temperature of reaction inhibits the growth of moulds etc., and only a small increase (from 10.6 to 13%) of free fatty acids occurs. The quality of the oil so produced is superior to the average, and its m.p. is normal. The residue, containing calcium lactate, amino-acids, and bacteria, is dried and constitutes a good cattle food. The cost of the operation is stated to be considerably cheaper than that of the standard pressing process. E. LEWKOWITSCH.

Determination of unsaturation of fats and fatty acids. I. J. VAN LOON (Chem. Umschau, 1930, 37, 85–87; cf. Inaug. Diss., Delft, 1929).—The terms "true," "apparent," and "partial" iodine values are defined and discussed. The author considers that iodine values should be determined on the fatty acids of an oil and not on the crude oil. Experiments (details to be published later) showed that, except in a few special cases, the Wijs method with prolonged reaction time gives constant and true iodine values.

E. LEWKOWITSCH.

Sub-iodine value of linseed oil. F. FRITZ (Chem.-Ztg., 1930, 54, 213).—Attempts were made to determine a characteristic sub-iodine value ("Teiljodzahl") for linseed oil by various methods, including direct titration of the oil dissolved in carbon tetrachloride with a pale Wijs iodine solution: values about 117 were obtained, but great difficulty was found in determining the endpoint. E. LEWKOWITSCH.

Bellier test for sesamé oil. E. F. COOK (J. Amer. Pharm. Assoc., 1930, 19, 361–366).—The Bellier test for sesamé oil is uncertain and the following modification is described. Two drops of the oil are added to 1 c.c. of colourless nitric acid (d 1.4) and, after shaking, a small crystal of resorcinol is added. On agitating, a green colour develops in the oil and extends into the acid. On the addition of either benzene or chloroform practically all the colour remains in the acid, but fades within 3–5 min. This test gave a positive reaction with all the sesamé oils examined and detected 1 pt. of the oil in 250 pts. of another fixed oil. Repeated extraction of sesamé oil with glacial acetic acid removed the material responsible for the green colour in the Bellier test. The material extracted was separated into a resinous mass, which gave a deep green colour, and sesamin, which gave a negative reaction. E. H. SHARPLES.

Composition of porpoise-jaw oil. A. H. GILL and C. M. TUCKER (Oil & Fat Ind., 1930, 7, 101–102).—The oil from the head of a porpoise, *Tursiops truncatus* (caught off Hatteras) had: d_{20}^{25} 0.9241, n_D^{20} 1.4519, saponif. value 293, iodine value (Hanus) 28.3, Reichert-Meissl value 139, acid value 2.88, cold test -19° , viscosity (Saybolt) 101 sec./100° F., α $+0.3^\circ$ (1-dm. tube). The unsaponifiable matter (solid at 0°) dissolved completely in acetic anhydride and appeared to consist principally of dodecyl alcohol, with small amounts of a substance, m.p. 61° (tetradecyl alcohol?). The approximate analysis of the oil is given as: dodecyl alcohol 18.7, glycerol 18.5, isovaleric acid

62.6, palmitic acid 6.1, oleic acid 3.5%. The presence of mono- or di-valerin is suspected. E. LEWKOWITSCH.

Vegetable oils of the Union of S.S.R. V. Nature of fatty oils of *Capparidaceæ* in connexion with the climate of district of origin. S. L. IVANOV and J. S. ELAKOV (Chem. Umschau, 1930, 37, 83—84; cf. B., 1930, 154).—Seeds of *Capparis herbacea*, Willd., from Turkestan and from Crimea (rather milder climate), which represent the most northerly limits of distribution of the *Capparidaceæ*, yielded 28—30% of pale red oils having respectively: d_{25}^{25} 0.9144, 0.9102; butyrorefractometer at 25° —, 70.5; acid value 37.14 (rainy season), 7.74; saponif. value 200.0, 190.51; iodine value 110, 104.6—105.4; Reichert-Meissl value —, 1.1; hexabromides nil, nil; unsaturated acids —, 98.8% (Varretrapp), or —, 84% (Bertram); saturated acids —, traces (Varretrapp), or —, 16.5—16.65% (Bertram), or —, 12.12% (Kaufmann); thiocyanogen value 75.89%, —. Thiocyanometric analysis (Kaufmann) gave the constitution of the oil as 12.12% of saturated glycerides, 54.35% of oleic glycerides, and 33.58% of linoleic glycerides. The nature of the unsaturated acids is not clear; besides ordinary linoleic acid a mono-ethylenic acid, apparently of the palmitoleic type, is present; on oxidation it yields a hydroxy-acid (m.p. 118°, neutralisation value 198.5). The nature of *Capparidaceæ* oils from other countries is discussed.

E. LEWKOWITSCH.

Re-esterification ("Umesterung") of neutral fats with butyric acid. K. TÄUFEL and W. PREISS (Z. Unters. Lebensm., 1929, 58, 425—433).—Re-esterification, a process whereby the two components of a glyceride may be replaced by other alcohols and acids, by heating, if necessary under pressure, in an inert atmosphere, and in the presence of catalysts, has been applied commercially to the production of edible fats (cf. B.P. 160,840 and G.P. 407,180; B., 1922, 945 A; 1925, 225). An attempt has now been made to prepare materials for use as butter substitutes by the union of neutral fats with butyric acid. A mixture of 400 g. of beef tallow with 30 g. of butyric acid was heated gradually up to 235° over a period of 70 hrs. under reflux in an atmosphere of carbon dioxide with 3% of tin filings as catalyst, 7 g. of glycerin being added in the early stages. After removal of the residual acid with sodium carbonate solution the product had a yellow colour, buttery consistency, and pleasant taste, the m.p. had fallen from 43° to 37°, and 80% of the butyric acid had entered into combination. By similarly treating a hardened blubber oil, e.g., "Candelite extra" (400 g.), with butyric acid (30 g.) and glycerin (7 g.), the product had a pleasant smell resembling that of melted butter, and its consistency and taste were similar to those of beef tallow. The m.p. had been lowered from 50.0—51.5° to 42.5°. The material was very stable, and was apparently unchanged after keeping for 5 months. When kneaded with water about 51% was absorbed, whereas the original fat would absorb only 32%. When emulsified with water above the m.p. both the untreated and the "esterified" fat separated out again in 30 min. When emulsified with skim milk the emulsions of both treated and untreated fats separated when kept warm, but when stored at room temperature, although the

emulsion of the untreated fat separated, that from the re-esterified "Candelite extra" remained homogeneous. The cold fat when pressed was found to have retained about 6% of water. H. J. DOWDEN.

Sulphonated oils. C. RIESS (Collegium, 1930, 13—18).—Measured amounts of sulphonated oleic acid were kept for 7 days in contact with different amounts of 0.2*N*-sodium hydroxide, -hydrochloric acid, and -sulphuric acid, respectively, but practically no acid was split off the oil. To determine the amount of SO₄ which can be hydrolysed, 5 g. of the oil should be boiled under reflux for 30 min. with 25 c.c. of *N*-hydrochloric or -sulphuric acid, ether and sodium chloride added to it, the product titrated with *N*-alkali in presence of methyl-orange, and the SO₄ calculated from the amount of alkali remaining in excess. Excess alkali is present with the oil if the alkali titration is less than 25 c.c. The number of alkali-neutralised carboxyl groups in the oil was arrived at by titrating the oil with acid in presence of methyl-orange, and of the free carboxyl groups by titrating in alcoholic solution, using phenolphthalein as indicator. Examples of such analyses of 3 samples of Turkey-red oil are quoted, and the number of carboxyl and SO₄ groups present in samples of Turkey-red oils prepared under different conditions of temperature and duration of action is given. It is shown that positive, negative, or nil Procter-Searle figures can be obtained with sulphonated oils according as the number of alkali-combined carboxyl groups are less than, greater than, or equal to that of the SO₄H groups.

D. WOODROFFE.

Sulphonated oils. III. Properties of aqueous solutions of pure sodium ricinoleate, sodium oleate, or the sodium salt of the sulphuric acid ester of ricinoleic acid. K. NISHIZAWA and K. WINOKUTI (Chem. Umschau, 1930, 37, 33—39; cf. B., 1929, 402).—The colloidal and physical properties of these salts were compared; the last-mentioned showed the greatest degree of dispersion in aqueous solution and the solution showed the greatest surface tension towards air and kerosene (sodium oleate solutions having the lowest of the three) and the smallest viscosity. The solution of the sodium salt of the ester revealed the greatest stability towards sulphuric acid, lime, and magnesia (as measured by the amounts required to produce turbidity), and, in general, this salt exhibited the peculiar properties of sulphonated oils to an enhanced degree.

E. LEWKOWITSCH.

Preparation of pure sodium sulphoricinoleate from ricinoleic acid and sulphuric acid. K. NISHIZAWA and M. SINOZAKI (Chem. Umschau, 1930, 37, 40—44; cf. Grün and Woldenburg, A., 1909, i, 284).—The pure sulphuric acid ester of ricinoleic acid has been prepared for the first time from crude ricinoleic acid by the very slow addition of concentrated sulphuric acid to its ether solution at low temperatures. The influence of experimental conditions on the synthesis was investigated. Further details (accidentally omitted from previous accounts; cf. A., 1929, 1424) are given for the preparation of the pure, crystalline sodium salt of the ester. The acid potassium salt of the ester is very readily purified by recrystallisation from water, hence the new synthesis affords a ready method for the

preparation from crude ricinoleic acid of the pure acid salt, and from this in turn pure ricinoleic acid can be obtained by hydrolysis. E. LEWKOWITSCH.

Oxidation of paraffin. ZERNER. **Petrol-water emulsions.** VARADHAN and WATSON.—See II. **Tri- β -hydroxyethylamine emulsions.** WILSON.—See III. **Fat in washed wool.** KRAIS and BILTZ.—See V. **Fat products and leather.** SCHORLEMMER.—See XV.

See also A., May, 561, **Determination of water [in fatty substances]** (NOTEVAP). 577, **Polymerisation of methyl esters of highly unsaturated acids of train oil** (KINO).

PATENTS.

Deodorisation of fats and fatty oils. H. BOLLMANN, Assr. to M. F. FOSTER (U.S.P. 1,754,598, 15.4.30. Appl., 27.2.25. Ger., 3.9.24).—See B.P. 231,791; B., 1925, 459.

Bleaching of fatty oils, mineral oils, and the like. H. BOLLMANN (U.S.P. 1,754,599, 15.4.30. Appl., 16.12.25. Holl., 18.11.25).—See B.P. 245,745; B., 1927, 227.

Manufacture of anhydrous soap gels. V. R. KOKATNUR (U.S.P. 1,753,659, 8.4.30. Appl., 13.5.26).—See B.P. 251,290; B., 1927, 755.

Cleansing and emulsifying agent. W. PUNGS, Assr. to I. G. FARBEIND. A.-G. (U.S.P. 1,757,441, 6.5.30. Appl., 16.10.26. Ger., 21.10.25).—See B.P. 283,786; B., 1928, 237.

Treatment of soap [with rays from mercury-vapour lamps]. E. H. MORRIS (B.P. 328,043, 13.9.29).

Sulphonic acids (B.P. 298,559 and 326,815).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Catalysis in painting technique. W. OSTWALD (Z. angew. Chem., 1930, 43, 348—349).—Painters of the fifteenth century added beaten white of egg or white of egg which had been stirred with a twig from a fig tree to their paints as a binding material. The function of the twig was to break up the cell-walls of the protein; this action is shown to be due to the presence of a ferment in the sap of the fig tree. Size used in the preparation of distempers mixes completely with water only at the m.p.; at lower temperatures the mixture separates into two conjugate layers. Addition of chloral hydrate, which dissolves both in the size and water phases, lowers the m.p. of the size and hence causes the mixture to remain liquid at the ordinary temperature. A similar action is produced by hydrolysing part of the proteins in the size by addition of dilute mineral acids or by boiling with lime. A. R. POWELL.

Hiding power and tinting strength of pigments and paints. H. A. GARDNER, G. G. SWARD, and S. A. LEVY (Amer. Paint and Varnish Manufs.' Assoc., Mar., 1930, Circ. 362, 235—272).—The available methods for determining hiding power are reviewed, such drawbacks as personal equation, limited range of applicability, etc. being indicated. The differences between the hiding powers of wet and dry films are thought to be less than those obtained by different operators when

working with the cryptometer on wet films. It is considered that a simple practical test involving the brushing of paint over a surface of suitably contrasting pattern is satisfactory for normal purposes. Details of experiments using reflected and transmitted light with various types of background are given. Linoleum printed with a chessboard pattern (4-in. squares) was eventually adopted, sufficient paint being applied for perfect hiding and results being expressed as sq. ft. covered per gal. Such tests usually give results approx. 20% higher than the cryptometer readings. Tests carried out on paints aged for two weeks showed in some cases (notably that of titanium oxide) a decrease in hiding power with age, possibly due to progressive elimination of adsorbed air. A further series of experiments indicates that there is apparently no direct ratio between tinting strength and hiding power of pigments, but that the order of the pigments arranged in increasing magnitude is the same for both properties. The relation between brightness and hiding power is also discussed. S. S. WOOLF.

Identification of pigments used in painting at different periods, and other methods of examining pictures. A. P. LAURIE (Analyst, 1930, 55, 162—179).—Microscopical examination of the picture itself will yield information as to the medium, a sample of which should be mounted and treated by microchemical tests and should also be examined optically. The properties and reactions of the following ancient pigments found in illuminated MSS. and pictures are described: white lead, gold, vermilion, red lead, orpiment, massicot yellow, Naples yellow, yellow lake (Dutch pink), gamboge, malachite, verdigris, transparent copper green, Egyptian blue, ultramarine real, azurite, blue and green verditer, lakes from the *Kermes* insect, sapan wood, lac lakes from *Coccus lacti*, crimson and madder lakes, Tyrian purple, and smalt. A number of modern pigments are also discussed. Tests for recognising microscopical fragments of certain pigments in a dried oil film, such tests to be followed by dissolution of the particle and confirmation microchemically, are indicated and the results tabulated; confirmatory tests with potassium mercuric thiocyanate for lead, copper, and zinc salt, and the triple nitrite reaction for lead and copper, are described. The special pigments characteristic of Byzantine, Scot-Irish, and English MSS. are discussed and also those of the Venetian Ducali and the Coram Rege Rolls. The use of data obtained by examination of the pigments on pictures, of the brushwork, and identification of mediums is illustrated, and the application of X-rays as an additional means of deciding authenticity and for ascertaining whether there has been painting over another painting is suggested. D. G. HEWER.

What is zinc oxide? K. WÜRTH (Chem.-Ztg., 1930, 54, 201).—The technical designations of the pigments known as "zinc oxide" and "zinc white" are unsatisfactory, as the latter contains up to 99% ZnO whereas the former frequently contains only 50—60% ZnO together with other substances. It is now proposed that the name "zinc oxide white" should be employed for the former substance, and "zinc oxide colours" as a general designation for all pigments containing this constituent. H. F. HARWOOD.

Testing the permeability of "filler" films [for automobile finishing]. W. TOELDT (Farben-Ztg., 1930, 35, 1255—1256).—Such properties as colour, gloss, durability, etc. are not of vital importance for a filler, covered as it normally is by lacquer coats and in some cases by surfacing ("insulating") coats. Porosity to air and moisture is the most significant factor in evaluating a filler, and an apparatus is described for estimating this by measuring the volume of air transmitted by isolated, dry, filler films. The decrease in porosity with increase in content of binding vehicle is demonstrated by this means.

S. S. WOOLF.

Electrolytic preparation of a white lead and its properties. T. OKUNO (Mem. Coll. Eng. Kyushu, 1930, 5, 145—170).—The electrolysis of solutions of potassium chlorate and of sodium carbonate between lead electrodes has been investigated. The adsorbing power, refractive index, sp. gr., and uniformity of grain of white lead prepared electrolytically have been determined.

C. W. GIBBY.

Potentiometric determination of acidity in writing inks. H. A. BROMLEY and L. W. CAUSER (Analyst, 1930, 55, 277—279).—Callan and Horrobin's method (B., 1930, 154) has been improved by assuming an end-point corresponding to the p_H of the colour indicator used during an ordinary titration, interpolating this value into the standard equation, and then determining the null-point corresponding to E . Thus, with methyl-orange (p_H 2.8—4), $3.4 = (E - 0.250)/0.0575$ for a calomel half-cell, or $3.4 = E/0.0575 \pm K$ with a second platinum wire immersed in a standard buffer solution saturated with quinhydrone. When the latter method is used the potentiometer is adjusted to give the calculated value for E on the millivoltmeter, and the titration is continued until there is no further deflection of the galvanometer needle, which indicates the end-point, observed by means of a magnifying glass. An agar bridge is preferable to a capillary bridge.

D. G. HEWER.

Use of iron and cobalt salts in the preparation of lacquers [for patent leather manufacture]. E. SCHAD and C. RIESS (Collegium, 1930, 20—24).—The effect of adding different amounts of hydrated ferric oxide, ferric linoleate, cobalt oxalate and linoleate was determined on (a) the duration of boiling the linseed oil; (b) the sp. gr., viscosity, colour, hardness, and elasticity of the lacquer, and (c) the colour, hardness, and elasticity of the film produced on the leather. The sp. gr. of the lacquer obtained after heating for 10 hrs. was increased by additions of the driers, either separately or together. The viscosity of the heated product was diminished by additions of 0.25% or 0.5% of cobalt oxalate, but was increased by additions of ferric oxide or cobalt and iron linoleates. The viscosity of the lacquer was increased so much after heating for 10—15 hrs. with additions of more than 0.2% of ferric oxide that it could not be determined. The lag period in the action of the ferric oxide and cobalt oxalate is probably due to the conversion of these compounds into a more active form. A sudden increase in the viscosity occurred after 25 hrs. heating of the oil to which 0.5% of cobalt oxalate had been added; but it was so rapid that it was

impossible to determine the end-point. The time of boiling is reduced by additions of driers, iron salts being especially effective. The time is reduced to one half by additions of 0.5—0.75% of ferric oxide. Figures are quoted to show the reduced time required for the complete drying of films prepared from lacquers to which additions of driers have been made.

D. WOODROFFE.

Analytical data of Okume resin. M. TOMEO and J. GARCÍA-VIANA (Anal. Fis. Quím., 1929, 27, [tecn.], 135—140).—Okume resin, an oleo-resin derived from a tree (? *Aucoumea Klaineana*) native to Spanish Guinea, has been examined after probable deterioration by storage. After filtering the fused resin from impurities, it distils at 105—205°, and yields on steam-distillation 9.9% of essential oil and 89.0% of residue. The essential oil has d^{15}_4 0.8670, n^{15}_D 1.4790, $[\alpha]^{15}_D$ —27.5°, f.p. below —20°, b.p. 168°/700 mm., 175°/760 mm. (90% distils at 65—75°/25 mm. or 174—177°/760 mm.), solubility in alcohol: 1 in 7 pts. of 90%, 1 in 29 pts. of 80%, 1 in 60 pts. of 65%, acid value 1.5, ester value 12.3, acetyl value 0.91. It appears to contain 90—95% of an unidentified monocyclic terpene. The residue is completely soluble in alcohol, benzene, carbon disulphide, and ether. It has d^{15}_4 1.062, m.p. 90° (sintering at 65—70°), b.p. 125°, precipitation point 1.5—2, acid value 26, ester value 110, acetyl value 0.1, iodine value 25.52. It somewhat resembles colophony, and gives the same colour reactions. Substituted for colophony in a varnish mixture, a superior product is obtained.

R. K. CALLOW.

PATENTS.

Manufacture of coating preparations and artificial materials. O. Y. IMRAY. From I. G. FARBE-ENIND. A.-G. (B.P. 326,482, 8.9.28).—A cellulose ester of an acid of high mol. wt., e.g., a fatty acid, naphthenic acid, or resin acid, is dissolved in a drying oil, e.g., linseed or tung oil, further solvents or diluents and soluble or insoluble colouring matter being introduced if desired. Substances that react with one or more constituents of the preparation and promote hardening, e.g., cobalt or manganese resinate or oleate, benzyl chloride (in the presence of ferric chloride as a catalyst), may also be incorporated.

S. S. WOOLF.

Coating and similar compositions. A. E. WHITE. From ELLIS-FOSTER Co. (B.P. 327,095—6, 19.9.28).—(A) A cellulose ester, e.g., nitrocellulose, is mixed with a greater amount of a synthetic resin of low acid value, produced from a natural resin or resin acid, e.g., rosin, kauri, Congo, etc., a polyhydric alcohol, e.g., glycerol, or a derivative, and an organic non-resin acid, e.g., phthalic acid. Solvents, pigments, plasticisers, etc. may be added. (B) The synthetic resin complex contains cottonseed oil or its derivatives, e.g., heat-treated fatty acids.

S. S. WOOLF.

Composition of matter [water-glass paint]. P. T. HANNEN and H. D. BRUCE, Assrs. to U.S.A. (U.S.P. 1,744,116, 21.1.30. Appl., 10.4.28).—Instruments or objects, e.g., thermometers, are engraved and the engraved marks are filled in with a composition comprising a 10% solution of sodium silicate and an equal volume

of water, pigmented with manganese dioxide, carbon, lead chromate, etc. [Stat. ref.] S. S. WOOLF.

Electro-paint. L. J. D. HEALY, Assr. to FISK RUBBER Co. (U.S.P. 1,744,469, 21.1.30. Appl., 27.6.25).—A mixture of uncured rubber, finely-divided asbestos, and glue, in approximately equal proportions, and a rubber solvent is claimed as an insulating paint, suitable for use in electroplating. If desired, the paint may be hardened by means of acid sulphur chloride after air-curing. S. S. WOOLF.

Painting processes. G. B. ELLIS. From M. RENSCH (B.P. 326,649, 27.2.29).—In a paint system in which the second coat is applied to the first while the latter is still wet, the paint material for the first coat is treated with a sufficient quantity of an oxide or hydroxide of an alkaline-earth metal, or of the soaps of these metals with resinic acids, oleic acid, or the acids of waxes, whilst the vehicle for the covering coat contains as an essential constituent thickened fatty oils, boiled oils, or sulphonated fatty oils etc., caoutchouc or its decomposition products, or cellulose esters or ethers. S. S. WOOLF.

Applying a firmly-adhering metal coat to insulating plates, particularly for use in electric condensers. E. PFIFFNER, Assr. to RADIO PATENTS CORP. (U.S.P. 1,744,281, 21.1.30. Appl., 11.11.26. Ger., 12.11.25).—A sheet of cellulose ester is treated with a hydrolysing solution containing a metallic salt reactive with free cellulose to precipitate a metallic reduction product, *e.g.*, ammoniacal silver nitrate, whereby the sheet is superficially hydrolysed and a coating of silver deposited thereon. S. S. WOOLF.

Manufacture of coated articles. BRIT. CELANESE, LTD. (B.P. 303,898, 11.1.29. U.S., 12.1.28).—Intimate pulverulent mixtures of finely-divided organic derivatives of cellulose, *e.g.*, cellulose acetate, and one or more plasticisers, with or without fillers, dyes, pigments, etc., prepared (as in B.P. 282,723; B., 1928, 853) in the absence of volatile solvents or water, are applied to sheets or other articles and caused to coalesce under heat and pressure, *e.g.*, at 50—80° under 300—500 lb./in.², to form adherent coatings. S. S. WOOLF.

Paint and varnish remover. E. I. DU PONT DE NEMOURS & Co. (B.P. 315,212, 25.4.29. U.S., 9.7.28).—Solvents containing acetone (3 pts.), benzene or toluene (2 pts.), and ethylene glycol (1 pt.) are claimed. S. S. WOOLF.

Removing paint [from metals]. J. H. GRAVELL, Assr. to AMER. CHEM. PAINT Co. (U.S.P. 1,744,463, 21.1.30. Appl., 22.6.26).—An aqueous mixture (proportions stated) of sodium cresylate and hydroxide is claimed for removing paint, *e.g.*, high-baked black enamels, from steel motor-car bodies, fenders, etc. The articles are immersed in the bath maintained at 82°, washed, dipped in dilute acid (phosphoric) to remove remaining alkalinity, again washed, and dried to fit them for re-painting. S. S. WOOLF.

Working up of crude turpentine. E. WECKER (B.P. 327,166, 29.11.28. Addn. to B.P. 213,267; B., 1925, 619).—Crude turpentine is treated below 90° *in vacuo* with wet steam or a mist of water or other

liquid, if desired in admixture with steam or another gas or vapour. S. S. WOOLF.

Production of pigments and turbidity agents. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, and L. WEISS (B.P. 327,142, 27.12.28).—Soluble or volatile compounds of titanium, zirconium, and tin, in their normal condition or as concentrated solutions, are converted into hydroxides or oxides by treatment with suitable agents, *e.g.*, alkaline lyes, superheated steam, etc., and the conversion products are (preferably forthwith) heated to 500—800° if for use as pigments or to 900—1500° for turbidity agents, highly dispersive oxides resulting. The original conversion may be effected at high temperatures, *e.g.*, 600°, in which case subsequent heating may be omitted. [Stat. ref.] S. S. WOOLF.

Preparation of colloiddally dispersed materials, especially pigments in different media. IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, A. SHEPHERDSON, and A. STEWART (B.P. 326,516, 12.9.28).—An aqueous solution of a water-soluble derivative (capable of reconversion into the insoluble form) of the substance to be dispersed, *e.g.*, the leuco-compound of a dyestuff, or an aqueous solution of chemicals which produce the insoluble final product by interaction with other aqueous solutions of chemicals in the presence of non-aqueous media, *e.g.*, the formation of barium chromate by addition of barium chloride to potassium chromate, is emulsified with a non-aqueous medium, *e.g.*, linseed or castor oil, in the presence, if desired, of a protective colloid. The water-insoluble material is formed in the emulsion which is subsequently broken by suitable means, and the colloiddally dispersed material collects in the non-aqueous phase, volatile matter being then removed. S. S. WOOLF.

Varnishes, lacquers, and like coating compositions. BRIT. CELANESE, LTD. (B.P. 299,781, 29.10.28. U.S., 31.10.27).—The compositions for coating metallic and other surfaces comprise a cellulose derivative (ester or ether), a volatile solvent, and a synthetic ketone-phenol-aldehyde resin of the type described in B.P. 299,065 (B., 1930, 338); gums, high-boiling solvents, plasticisers, etc. may be added as desired. E. LEWKOWITSCH.

Brushing lacquer. S. D. SHIPLEY, Assr. to ATLAS POWDER Co. (U.S.P. 1,744,085, 21.1.30. Appl., 21.10.25).—The active solvent in the lacquer is reduced to a minimum for retention in solution of the nitrocellulose content, relatively large proportions of gasoline being used as diluent. A preferred composition is ethyl acetate 8%, camphor oil 1.5%, butyl acetate 20.5%, gasoline 37.5%, toluol 27.5%, resin solution 5%, combined with 3 oz. of nitrocellulose per gallon. S. S. WOOLF.

Recovering vapours of varnishes and the like. D. LARKIN (U.S.P. 1,743,554, 14.1.30. Appl., 13.3.25).—Varnish-fume recovery apparatus is claimed, comprising a casing containing spaced vertical partitions having openings alternately at the upper and lower ends, vertical overflow pipes passing through the bottom wall of the casing, and vertical spray-nozzles passing through the top wall of the casing and having

their discharge ends in spaced parallel relation with the intake ends of the overflow pipes. By such an arrangement a varnish-laden atmosphere when passed through the casing is folded by the partitions into parallel columns having currents moving diagonally through the spaces between the nozzles and the overflow pipes; simultaneously jets of precipitating fluid (water) are injected medially into the columns and directed downward towards the intake ends of the overflow pipes in lines at angles to the movements of the fume-currents. The water is separated from the condensed vapour by processes depending on immiscibility or difference in sp. gr., and is then returned automatically to the spray-nozzles. S. S. WOOLF.

Phenolic resins and their manufacture. H. WADE. From BAKELITE CORP. (B.P. 326,545, 15.12.28).—A fusible, non-reactive, phenolic resin is obtained by heating a fusible, non-reactive, phenol-methylene resin, *e.g.*, a formaldehyde resin, with furfuraldehyde, a basic condensing agent, *e.g.*, lime, and, if desired, a small proportion of a methylene-containing hardening agent, *e.g.*, hexamethylenetetramine, and/or a lubricant, *e.g.*, stearic acid, at 130–150° for a time sufficient to convert the resin into a mass which is brittle when cold, but insufficient to destroy its non-reactive character. The fusible methylene resin may be heated with the lime and thereafter furfuraldehyde added and the heating continued. S. S. WOOLF.

Manufacture of phenol-formaldehyde condensation product. G. W. STRYKER (U.S.P. 1,737,031, 26.11.29. Appl., 1.10.27).—Phenol is heated and agitated with a predominating quantity of formaldehyde in the presence of a catalyst (ammonia), for not more than 80 hrs. at 48–57°, water being thereby eliminated without stratification of the mass, and the resultant thick syrupy resin is heated for 4–5 hrs. at 82–93°. S. S. WOOLF.

Manufacture of phenol-furfuraldehyde resin. E. E. NOVOTNY, Assr. to J. S. STOKES (U.S.P. 1,737,121, 26.11.29. Appl., 29.8.23).—A potentially reactive liquid or semi-solid condensation product of phenol and furfuraldehyde is claimed, wherein the constituents are completely in chemical combination, an active compound containing the methylene linking, *e.g.*, formaldehyde, being incorporated as hardening agent. In the absence of heat such product maintains its condition without reaction for a considerable time. The product of partial condensation of a phenolic substance with an excess over equimolecular proportions of another substance, which is itself capable both of volatilisation and of resinification by catalytic action without chemical combination with any other reagent, and which is adapted to act as an ultimate hardening agent for the intermediate condensation product, is also claimed, the condensation reaction being controlled so that it may be arrested at any desired stage. S. S. WOOLF.

Manufacture of artificial resins. BRIT. CYANIDES CO., LTD., E. C. ROSSITER, and W. C. DAVIS (B.P. 327,154, 22.12.28).—A phenol is condensed, at 120–130° in the presence of 1–10% of its weight of dicyanodiamide, with an aldehyde, *e.g.*, formaldehyde or furfuraldehyde. S. S. WOOLF.

Manufacture of objects [of high gloss] by moulding plastic masses of condensation products. A. SCHMID, and METROPOLE DEVELOPMENTS, LTD. (B.P. 326,475, 12.12.28).—Condensation products in which the process of polymerisation is effected by acid or acid-yielding media are cast in mechanically robust moulds having a tin surface capable of being highly polished. S. S. WOOLF.

Manufacture of [artificial resin] foils, films, etc. WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 318,580, 15.2.29. Ger., 6.9.28).—Viscous solutions, preferably preheated to 100°, of condensation products of aldehydes with urea or its derivatives in the presence of softening agents, *e.g.*, glycerol, methycyclohexyl adipate, etc., which have preferably been added after the commencement of the urea-aldehyde condensation, are applied to a travelling base and allowed to solidify thereon, the films thus formed being removed when they cease to be superficially sticky. S. S. WOOLF.

Refining of raw oil of turpentine. F. SCHLOSSER and M. PAQUIN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,755,668, 22.4.30. Appl., 31.8.25. Ger., 12.9.24).—See B.P. 239,878; B., 1925, 999.

Preparation of coloured nitrocellulose varnishes. E. RICHTER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,756,100, 29.4.30. Appl., 14.4.27. Ger., 1.2.26).—See B.P. 291,539; B., 1929, 614.

[Stoving] oven. H. L. WARD, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,744,817, 28.1.30. Appl., 9.12.24).

Azo pigments (B.P. 302,173).—See IV. **Cellulose ester coating compositions** (B.P. 326,515).—See V. **Non-splintering glass** (B.P. 326,520).—See VIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Behaviour of progressively racked rubber with respect to ageing, hysteresis, m.p., and energy effects. H. FEUCHTER and E. A. HAUSER (Gummi-Ztg., 1930, 44, 1307–1308).—A condensed version of a paper already abstracted (*cf.* B., 1930, 112). D. F. TWISS.

Coal and carbon as raw materials. MATIGNON.—See II. **Butadiene from cracked hydrocarbons.** FROLICH and others.—See III.

See also A., May, 609, **Isoprene and caoutchouc** (STAUDINGER and others).

PATENTS.

Vulcanisation of rubber. I. G. FARBENIND. A.-G. (B.P. 311,735, 7.5.29. Ger., 15.5.28).—2-Amino-4:5-dihydroglyoxalines, especially the 2-*p*-toluidino-compound, are used as vulcanisation accelerators.

C. HOLLINS.

Composite rubber-faced surfacing material. L. P. F. F. CRESSON (B.P. 325,302, 21.11.28. Addn. to B.P. 303,400).—A surfacing material comprises a base of cement or the like, reinforced with iron, if desired, and faced with rubber which has been vulcanised *in situ*, there being sufficient sulphur at the interface to cause the formation of a stratum of hard rubber; for this purpose a layer of vulcanised rubber latex com-

position containing sufficient free sulphur may be applied to the surface of the cement base. The rubber facing or the part of it adjacent to the base contains ingredients such as magnesite cement or lead oxide capable of combining with the silicic acid of the cement, thereby ensuring interfacial unity between the two parts.

D. F. TWISS.

Concentration of rubber latex. I. TRAUBE, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,754,842, 15.4.30. Appl., 3.9.24. Ger., 23.10.23).—See B.P. 226,440; B., 1925, 139.

Artificial compositions, especially those resembling rubber. W. FRANKENBURGER and C. STEIGERWALD, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,756,943, 6.5.30. Appl., 5.7.28. Ger., 23.7.27).—See B.P. 294,474; B., 1929, 1024.

Dry moulding [of rubber articles]. W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,756,411, 29.4.30. Appl., 10.12.27).—See B.P. 302,151; B., 1930, 471.

Accelerator of vulcanisation [of rubber]. J. TEPPEMA, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,757,930, 6.5.30. Appl., 10.12.27).—See B.P. 302,142; B., 1930, 113.

Stabilised dispersions (B.P. 301,805).—See II.
Vulcanisation accelerators (B.P. 306,558 and 306,842).
Vulcanised rubber (B.P. 326,525).—See III.

XV.—LEATHER; GLUE.

Manufacture and application of fat products in the leather industry. K. SCHORLEMMER (Collegium, 1929, 526—536).—Degras can be made from herring and other fish oils and is miscible with wool grease or mineral oil. Fats undergo oxidation in the leather and the acidity has been observed to increase. Sulphonated oils are used on chrome-tanned leathers. Thus 52 kg. of neatsfoot oil is treated with 3.4 kg. of concentrated sulphuric acid, kept overnight, and repeatedly washed with solutions of Glauber's salt. Pieces of pelt which had been treated with 1:1 mixture of sulphonated cod oil and sulphonated neatsfoot oil yielded a product which possessed many of the properties of leather. There was a contraction of the fibres, and this has been observed also in fat-liquoring patent leather with these oils. The Fahrion hot-water test for fat-liquored chrome leather gave values 1—2% higher than those for untreated leather. In the manufacture of sulphonated oils, 10—25% of sulphuric acid (calc. on wt. of oil) is customary. The temperature should be kept as low as possible. The time of reaction should be 24 hrs., or longer for sulphonated castor oil if the product is to give a clear solution in water. A superior product is obtained if the sulphonated oil is washed with Glauber's salt solutions than if brine liquors are employed, and it requires least sodium hydroxide addition to render it completely soluble in water. Similar effects are produced by washing sulphonated neatsfoot oil with sodium sulphate and sodium chloride solutions, respectively. The fat-liquoring process is sometimes affected by the shade which is being produced. Examples are quoted where the sulphonated oil required to be more acid for dyeing blacks and less acid for certain pale shades. Sulphonated oils which have separated during storage in cold weather

can be restored to their original condition by adding ammonia and alcohol.

D. WOODROFFE.

Tannins in [leaves of] chestnut and Sicilian sumac. W. MÜNZ (Collegium, 1929, 499—512).—From the leaves of the chestnut (*Castanea vesca*) 3.8% of a tannin was isolated, which was shown to be a galloylhexose, a simpler substance than oakwood tannin, which yields more ellagic acid. The tannin from sumac leaves (*Rhus coriaria*) was prepared by the ethyl acetate method. Its acidity was high, but it contains no free carboxyl groups, since it is extractable from alkaline solutions by ethyl acetate. It has $[\alpha]_D^{20} +53.3^\circ$ in 5% solutions, increasing with increasing dilution. By acid hydrolysis 82.2% of gallic acid and 6% of dextrose were obtained; treatment with tannase afforded 88% of gallic acid and 8.2% of dextrose. On methylation with diazomethane and subsequent hydrolysis with methylalcoholic potash, only two thirds of the acid was recovered. Sumac tannin is largely pentagalloylglucose or is closely related to it.

D. WOODROFFE.

Violet-fluorescing material in pine bark and its extracts. O. GERNGROSS (Collegium, 1929, 512—519).—By removing the resin from pine bark and then extracting with dry ether, an almost colourless powder was obtained by evaporating the ether. This powder is shown to be the cause of the violet fluorescence observed when infusions of pine bark or its extracts are viewed in the light of a Wood lamp. On acetylation, a hepta-acetyl compound (sinters 75° , m.p. about 100°), of empirical formula $C_{33}H_{33}O_{13}$, was obtained, which no longer showed fluorescent properties until the acetyl groups were removed.

D. WOODROFFE.

Vegetable tanning materials of Lybia. G. A. BRAVO (Annali Chim. Appl., 1930, 20, 171—180).—The materials were extracted in a Procter extractor with water at about 80° , the solution being analysed by both the filter and the Baldracco-Darmstadt methods. The percentage results obtained are respectively as follows:—Root bark of *Rhus oxyacantha*, Cav.: tans 17.15, 15.65; soluble non-tans 6.05, 7.55; insoluble non-tans (by diff.) 61.5, 61.5; moisture 15.3, 15.3. Bark of *Acacia saligna*, Benth.: tans 15.55, 14.43; soluble non-tans 9.58, 10.70; insoluble non-tans (by diff.) 63.07, 63.07; moisture 11.8, 11.8. Bark of *Acacia longifolia*: tans 9.72, 8.58; soluble non-tans 10.84, 11.98; insoluble non-tans (by diff.) 66.87, 66.87; moisture 12.57, 12.57. Root bark of *Acacia lophanta*, Willd.: tans 9.06, 7.98; soluble non-tans 11.70, 12.78; insoluble non-tans (by diff.) 67.37, 67.37; moisture 11.87, 11.87. *Acacia farnesiana*, Willd.: tans 5.2 (filter), soluble non-tans 5.4; insoluble non-tans (by diff.) 76.12; moisture 13.28. Bark of *Tamarix articulata*, Vhl.: tans 14.72, 14.14; soluble non-tans 9.62, 10.2; insoluble non-tans (by diff.) 61.78, 61.78; moisture 13.88, 13.88. Galls of *Tamarix articulata*, Vhl.: tans 36.77—43.91; soluble non-tans 16.76—9.68.

T. H. PORE.

Water content of solid vegetable tannin extracts. W. VOGEL (Collegium, 1930, 3—13).—A solid Argentine quebracho extract contained 26.2% of moisture as it was run out of the extract plant, 25% after allowing 4 days for complete solidification, 22.2% after it had

been kept during the hot season in an Argentine factory, 21.8% after transportation overseas, and 18.5% after 1 year in an unheated room in the temperate zone. Some bags of extract were kept for 3–4 years in a temperate climate, when the moisture content was found to be 12–13% and 14–15% for crude and sulphited extracts, respectively; this value is the average for air-dry quebracho extracts, and varies slightly with the temperature and humidity of the surrounding atmosphere. The moisture content of finely-powdered quebracho extract is lower, viz., 10% and 11–13% for the crude and sulphited extracts, respectively. Under the same external conditions, 9% and 10.5% of moisture is absorbed by the absolutely dry crude and sulphited extracts, respectively. Generally, 10–12% of water (6.7–14.3%) is contained in powdered, air-dry, tannin extracts. Less water is taken up by absolutely dry, powdered extracts generally than corresponds with the water content of the air-dry material.

D. WOODROFFE.

Neutralisation of one-bath chrome-tanned leather. W. SCHINDLER, K. KLANFER, and E. FLASCHNER (Collegium, 1929, 472–499).—Pieces of one-bath chrome-tanned leather were neutralised, and the amount of SO_4 was determined in the finished leather by the amount of 0.1*N*-sodium bicarbonate neutralised in warm solution. The neutralising agents could be divided into three groups: (A) sodium bicarbonate and carbonate, (B) sodium hydroxide and silicate, (C) ammonia with and without ammonium chloride. Borax is intermediate between A and B. More acid was neutralised in the grain than in the middle layer of leather treated with 2% of sodium bicarbonate or its equivalent of sodium carbonate. The difference was less marked after prolonged treatment. The acid content of the grain was much less with leathers treated with the B materials, whilst practically no acid was removed from the middle tissue. The same effect was obtained to a certain extent with borax. Practically no acid appeared to be removed from the leather by treatment with group C, although the alkalinity of the neutralising liquor was diminished. It is suggested that entry of the ammonia or ammonium salts into the chromium complex takes place. The intense neutralising effect of borax and sodium silicate, respectively, is attributed to the replacement of the combined acid in the leather by the free boric acid or silicic acid.

D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leathers. V. W. J. CHATER (J. Soc. Leather Trades' Chem., 1930, 14, 28–37; cf. B., 1929, 1025).—A number of calf-pelt strips were treated with buffer solutions having p_H 1–12, then shrunk according to the author's method, the shrunken pieces being dried out and measured. The dimensions were found to be a function of the p_H . Shrinkage curves were derived for beef tissue and for oak-bark- and quick-tanned leathers. The initial shrinkage temperature of these leathers was increased by soaking them in water, and still more so by soaking in solutions of formaldehyde. Shrinkage curves were derived for sole leathers in various stages of tanning.

D. WOODROFFE.

Determination of sulphuric acid in leather. J. VON SCHROEDER (Collegium, 1929, 520–526).—Free sulphuric acid was detected, by the Balland and Maljean method ("Leather Chemists' Pocket Book," Spon, 1919, 190), in vegetable-tanned leathers on which no sulphuric acid had been used, but only sulphited extracts. Low results for alkali sulphate are obtained by van der Hoeven's method (B., 1924, 758, 841), and not all the sulphuric acid is removed from the leather by Immerheiser's method (B., 1920, 731 A). The following method has been devised by the author. The leather (20 g.) is soaked in 100 c.c. of distilled water for a few hours, the liquor poured off, the leather soaked in a further quantity of water, and this extract added to the first, the process being repeated over a period of 2 days until 1 litre of extract has been obtained. The extract is then poured through hide powder in a filter-bell, the first 150 c.c. of filtrate being rejected, and the total sulphates are determined as barium sulphate in the next 250 c.c. A second 20 g. of the leather are extracted similarly after 3–5 g. of sodium acetate have been added to it, the extract is acidified with acetic acid, filtered through a filter-bell as above, and the total sulphates are determined. The difference between the two determinations enables the free sulphuric acid to be calculated. Negligible amounts have been obtained by this method on acid-free leathers which had given a high Balland and Maljean figure. Hydrochloric acid when determined by the new method is found to be unaffected by the presence of sulphites, synthetic tannins, sulphonated oils, or magnesium salts.

D. WOODROFFE.

Physico-chemical problems in the glue and gelatin industry. G. HEDRICH (Z. Electrochem., 1930, 36, 156–163).—A survey. H. F. GILLBE.

Modified shaking method for analysing tannins, and the Darmstadt apparatus. G. BALDRACCO (J. Soc. Leather Trades' Chem., 1929, 13, 365–375, and Collegium, 1929, 450–453).—See B., 1929, 485.

Lacquers for patent leather. SCHAD and RIESS. —See XIII.

PATENTS.

Dividing liquid substances into drops (B.P. 313,566).—See I. **Paper etc. from leather waste** (B.P. 326,936).—See V. **Protecting fur, hair, etc. against moth** (B.P. 326,451 and 326,567).—See VI.

XVI.—AGRICULTURE.

Classification of moorland soils (podsolised moor soils). W. KASSATKIN (Mitt. Staatl. Inst. Versuchsagron. Abtg. Ackerbau, Leningrad, 1928; No. 14, 38 pp.; Bied. Zentr., 1930, 59, 149–150).—A discussion of the formation and classification of soils of the State Institute. A. G. POLLARD.

Soil structure. III. Influence of soil swelling on the determination of capillary and non-capillary porosity by means of saturation of soil with water. A. T. TRULIN (Rep. Agric. Exp. Stat. Perm., No. 3, 1929; Proc. Internat. Soc. Soil Sci., 1930, 5, 35).—During the saturation of soil with water for determining the capillary porosity, the swelling of the colloids is sufficient to produce false values. Replacement of water

by an inert liquid, *e.g.*, xylene, overcomes this fault. Differences between the capillary and non-capillary porosity of soils indicated by water and by xylene increase with the proportion of colloids and the size of the aggregates. (Cf. B., 1929, 568.) A. G. POLLARD.

Nitrate-assimilating power of the soil, and some nitrate-assimilating soil bacteria. F. B. SMITH (Proc. Iowa Acad. Sci., 1928, 35, 69—74).—The nitrate-assimilating power of soil is measurable.

CHEMICAL ABSTRACTS.

Effect of calcium carbonate and of sulphuric acid on the acidity of various peats. P. TUORILA (Wiss. Veröff. finn. Moervereins, 1928, No. 8, 75 pp.; Bied. Zentr., 1930, 59, 103—106).—Neutralisation of acid peats was least rapid with calcium carbonate, followed in ascending order by lime, sodium carbonate, and sodium hydroxide. The effects of the hydroxide and carbonate of either base were similar at $p_H < 6.0$, but at $p_H 7.0$ the hydroxides were more active. The rate of neutralisation with calcium carbonate increased with the initial p_H value of the peat. Methods for calculating the lime required for neutralisation are given. Difference in the botanical composition of the peats affected the change of p_H per unit of lime added. In individual peats this value was not appreciably influenced by the extent of humification nor by the ash content. Changes in p_H following liming were to some extent correlated with the density of the peat. Alterations in p_H resulting from treatment with a given amount of sulphuric acid tended to decrease as the total amount of acid applied was increased. The sulphuric acid of superphosphate acted similarly. A. G. POLLARD.

Lime : magnesia ratios in dolomitic limestones as influencing solution and soil reaction. W. H. MACINTYRE and W. M. SHAW (J. Amer. Soc. Agron., 1930, 22, 14—27).—The higher is the proportion of calcium, the greater is the alkalinity of carbonated water extracts and the more quickly is equilibrium attained in initial carbonated water suspensions. The effect of repeated treatment is recorded. Calcium and magnesium carbonates, when added to the suspensions, are reciprocally repressive on solubility and mutually protective to the dolomite solid phase. Acids produced in a soil dolomite medium combine preferentially with magnesium. Under humid conditions, addition of dolomitic limestone cannot produce a toxic condition from an accumulation of magnesium *per se*.

CHEMICAL ABSTRACTS.

Application of lime and phosphates to podsol soils. A. T. TUULIN and A. E. WOSBUTSKAJA (Agric. Exp. Stat. Perm, 1926, No. 1; Proc. Internat. Soc. Soil Sci., 1929, 4, 390—393).—Crop increases following the application of rock phosphate (with potash and nitrogen) to podsols showed two maxima in the regions $p_H 4.0$ — 4.5 and 7.5 — 8.0 . The acid maximum is attributed to increased phosphate solubility under acid conditions, and the alkaline maximum to the indirect effect of increased nitrification. Examination of the plant sap confirmed the rapid intake of phosphorus within the two optimum ranges. The value of lime used in conjunction with superphosphate lies in its stimulative effect on nitrification. (Cf. B., 1929, 758.) A. G. POLLARD.

Phosphate manuring on acid soils. H. KAPPEN (Superphosphat, 1929, 5, 199—206; Bied. Zentr., 1930, 59, 113—114).—Pot-culture trials with spurrey are described in which the efficiency of phosphatic fertilisers on acid, neutralised, and alkaline soils is examined. On acid mineral soils superphosphate was as effective as basic slag. On neutralised mineral soils superphosphate was superior to basic slag for lime-sensitive plants (spurrey), but inferior for acid-sensitive plants (rape). Superphosphate did not increase soil acidity. The neutralising effect of basic slag on acid soils was small. Rock phosphates were not sufficiently decomposed either by zeolitic acids in acid soils, or by simultaneously applied physiologically acid fertilisers, to permit of their substitution for easily soluble phosphates. The unusually great assimilating power of summer rape enables this plant to utilise rock phosphates to an extent which is independent of the degree of soil acidity.

A. G. POLLARD.

Influence of tillage on the porosity and structure of soil. NOVÁK-ŠIMEK (Bull. Czechoslov. Acad. Agric., 1929, [v], 558; Proc. Internat. Soc. Soil Sci., 1930, 5, 33—35).—The structure of soil is examined by grading the sample in its natural condition by means of a series of sieves (20-, 10-, 5-, and 2-mm. circular holes). Analyses so made agree well with determinations of pore space.

A. G. POLLARD.

Chemical characteristics of soils from the experimental fields of the Department of Agriculture of the [Russian] State Institute for experimental Agronomy. N. SOKOLOV (Mitt. Staatl. Inst. Versuchsg. agron., Abtg. Ackerbau, Leningrad, 1928, No. 12; Bied. Zentr., 1930, 59, 147—148).—The nutrient contents, absorptive capacity, degree of saturation, and relative productivity of these soils are recorded.

A. G. POLLARD.

Photoactivity of [Czechoslovakian] soils. L. SMOLÍK (Bull. Czechoslov. Acad. Agric., 1929, [v], 569; Proc. Internat. Soc. Soil Sci., 1930, 5, 32).—Photoactivity in the soils is probably a variable property. Few soils exhibit photoactivity, and in upper profiles this cannot be induced by the exposure of soil to solar radiation, to a strong electric field, or by storage *in vacuo*, in oxygen, or in carbon dioxide.

A. G. POLLARD.

Properties of soils which influence soil erosion. H. E. MIDDLETON (U.S. Dept. Agric., Tech. Bull. 178, 1930, 16 pp.).—A study of the physical and chemical properties of three erosive and three non-erosive soil types indicates that the properties having the greatest influence on soil erosion are indicated by the dispersion ratio, the ratio of colloid to moisture equivalent, the erosion ratio, and the silica-sesquioxide ratio. The following are tentatively suggested as limiting values for these ratios: for non-erosive types a dispersion ratio of < 15 , a ratio of colloid to moisture equivalent of > 1.5 , and an erosion ratio of < 10 . No chemical property studied was found useful in differentiating erosive and non-erosive soils, though it is considered that the dispersivity of a soil is influenced by the quantity and nature of the exchange bases present, and the silica-sesquioxide ratio is the determining influence on physical properties.

E. HOLMES.

Dynamics of physical properties of soils in different cultural conditions. N. KOURTIAKOV (Ann. Sci. Agron., 1929, 46, 460; Proc. Internat. Soc. Soil Sci., 1930, 5, 39—40).—Changes in soil porosity, aeration, and power of imbibition during a 4-year rotation are examined.

A. G. POLLARD.

Dynamics of soil solutions in podsol areas. S. A. ZACHAROV (Trans. Agric. Inst., Krasnodar, 1929, 6; Proc. Internat. Soc. Soil Sci., 1930, 5, 38—39).—Factors affecting the concentration of the soil solution are examined and the seasonal changes as the solution passes from one horizon to another are described.

A. G. POLLARD.

Solubility of Van Bemmelen's A-zeolitic complex in hydrochloric acid under different conditions of time and amount of acid. R. TRNKA and HAUPT (Mitt. Czechoslov. Akad. Landw., 1929, 5, 577; Proc. Internat. Soc. Soil Sci., 1930, 5, 28).—In the extraction of soils with 20% hydrochloric acid solution (Van Bemmelen) an increase in the quantity of acid used or in the period of extraction leads to a steady increase in the amount of material dissolved. During prolonged extraction difficultly-soluble complex silicates may be decomposed. The prescribed conditions of extraction must be adhered to, since the process is not an exact one and serves merely as a means of comparison of soil properties.

A. G. POLLARD.

Acid and alkaline extracts of soils. V. I. PARAMONOV and B. P. NIKOLSKI (Mitt. Ackerbau abt., Inst. exp. Agron., Leningrad, 1929, No. 19, 24 pp.; Bied. Zentr., 1930, 59, 145—146).—Soils of similar degrees of unsaturation with bases but with varying humus contents were extracted with 0.05N-acid and alkali and the extracts titrated electrometrically. High proportions were present in all extracts. Acid removed more alumina from unsaturated than from saturated soils. For the titration of extracts containing aluminium, bromothymol-blue or litmus was preferred as an indicator. The use of phenolphthalein, as in the Daikuhara method, led to less satisfactory results. The amount of exchangeable bases in a soil was approximately equal to the amount of acid neutralised by the soil during the acid extraction. Certain soil minerals suffer decomposition by 0.05N-sulphuric acid. The exchangeable base content of chernozem soils was decreased by saturation with ammonia following treatment with ammonium chloride solution. Treatment with calcium chloride solution increased the exchangeable base content. Alkaline soil extracts showed considerable buffer capacity, which increased with increasing humus content and with decreasing calcium content. Organic matter in these extracts was coagulated at pH 3—4. Aluminium hydrosol is protected by the humus and is not coagulated.

A. G. POLLARD.

Soil acidity. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1928, [22], 511—516; Proc. Internat. Soc. Soil Sci., 1930, 5, 22—23).—Results of earlier investigations (B., 1929, 32, 569) are confirmed on other soil types. The toxicity of acid soils cannot be ascribed to the presence of soluble aluminium, the concentration of which did not exceed 1 pt. per million even in very acid soils. In determining the buffer capacity of soils by

titration with soda, the calculated amounts of lime required to produce a given reaction are too low. Better results are obtained by titration with calcium hydroxide or bicarbonate. In North German and Swedish soils there is a relationship between buffer action and hygroscopicity which is not apparent in the soils of Java.

A. G. POLLARD.

Nitrogen content of the soil as related to the precipitation-evaporation ratio. H. JENNY (Soil Sci., 1930, 29, 193—206).—Relationships between the nitrogen content of soils and the mean temperature and humidity of the atmosphere are examined. In both temperate and subtropical areas the nitrogen content increases logarithmically with the N : S quotient (i.e., moisture precipitation : saturation deficit in the air; cf. Meyer, B., 1926, 684). In subtropical areas the nitrogen content of timber soils is not influenced by humidity factors. In temperate soils the C : N ratio (average 11.3) does not vary with humidity conditions.

A. G. POLLARD.

Periodicity of the nitrate content of soils. H. N. BATHAM and L. S. NIGAM (Soil Sci., 1930, 29, 181—190).—A survey of the nitrate contents of soils from many parts of the world shows a general similarity of seasonal changes. Nitrification is mainly controlled by the intensity of solar activity.

A. G. POLLARD.

Sulphate-sulphur in certain types of soils. H. STREMMER (Chem. Erde, 1930, 5, 254—259).—Peaty soils from the neighbourhood of Danzig contain small amounts of sulphates, free sulphuric acid, and sulphide-sulphur, which vary at different depths and in different seasons of the year (cf. A., 1929, 168).

L. J. SPENCER.

Amount of "lipoids" in the principal Moravian soils. L. SMOLIK (Bull. Czechoslov. Acad. Agric., 1928, No. 3, 249; Proc. Internat. Soc. Soil Sci., 1930, 5, 31).—The "lipoid" content (including fats, waxes, phytosterol, phosphatides, etc.) of a number of soils was determined by extraction with ether for 6 hrs. Values varied from 0.0 to 0.8%.

A. G. POLLARD.

Exchange capacity of organic and mineral soil constituents. N. SOKOLOV (Mitt. Ackerbauabt. Staatl. Inst. exp. Agron., Leningrad, 1929, No. 13, 13 pp.; Bied. Zentr., 1930, 59, 146—147).—The high base-exchange capacity of chernozem soils is not primarily attributable to their humus content, but depends on the nature of the parent rock and the process occurring during soil formation. Humus tends to increase the exchange capacity of chernozem soils, but decreases that of podsoils owing to the progressive decomposition of the unsaturated complex. The high exchange capacity of humus-bearing marl soils is traced exclusively to the proportion of parent rock which they contain.

A. G. POLLARD.

Soil moisture phenomena in a saturated atmosphere. L. B. LINFORD (Soil Sci., 1930, 29, 227—237).—Experimental evidence is advanced indicating that the angle of contact between glass and water and between many soil minerals and water is zero or very small. Foreign matter, notably fats and fatty acids, largely increases the angle of contact. The bearing of this on measurements of the hygroscopic phenomena in soil is discussed.

A. G. POLLARD.

Biochemical reactions in the formation of humus by micro-organisms in the soil. J. STOKLASA (Biol. der Pflanzen, 1929, 17, 272—295; Proc. Internat. Soc. Soil Sci., 1930, 5, 43—46).—The process of humus formation from plant materials as a result of the inter-related activities of animals, insects, and soil fungi and bacteria is examined and discussed. Humus is to be regarded as a respiratory by-product of micro-organisms. Lignin compounds are the real sources of material for the production of humus, which is a mixture of decomposed and undecomposed lignocellulose, lignin, and microbiological cell substances. The nature of forest humus varies with the species of tree from which it is formed. A. G. POLLARD.

Rhythmic variations in the activity of soil micro-organisms. N. JOHANSSON (Svensk Bot. Tidskr., 1929, 23, 241—260; Proc. Internat. Soc. Soil Sci., 1930, 5, 47—48).—The rate of production of carbon dioxide by soil micro-organisms is rhythmic in character. The nature of the variations in northern soils differs from that in tropical soils. Carbon dioxide production is usually greater by day than by night, differences being more marked at periods of maximum activity. A. G. POLLARD.

Azotobacter in Finnish soils. W. BRENNER (Agrogeol. Med., Helsingfors, 1924, No. 20; Proc. Internat. Soc. Soil Sci., 1930, 5, 48).—The primary cause of the rarity of *Azotobacter* in the soils is their acidity and lack of buffer capacity, but instances are recorded of soils which after suitable adjustment of reaction still prove unfavourable to this organism. A. G. POLLARD.

Reaction of Finnish soils. W. BRENNER (Agrogeol. Med., Helsingfors, 1924, No. 19; Proc. Internat. Soc. Soil Sci., 1930, 5, 20—21).—Finnish soils are classified according to type, origin, and reaction. A. G. POLLARD.

Reaction of cultivated Finnish soils. A. SALMINEN (J. Agric. Sci., Helsingfors, 1929, 40—48; Proc. Internat. Soc. Soil Sci., 1930, 5, 21).—A survey. A. G. POLLARD.

Variations in the reaction of cultivated soils. A. SALMINEN (J. Agric. Sci., Helsingfors, 1929, 129; Proc. Internat. Soc. Soil Sci., 1930, 5, 12—14).—Changes in the acidity of field soils at varying depths due to changes in moisture content are recorded. A. G. POLLARD.

Influence of various mineral carbonates on soil reaction. W. BRENNER (Bull. Agrogeol. Inst. Finland, 1928, No. 25; Proc. Internat. Soc. Soil Sci., 1930, 5, 10—12).—The action of various liming materials on acid peat and clay soils is examined. By the addition of successive quantities of chalk, soil reaction gradually attains a constant p_H value of 7.0—7.2 for peats and 6.8 for clays. The neutralising value of limestone preparations of the same fineness of division are proportional to their content of calcium carbonate. The action of dolomite on soils is less definite, being more rapid with clays than with peats. The effect of fineness of grinding of chalk on its reaction with soil is small, while the soil still remains acid, but further action is slower as the particle size increases. This effect is more marked with dolomite than with chalk, and is

more apparent in peat than in clay. Complete neutralisation of peat occupies several months. In general, dolomite ground to pass a 0.2-mm. sieve is a serviceable neutralising agent for acid soils if a month be allowed for its action. A. G. POLLARD.

Soil reaction and agricultural crops. A. LØDDESOE (Meld. Norges Landbruksh., 1929, 9, [4—5]; Proc. Internat. Soc. Soil Sci., 1930, 5, 14—16).—Superphosphate and potash salts, applied singly or mixed, caused no change in the reaction of acid soils, but Norwegian saltpetre alone or mixed with superphosphate and potash salts produced slight changes towards alkalinity. Stall manure without artificial fertilisers did not affect soil reaction. Titration of the soils under examination showed that manuring slightly increased their acid-fixing capacity, but the effects on base-fixing capacity were irregular. Use of fertiliser mixtures containing calcium increased the exchangeable calcium of soils. Soils rich in exchangeable calcium were usually correspondingly poor in exchangeable potassium and magnesium. Use of fertilisers containing potash did not increase the exchangeable potassium of soils. Relationships between the p_H value of a soil, its total content of exchangeable bases and their nature and proportion are dependent on the nature of the absorbing soil complex. A. G. POLLARD.

Soil reaction in the surroundings of Rožnov, with special reference to meadows and pastures. NOVÁK and MALÁŠ (Mitt. Czechoslov. Acad. Landw., 1928, 4, 461; Proc. Internat. Soc. Soil Sci., 1930, 5, 17—19).—Relationships between soil reaction and the growth of characteristic plants are examined in detail. Cultivated soils generally have a higher p_H value and a wider range of variation than uncultivated ones. The profiles of cultivated soils show a decrease in p_H value with increasing depth. The reaction of forest soils of similar type varies with the predominant type of tree. Particular species of grasses associated with soils of various ranges of reaction are recorded. A. G. POLLARD.

Degradation of rendzina soils in the Moravian Karst. J. ZVORYKIN (Mitt. Czechoslov. Akad. Landw., 1929, 5, 598; Proc. Internat. Soc. Soil Sci., 1930, 5, 59—60).—Chemical and mechanical analyses of the various profiles of rendzina soils are recorded and the chemistry of their degradation processes is discussed. A. G. POLLARD.

Methods for the chemical and physical analysis of soils. I. KANIWETZ (Pub. Centrallab. Agric. Chem., Kiev, 1928; Proc. Internat. Soc. Soil Sci., 1930, 5, 40—42).—The effect of solutions of varying concentrations of hydrochloric and acetic acids and of sodium and ammonium chlorides on the dispersion of soils for mechanical analysis is examined. The best dispersion was produced by *N*-sodium chloride. Repeated boiling of the suspensions did not alter the degree of dispersion. Pretreatment of the soil with hydrochloric acid followed by ammonium chloride tended to reduce the observed clay fraction. The new British method for mechanical analysis is not satisfactory if a neutral salt solution or hydrochloric acid alone is used. A modified method consists in washing the soil with

0.05N-hydrochloric acid or 0.16N-acetic acid until no more calcium is removed, then with N-sodium or ammonium chloride solution, and, finally, heating for 6–24 hrs. Subsequent analysis is thereby shortened. With humus soils containing chalk the sedimentation process is slow, and some "active" clay may be adsorbed by the humus. Changes in the colloidal dispersed soil fraction are influenced by the sedimentation period, particularly when hydrochloric acid treatment has been adopted. Samples of soil suspension for weighing are preferably evaporated on a water-bath or coagulated with 10% sulphuric acid. Use of concentrated acid leads to considerable losses in the recorded fractions. Washing of fractions <0.01 mm. in diam. on filter-paper is attended with possible loss of material. The temperature of water used for sedimentation must be controlled with care.

A. G. POLLARD.

Determination of the nutrient requirement of soils. Neubauer's method. E. I. SANOTZKI and A. T. KIRSSANOV (Staatl. Inst. exp. Agron., Abtg. Ackerbau, Leningrad, 1928, No. 13, 23 pp.; Bied. Zentr., 1930, 59, 145).—Neubauer's method gives dependable results only with the heavier types of soil. The mixing of sand with soil produces very different effects on light and on heavy soils. The assimilable phosphate content is closely related to the proportion of water retained in air-dried soil samples.

A. G. POLLARD.

Determination of water-soluble phosphorus compounds in soils. F. TERLIKOWSKI and L. KRÓLIKOWSKI (Rocz. Nauk Roln. Leśn. 1929, 21; Proc. Internat. Soc. Soil Sci., 1930, 5, 28–29).—Numerous comparative analyses of the soluble phosphate content of soils by Spurway's method and that of Wrangell (B., 1926, 841) are recorded.

A. G. POLLARD.

Determination of the phosphate requirement of soils by pot-culture methods. ANON. (Superphosphat, 1930, 6, 25–27).—The limits of error and the significance of experimental values in the correlation with Mitscherlich pot experiments and field results are discussed (cf. Landw. Jahrb., 1928, 67, 457).

A. G. POLLARD.

Results of soil experiments in the Rhine Palatinate especially regarding their root-soluble phosphate contents. M. KLING (Superphosphat, 1930, 6, 39–43).—Analysis of a large number of soils is recorded. Among these the root-soluble phosphate contents (Neubauer) was lowest in pasture soils, higher in arable soils, and highest in vineyard soils. The proportion of root-soluble phosphate in soils below which fertilisers are necessary are, per 100 g. of dry soil: for cereals and pastures 8 mg.; for root crops, clovers, and vines 15 mg.; for tobacco 4 mg. P_2O_5 .

A. G. POLLARD.

Determining soil organic matter by means of hydrogen peroxide and chromic acid. W. T. DEGTJAREV (Soil Sci., 1930, 29, 239–245).—Modifications of Schollenberger's method (B., 1927, 662) are described. The sample used is reduced to 0.2–0.15 g. Standard chromic acid is prepared by heating 16 g. of chromic oxide with 1 litre of concentrated sulphuric acid at 165° for 30 min. The soil is heated with 10 c.c. of the chromic acid solution at 165° in a sulphuric

acid bath for 10 min., cooled, washed into a beaker, and titrated with ferrous ammonium sulphate solution in the presence of diphenylamine. A further oxidation process is described in which the crushed and sieved sample (0.15–0.2 g.) is treated in a Kjeldahl flask with 10–15 c.c. of hydrogen peroxide and to which is added carefully 10–15 c.c. of chromic acid solution. The heat generated by vigorous mixing of the contents of the flask suffices to oxidise the organic matter, and the mixture is washed out and titrated as before.

A. G. POLLARD.

Determination of adsorbed bases in soils containing carbonates. I. KANIWETZ (Rep. Zentrallab. Agric. Kiev, 1928; Proc. Internat. Soc. Soil Sci., 1930, 5, 29–30).—A process for determining adsorbed bases in soils, depending on leaching with water instead of dilute acid, is examined. Continuous leaching with water affects the adsorptive capacity of soils (as determined by N-barium chloride) in a manner varying with the soil type. These changes are independent of temperature, and are mainly controlled by the chalk content of the soil. Changes in p_H values during leaching are small, and vary with the amount of chalk present and the carbon dioxide content of the water used.

A. G. POLLARD.

Determination of p_H of soils by the quinhydrone electrode. P. DE CONINCK (Ann. Soc. Sci. Bruxelles, 1930, 50, 6–13).—Soils of different origin were suspended in water, and the p_H of the liquid extract was determined (a) with the soil in suspension, (b) after decantation, (c) after filtration, (d) after centrifuging. The p_H of the decanted liquid was also measured after filtration and centrifuging. The measurements were made over a period of several days, and although consistent amongst themselves in respect to time, the values differed for the processes (alone) used to prepare the liquid. No explanation of the results is offered. The Breckpot type of quinhydrone electrode was used.

J. O. CUTTER.

Determination of p_H values in soils. L. SMOLIK (Bull. Czechoslov. Acad. Agric., 1929, [v], 192; Proc. Internat. Soc. Soil Sci., 1930, 5, 9–10).—In using the quinhydrone electrode for soil determinations, Veitch's hydrochloric acid-potassium chloride electrode is suitable for field work, although the saturated calomel electrode is preferable for exact laboratory determinations. The gold electrode is less sensitive than platinum. Soil suspensions used for these measurements should be well stirred. After settling, the deposited soil shows a higher p_H value than the suspension. Drying of soils at 100° decreased their p_H values. Ignition decreased the p_H values of acid and neutral soils and increased that of alkaline soils. Autoclave drying of wetted soils decreased their acidity. Changes in p_H values of soils due to drying are smaller in light than in heavy soils.

A. G. POLLARD.

Factors affecting the estimation of lime requirement from p_H values. M. F. MORGAN (Soil Sci., 1930, 29, 163–180).—For a series of soils of similar type the "lime-absorption factor" (lime necessary to produce an increase of p_H 1) may be calculated by means of a series of factors depending on texture and humus content. The lime-absorption factor of a soil may be expressed as $0.119 \times \text{moisture equivalent}$ (Briggs).

A. G. POLLARD.

Determination of minute amounts of iodine in soils and waters. R. L. ANDREW (Analyst, 1930, 55, 269—277).—The iodine was determined by the method of von Fellenberg slightly modified. The prepared dried soil is ignited with potassium carbonate, the residue boiled with water, filtered, the filtrate evaporated to dryness, carefully ignited, cooled, 4 c.c. of 90% alcohol and 3 drops of water are added, and, after 15 min., the mixture is rubbed up, the alcoholic solution decanted, and extraction repeated three times. One drop of saturated potassium carbonate solution and 6 c.c. of water are added to the solution, the whole is evaporated, ignited, and, after cooling, 1 c.c. of 95% alcohol and sufficient water are added to give a pasty mass on stirring. The solution is decanted, extraction repeated, and, after evaporation, the very small residue is ignited; 4 drops of water are then added, the solution is transferred to the iodine extraction tube, and a series of standard tubes prepared. With small amounts (up to 0.001 mg.) of iodine 0.03 c.c. of chloroform is added to each tube, followed by 1—2 drops of sulphuric acid containing nitrite, the tube is shaken and centrifuged, and the depth of colours compared. Not less than 0.0002 mg. of iodine could be detected. In no case was there a recovery of more than 60—70% of the iodine, and the losses are not wholly due to heating. Reducing the amount of potassium carbonate did not improve the extraction. If the amount of iodine extracted is increased by one half, the quantity is a true indication of the amount in the soil. Soils from several districts of North Island of New Zealand had an iodine content of 10—2100 pts. in 10 million and potable waters of 0.0040—0.1200 pt. D. G. HEWER.

Determination of "volume-weight" of soils by means of vaseline. K. S. KIRITSCHENKO (Rep. Cuban Agric. Inst., 1929, 6; Proc. Internat. Soc. Soil Sci., 1930, 5, 35—36).—Vaseline is used to fill the pore-space of soils in determining their volume-weight.

A. G. POLLARD.

"Kalkammonsalpeter." K. NEHRING (Z. Pflanz. Düng., 1930, 9B, 120—125).—No nitrogen losses occurred during the storage of "Kalkammonsalpeter," although unless kept dry the fertiliser tended to cake and become difficult to distribute. In soil, "Kalkammonsalpeter" reacted as physiologically neutral, and in pot cultures its nitrogen was as efficient as that of sodium nitrate. In field trials with soils exhibiting much exchange acidity the nitrogen of "Kalkammonsalpeter" was not utilised by acid-sensitive crops, e.g., turnips.

A. G. POLLARD.

Superphosphate mixtures [fertilisers]. KLEBERGER (Superphosphat, 1930, 6, 21—22).—Field trials are quoted in a discussion of the value of "ammonium superphosphate" as compared with equivalent mixtures of simple fertilisers for the growth of barley and other cereals.

A. G. POLLARD.

Content and solubility of potash and phosphates in surface and sub-soils of various types. J. HASENBÄUMER and R. BALKS (Z. Pflanz. Düng., 1930, 9B, 97—109).—In light and medium soils and subsoils the phosphate and potash contents (citric-soluble, root-soluble, and soluble in 10% hydrochloric acid) varied

directly with the proportion of clay (<0.01 mm. in diam.). The proportion of potash soluble in 10% hydrochloric acid averaged 0.93 pt. per 100 pts. of clay in surface soils and 0.87 in subsoils. The amount of potash soluble in the acid increased with the calcium content of the soil, but not directly; with higher lime contents the relative increase in soluble potassium declined. No relationship was apparent between the reaction or chalk content of a soil and the "relative solubility" or citric-solubility of phosphates or the root-solubility of the potash. In soils of similar potash content the root-solubility and "relative solubility" of the potash was greater in the surface than in the subsoil. Similar relationships exist with the phosphates, but differences in the phosphate content of surface and subsoils are much less marked. On heavy soils the proportionality between nutrient and clay contents is irregular, notably in cases where the subsoil has a greater proportion of clay than the surface soil.

A. G. POLLARD.

Solubility of phosphoric acid compounds in certain soils. S. M. ANTONOV (Siberian Inst. Agric. & Forestry, 1928, 10; Proc. Internat. Soc. Soil Sci., 1930, 5, 27—28).—The effect of lime and of sulphuric acid on the soluble phosphate content of black earth and saline soils is examined. Lime increased the water-soluble phosphate content to an extent paralleled by increases in alkalinity. The presence of normal calcium phosphate in these soils is unlikely. Changes in phosphate solubility following acid treatment do not point to the presence of iron or aluminium phosphates. The water-soluble phosphate contents of saline soils was markedly greater than that of black earths. Liming increased the nitrate contents and crop yields of saline soils.

A. G. POLLARD.

Distribution of assimilable phosphorus and potash in arable soils. T. ROEMER (Superphosphat, 1930, 6, 14—20).—There is a general parallelism, but no strict proportionality, between the results of Neubauer trials and Dirks' method for determining the assimilable nutrients in soil. The latter method is utilised for determining the amounts of assimilable potash and phosphate at various soil depths. Below the cultivated surface layer the phosphate content declines sharply. Phosphatic fertilisers affect the surface soil only; below plough depths differences in the assimilable phosphate contents are small. Extensive root development of deep-rooting plants occurs in soil layers poor in nutrients. Distribution curves of fertilisers in various soil layers after the customary cultural operations are recorded.

A. G. POLLARD.

Rapid method of determining the effect of phosphate fertilisers on the yield of crops. A. NĚMEC (Compt. rend., 1930, 190, 1071—1073).—Addition of phosphate to soils markedly deficient in phosphorus does not cause an increase in the yield of the crops grown in them when the content of ferric oxide exceeds 50 mg. per kg. The soils do not utilise the added phosphate. The iron oxide content is determined colorimetrically by treating the 1% citric acid extract of the soil with 10% hydrochloric acid and a few drops of potassium permanganate solution, diluting, and adding

potassium thiocyanate. The colour is matched with a standard prepared from ferric chloride. Determination of the water-soluble silica also shows the effect of the added phosphate (cf. B., 1928, 420). Combination of the two methods gives a rapid method for determining the phosphoric acid requirement of the soil and the probable effect of added phosphate on the yield of crops.

H. BURTON.

Determination of the manurial requirement of soils. G. BARBIER (Ann. Sci. Agron., 1929, 46, 292; Proc. Internat. Soc. Soil Sci., 1930, 5, 61–62).—A critical discussion of existing methods.

A. G. POLLARD.

Use of stall manure. L. L. BALASCHEV (Mitt. staatl. Inst. exp. Agron., Leningrad, Ackerbauabt., No. 21, 36 pp.; Bied. Zentr., 1930, 59, 155–156).—The analysis and efficiency of various types of stall manures is recorded. Horse manure is more effective than cattle manure, whether fresh or rotted. The use of peat litter is advantageous. Manuring with straw, spread in spring and ploughed in at midsummer, gave with the first crop positive results in some cases and negative in others. In all cases subsequent crops benefited. The risk of denitrification is small.

A. G. POLLARD.

Phosphate content of soil and phosphate manuring. O. ENGELS (Superphosphat, 1930, 6, 22–24).—A general review of the phosphate question in Germany.

A. G. POLLARD.

Effect of lime on the root solubility of phosphates and potash in arable soils. M. GRAČANIN and A. NĚMEC (Z. Pflanz. Düng., 1930, 9B, 126–131).—In a number of soil types examined no relationship existed between the root-soluble contents of potash and phosphate and the proportion of calcium soluble in 10% hydrochloric acid. By the addition of increasing amounts of lime the assimilability of potash and phosphates was shown to increase within the range p_H 4.4–6.9. This effect was relatively greater in soils which were naturally poor in root-soluble nutrients. Addition of chalk to soils already containing 5–13% CaO and with p_H 7.4–8.3 reduced the root-soluble phosphate content. Similar but less well-defined results were obtained for potash, but the liming of soil rich in root-soluble potash tended to reduce potash assimilation by plants.

A. G. POLLARD.

Fixation and mobilisation of phosphoric acid in different layers of chernozem. A. A. BYTCHIKHNE (Ann. Sci. Agron., 1929, 46, 575; Proc. Internat. Soc. Soil Sci., 1930, 5, 27).—Differences in the fertility of chernozem surface and subsoils are mainly due to differences in the easily assimilable phosphate contents. Towards the end of a fallow season there is a relative decrease in the proportion of easily assimilable phosphate present, especially in the subsoil. The fixation (immobilisation) of phosphate under these conditions is ascribed to the micro-organisms, which multiply considerably during the fallow period and utilise the soluble phosphate.

A. G. POLLARD.

Action of carbon dioxide in causing dissolution of soil phosphate. A. T. SCHLÖESING and D. LEROUX (Compt. rend., 1930, 190, 989–991).—The action of moist air containing 4.4% CO_2 on a soil poor in lime

could not be differentiated, by the determination of soluble phosphate, from that of air containing less than 0.004% CO_2 , even at the end of 2 years. The production of soluble phosphate in calcareous soils is small, and is increased only slightly by the presence of carbon dioxide in the water used for extraction. In the case of acid soils the amount of soluble phosphate is considerably greater, and is again slightly increased by the presence of carbon dioxide.

T. H. MORTON.

Principles of liming [of soils]. A. KIRSSANOV (Mitt. staatl. Inst. exp. Agron., Leningrad, 1929, No. 1, 60 pp.; Bied. Zentr., 1930, 59, 150–151).—The lime requirement of a number of podsol soils was determined by the litmus test of the potassium chloride extract, by Mitscherlich method, and by examination of the growth of clover. On soils of this type good crop yields can only be obtained by the use of lime and fertilisers.

A. G. POLLARD.

Capacity of different types of peat soils to absorb ammoniacal nitrogen. P. TUORILA (Wiss. Veröff. Finn. Moorkulturvereins, 1929, No. 9; Proc. Internat. Soc. Soil Sci., 1930, 5, 66–67).—Examination of aqueous ammoniacal solutions shows that within the range p_H 9.5–12 changes in the concentration of undissociated ammonium molecules and in the rate of evaporation of ammonia are small. From p_H 9.5 to 5.0 variations in these values are approximately inversely proportional to the hydrogen-ion concentration. The fixation of nitrogen by peat is largely a function of its acidity. The ammonia-fixing power of peat may be calculated from its "lime condition." Treatment with the amount of ammonia so calculated brings the reaction of the peat to p_H 6.5–6.8. Losses of ammonia from peat-water mixtures with $p_H < 6.5$ are insignificant, but above p_H 6.5 the evaporation of ammonia is considerable. Peats of various types have very different ammonia-fixing powers, and these bear no relationship to the amount of humus material present. Ammonia is a more efficient neutralising agent for the acid substances in peat than is calcium carbonate. The use of peat for preventing losses of ammonia from liquid manure is discussed. The covering of manure heaps with a thick compact layer of peat is recommended.

A. G. POLLARD.

Influence of moisture on nitrification, and the mobilisation and immobilisation of phosphoric acid and calcium in soil. P. HIRKO (Pub. Centrallab. Agric. Chem., Kiev, 1928; Proc. Internat. Soc. Soil Sci., 1930, 5, 69).—Nitrification in soils increased with their moisture contents up to 60% of their maximum capacities, but decreased at 80% of this value. Both in manured and unmanured soils, increased moisture contents corresponded with a reduction in the amounts of phosphate soluble in water and in 2% acetic acid, and an increase in the water-soluble calcium content. Under the conditions of the experiment there was a definite relationship between the proportions of phosphoric acid and calcium.

A. G. POLLARD.

Nitrogen manuring of fish-ponds. KUHNERT (Z. Pflanz. Düng., 1930, 9B, 109–111).—A reply to Walter and Nolte (B., 1930, 162).

A. G. POLLARD.

Plant-food requirement of rice. W. F. GERCKE

(Soil Sci., 1930, 29, 207—225).—The nutrient requirement of rice cannot be determined by growth to maturity in complete nutrient solutions. Under these conditions the plants absorb more mineral matter than is utilised for growth purposes. Relationships between crop yield and the absorption of a particular element depend on the total quantity of that element absorbed, the period during which absorption takes place, and the time required for the element to be utilised by the plant. Plants grown to maturity in a complete nutrient absorbed more calcium, magnesium, phosphorus, and sulphur than when partly grown in a complete nutrient and subsequently transferred to a solution deficient in these elements. Factors of primary importance to the growth of rice are those affecting the absorption and utilisation of phosphorus, nitrogen, and iron. The concentration and relative proportions of other essential elements in the nutrient solution are of lesser significance, provided they do not affect the availability of the former group. Of the elements present in the seed, iron is the first to become a limiting factor in growth. The work of Espino (Phil. Mag., 1920, 16, 455) is adversely criticised. A. G. POLLARD.

Top-dressing of potatoes with lime. F. MÜNTER (Z. Pflanz. Düng., 1930, 9B, 111—120).—Quicklime, applied as a top-dressing to potatoes, has no injurious effect even when, in wet weather, some scorching of the leaves occurs. On neutral soils in dry areas no advantage is obtained by top-dressing with lime. On soils of $pH < 5.5$ lime is necessary and is preferably applied before planting the seed tubers. Light dressings of lime on sugar beet and potatoes may be applied a considerable period after sowing without risk of injury. In some cases the starch content of potatoes is increased by liming. A. G. POLLARD.

Pasture management. Seasonal composition of certain South African pasture grasses in relation to their manuring and intensity of grazing. R. R. STAPLES and A. J. TAYLOR (S. Afr. J. Sci., 1929, 26, 139—153; Union of S. Afr., Dept. Agric., 1929, Div. Chem. No. 104).—The crude protein content of frequently-cut grass was much greater than that of the matured herbage, but proportionally less than is recorded in Europe. The phosphate content of fortnightly-cut grass was five times that of mature pasture, but the calcium content remained practically unaltered. Application of complete fertilisers did not appreciably affect the protein or calcium content of frequently-cut herbage, but the phosphate content and total yield were markedly increased. Frequent cutting reduced the total annual yield of dry matter, and certain of the natural herbage plants of the veldt were practically eliminated in the process. A. G. POLLARD.

Phosphorus fertilisation of meadow land. II. E. TRUNINGER (Landw. Jahrb. Schweiz, 1929, 43, 653—698; Chem. Zentr., 1929, ii, 3177).—Fertilisation for 10 yrs. with 90 kg. P_2O_5 per hectare annually did not produce phosphate saturation in the top layer. Various types of phosphorus fertiliser were compared; application of superphosphate to acid soil is not recommended. A. A. ELDRIDGE.

Effect of *Indigofera endecaphylla* on the nitrogen

and organic matter contents and the mechanical constitution of tea soils at Peradeniya. A. W. R. JOACHIM (Trop. Agriculturist, 1930, 74, 137—140).—Growth of a cover crop of *Indigofera endecaphylla* on tea soils prevented loss of the finer soil particles by erosion and considerably increased the soil content of nitrogen and organic matter. A. G. POLLARD.

Plant metabolism studies as an aid in determining fertiliser requirements. H. R. KRAYBILL (Ind. Eng. Chem., 1930, 22, 275—276; cf. Hepler and Kraybill, N.H. Agric. Exp. Sta. Tech. Bull. 28, 1925).—A preliminary paper, in which the value of a knowledge of the effects of the essential nutrients on plant metabolism is emphasised. A. G. POLLARD.

Value of results of local fertiliser trials in Sweden. G. SUNDELIN (Nord. Jordbrugsforsk., 1928, 102—119; Bied. Zentr., 1930, 59, 159—161).—The customary 8-plot fertiliser trials are discussed. One-year trials are of little value, and repeated trials are still liable to considerable error. A. G. POLLARD.

Determination of total nitrogen of plant extracts in presence of nitrates. G. W. PUCHER, C. S. LEAVENWORTH, and H. B. VICKERY (Ind. Eng. Chem. [Anal.], 1930, 2, 191—193).—Olsen's method (B., 1928, 101) for the determination of the nitrogen content of soils has been modified to apply directly to aqueous solutions. A suitable volume of the extract is mixed with 20—40 c.c. of water in a 700-c.c. Kjeldahl flask; 10 c.c. of sulphuric acid (1 : 1) and 3 g. of reduced iron powder are added, and the mixture is shaken for 10 min. It is then heated and boiled for 5 min., cooled, and 30 c.c. of concentrated sulphuric acid, a drop of mercury, a few angular quartz pebbles, and 5 g. of anhydrous sodium sulphate are added. The contents are digested until the acid layer is clear and the precipitate assumes a yellow colour. After heating for a further 1—2 hrs., a few crystals of potassium permanganate are added, the mixture is cooled, diluted with 300 c.c. of water, and 3—5 g. of sodium thiosulphate are introduced, followed by a small piece of paraffin, an excess of sodium hydroxide, and a little zinc. The ammonia is then distilled into acid in the usual manner. Ranker's observation (B., 1927, 536) that the salicylic acid-zinc method gives inaccurate results when applied to aqueous extracts of plant tissues containing nitrates has been confirmed. E. H. SHARPLES.

Soil researches on sugar plantations. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, [15]; Proc. Internat. Soc. Soil Sci., 1930, 5, 25—26).—Chemical analyses of plantation soils are recorded and the planning of field trials and evaluation of results are discussed. A. G. POLLARD.

The phosphate question in sugar-cane culture in Java. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1929, [3], 77—96; Proc. Internat. Soc. Soil Sci., 1930, 5, 24—25).—Soils are classified according to their contents of phosphate soluble in 2% citric acid solution. Results agree closely with field trials. In sand cultures optimum growth of sugar cane is obtained with nutrient solutions containing 2—9 mg. P_2O_5 per litre. The intake of phosphate by sugar cane is practically constant

throughout the whole growth period. (Cf. B., 1929, 756.) A. G. POLLARD.

Cylinder for separating fine soil particles by decantation. L. SMOLIK (Bull. Czechoslov. Acad. Agric., 1929, [iv], 470; Proc. Internat. Soc. Soil Sci., 1930, 5, 32).

Studies of roots in plantations and in alkaline soils. P. MAGYAR (Recherches Forestières, Sopron, 1929, 31, [2]; Proc. Internat. Soc. Soil Sci., 1930, 5, 56).

See also A., May, 565, **Detection of nickel in plants** (MARTINI).

PATENTS.

Insecticides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,803, 19.12.28).—A thioether, e.g., thio-*m*-cresol benzyl ether, or an ether (anisole, dibenzyl ether, diphenyl ether, etc.) is intimately mixed with cyclohexanone and tetrahydronaphthalene, with or without addition of another insecticide or of a powdered filler (talcum). The mixtures are effective against chicken lice. C. HOLLINS.

Production of potassium ammonium sulphate [mixed fertiliser]. R. GRIESSBACH and O. SCHLIEPHAKE, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,358, 15.4.30. Appl., 23.8.28. Ger., 16.9.27).—See B.P. 300,402; B., 1929, 68.

XVII.—SUGARS; STARCHES; GUMS.

Physical and chemical properties of sugar in affination and decolorisation. W. KONN (Z. Zuckerind. Czechoslov., 1930, 54, 173—186).—On washing raw beet-sugar with water (up to 30% by wt.), the colour, luminescence, and electrical conductivity all decrease, and crystals are obtained a normal solution of which has a colour equal to 1.17° Stammer and contains 0.5% of ash. When a 40% solution of pure sugar is used (up to 100% by wt. of the sugar treated), crystals are obtained which in their physical and chemical properties almost correspond to Czechoslovakian domestic sugar. In decolorising washed sugar with bone-char the best effect is obtained with 2%, whereby 56% of the colour is removed, the luminescence is diminished from 26 to 12 units, and the carbonate ash is reduced to 23% of its original value. Using kieselguhr, Norit, and Carboraffin, the conductivity remains in each case unaltered, but the colour and luminescence both fall.

J. P. OGILVIE.

Decrease of the salts in carbonatation [of sugar juice] as observed by electrical conductivity measurements. K. ŠANDERA and V. PREININGER (Z. Zuckerind. Czechoslov., 1930, 54, 247—253).—By plotting curves for values of alkalinity and electrical conductivity, the influence of certain salts on the course of carbonatation was determined. It was shown that the chloride, sulphate, phosphate, hydroxide, oxalate, citrate, aspartate, and glutamate of potassium and the sulphates of calcium and magnesium, or their corresponding ions, are eliminated from solution in agreement with previous results established by means of analytical methods. It was possible not only to follow these effects during the course of carbonatation from an initial alkalinity of 0.36% down to neutrality, but the authors were also able to demonstrate that each anion

forming insoluble calcium salts acts moreover in a very characteristic manner, either in exercising a secondary effect on the formation of buffer mixtures, or else in modifying the adsorbing power of the precipitate.

J. P. OGILVIE.

Sample dryer. HOPPER.—See I. Decolorising power of active carbon. EDELSTEIN.—See II. Soils of sugar plantations. Phosphates and sugar-cane culture. ARRHENIUS. Sugar cane [growth].—PARDO.—See XVI.

See also A., May, 581, **Determination of sugars in solution** (MEIJER). 583, **Digoxin—a new glucoside** (SMITH). **Influence of neutral salts on hydrolysis of starch** (ANGELESCU and MANOLESCU).

PATENT.

Production of glutaminic acid [in sugar factories]. K. BROMIG, ASSR. to DEUTS. GOLD- u. SILBERSCHEIDENANSTALT VORM. ROESSLER (U.S.P. 1,755,683, 22.4.30. Appl., 8.3.27. Ger., 22.3.26).—See B.P. 320,589; B., 1930, 77.

XVIII.—FERMENTATION INDUSTRIES.

Industrial dehydration of alcohol: production of water-free alcohol for motor fuels etc. H. GUINOT (Internat. Sugar J., 1930, 32, 77—82).—Modern methods (here outlined) for the production of absolute alcohol by taking advantage of the formation of an azeotropic mixture with benzene, will involve, starting from rectified alcohol at 94% (by wt.), a maximum expenditure of 2000 lb. of dry steam to make 100 gals. of alcohol at 100%. This steam consumption is reduced to 1350 lb. if one is content with alcohol at 99.7%, whilst, starting from alcohol at 92%, the steam consumption is about 1600 lb. to obtain alcohol at 99.7%. Hydrocarbon consumption is said always to remain less than 0.2% of the production of hydrated alcohol.

J. P. OGILVIE.

Alcoholometric corrections for temperatures below 0°. F. BORDAS and E. ROELENIS (Compt. rend., 1930, 190, 923).—It is proposed to determine corrections of alcoholometric tables between 0° and —30° by measurements made with a cylindrical vessel containing 300 c.c. of alcoholic liquid, placed in a large vessel containing acetone cooled by solid carbon dioxide, the whole being placed in a heat-insulated vessel. No results are given. C. A. SILBERRAD.

Recovery of oils and fats. BECKMAN.—See XII.

See also A., May, 640, **Action of amylase from *Sorghum vulgare* on potato starch** (PATWARDHAN). **Amylase from *Eleusine Coracana*** (PATWARDHAN and NARAYANA). 642, **Survival of dried yeast** (KRASSILNIKOV). **Nitrates as source of nitrogen for growth of yeast** (PIRSCHLE).

PATENTS.

Manufacture of yeast. "SELBI" (SOC. D'EXPLOIT. DE LICENCES DE BREV. IND.) (B.P. 304,314, 7.1.29. Fr., 19.1.28).—Vinasses from the distilleries of molasses, sugar factories, or yeast factories are used as the sole medium for the culture of alimentary or any other yeast. The pitching yeast may be previously cultivated in sterilised vinasses with added ammonium sulphate and

sodium phosphate, and thereafter used to ferment the vinasses with added superphosphate. C. RANKEN.

Filtration of beer wort for further treatment, and of beer and other foaming liquids. L. NATHAN, Assr. to HANSEN A.-G. (U.S.P. 1,754,432, 15.4.30. Appl., 6.9.27. Ger., 7.9.26).—See B.P. 280,395; B., 1928, 104.

Improvement of brewing waters. E. JALOWETZ (U.S.P. 1,755,544, 22.4.30. Appl., 18.8.26. Austr., 20.11.25).—See B.P. 261,708; B., 1927, 430.

Dry distillation of vinasses under reduced pressure. J. GUILLISSEN, Assr. to UNION CHIM. BELGE, Soc. ANON. (U.S.P. 1,756,191, 29.4.30. Appl., 8.11.23. Belg., 15.12.22).—See B.P. 208,516; B., 1924, 802.

Feeding-stuffs etc. (Austr.P. 107,279—107,281).—See XIX.

XIX.—FOODS.

Chemical and physico-chemical changes induced in wheat and wheat products by elevated temperatures. II. W. F. GEDDES (Canad. J. Res., 1930, 2, 65—90; cf. B. 1930, 262).—A detailed examination of the influence of time and temperature of heating samples of unbleached flour, milled from Western Canadian, hard red, spring wheat, on biochemical properties related to "strength," showed that for the most part the changes investigated were associated with decreasing baking quality. Gluten quality was impaired in all heat-treated samples. High positive correlations were obtained between viscosity, gas retention, and loaf volume; decrease in viscosity was approximately a linear function of the temperature for constant time of heating. Base of peptisation of flour proteins showed a progressive decrease with increased heating. High positive correlations were obtained between loaf volume and percentage of protein peptised. Marked decrease in diastatic activity followed severe heat-treatment, but decrease in proteolytic activity was first evident. A full bibliography is appended.

E. HOLMES.

Chemical and physico-chemical changes induced in wheat and wheat products by elevated temperatures. III. Influence of germ constituents on baking quality and their relation to improvement in flour induced by heat and chemical improvers. W. F. GEDDES (Canad. J. Res., 1930, 2, 195—213; cf. preceding abstract).—A detailed investigation to determine the cause of improved baking qualities observed after different heat-treatments of various grades of flour and on added germ is described. The main conclusions from the results (for details of which the original must be consulted) are as follows. Heat-treatment of straight-grade flour either matured with nitrogen trichloride, or previously extracted with ether, or of unaged fifth-middlings flour results in very little improvement in baking quality and very little response to the bromate method (addition of 0.001% of potassium bromate to the baking formula). Addition of germ to fifth-middlings flour causes a marked reduction in baking quality (poorer handling qualities of the dough, underfermented characteristics, decrease in loaf volume, and coarse, open texture) by the basic procedure, but the deleterious effects of germ are reduced by increase in the fermenta-

tion time, addition of bromate, or previous heat-treatment of the germ. The response to bromate decreases with increasing preheating treatment, and it is postulated that the improvement of natural flour induced by proper heat-treatment and the response to bromate are both associated with the presence of germ in the flour, and depend on the oxidation of certain germ constituents, probably the phosphatides. In agreement with this view addition of lecithin to middlings flour causes a marked response to bromate treatment, and heat-treatment of the germ induces a marked increase in hydrogen-ion concentration of the aqueous extract (p_H 6.51 for raw germ changing to p_H 5.93 after heating at 121° for 3 hrs.), and a decrease in the iodine value ([Wijs] 125.6 and 111.5°, respectively) of the residue from the ether extract. Heat-treatment is therefore detrimental to the gluten, but markedly decreases the deleterious effects of germ constituents; hence unaged flours containing low-grade mill-streams may show considerable improvement in baking quality after heat-treatment despite the injury to gluten quality, but the improvement will not equal that induced by chemical improvers which act primarily on the germ constituents. The suggestions of Kent-Jones (B., 1929, 463) are shown to be untenable both from theoretical and experimental considerations.

J. W. BAKER.

Relation between protein content and quality of wheat, as shown by different baking methods. R. K. LARMOUR (Cereal Chem., 1930, 7, 35—48).—Experimental milling and baking tests were carried out on 286 samples of pure varieties of wheat grown in Saskatchewan during 1926—8. Results showed that when the flour was baked by a simple formula of flour, water, salt, yeast, and sugar, the correlation factor between the protein content and the quality, as measured by baking tests, was too low to justify the practical value of the crude protein test in predicting quality. When, however, 0.001% of potassium bromate was added to the basic baking formula the correlation with protein content was high enough to warrant the commercial use of the protein test as a factor in the classification of hard spring wheat.

E. B. HUGHES.

Evaluating the quality of wheat varieties by co-operative tests. C. O. SWANSON (Cereal Chem., 1930, 7, 66—78).—Co-operative work was carried out by seventeen cereal chemists on Tenmarq, Kanred, Turkey, Blackhull, and Superhard wheats to determine the milling and baking qualities of Tenmarq, a new hybrid wheat developed by the Kansas Agricultural Experiment Station. Details of the methods used and the results of the tests are given. "Tenmarq" is shown to be superior to the other varieties tested.

E. B. HUGHES.

Bacteriology of wheat and flour. D. W. KENT-JONES and A. J. AMOS (Analyst, 1930, 55, 248—268).—The enumeration of blood-heat organisms in flour is made by transferring 10 g. of flour to a sterilised, wide-mouthed glass bottle containing 100 c.c. of a sterile 0.5% solution of sodium chloride and 10 g. of purified, ignited sand. The mixture is shaken for 2 min. and, as soon as the bulk of sand has settled, 5 c.c. of the suspension are transferred to a narrow-necked bottle containing 45 c.c. of sterile 0.5% sodium chloride solution. After shaking, 5 c.c. of the liquid are transferred to

another similar bottle containing 45 c.c. of sodium chloride solution. Finally 1 c.c. is transferred to a Petri dish to which are added the contents of a 10-c.c. tube of sterile, neutral agar melted in boiling water and cooled to 45°. The agar and extract are mixed and, when the agar has solidified, the inverted dish is incubated at 37° and a count taken in 48 hrs. Cool organisms are enumerated in the same way except that a nutrient gelatin plate is prepared from the final dilution and counts are made in 48—72 hrs. In each case the count multiplied by 1000 is regarded as giving the number of organisms per g. of flour. Counts made on samples of flours taken over a period of several weeks showed that contamination of flour with both blood-heat and cool organisms increases greatly as the grade of flour becomes lower. Lengthening of conditioning time, omission of washing, etc. also increase the number, and always cool organisms were more numerous than blood-heat ones. It is considered that patent flour at the time of milling should contain not more than 20,000 blood-heat organisms per g. and the straight-run flour not more than 50,000. Under normal conditions of storage both types of organism decrease considerably, and moisture content is a more important influencing factor than air temperature. In all cases blood-heat organisms die off more quickly than the cool ones. Most normal patent flours gave a negative result for the *B. coli* test with 0.025 g. and a positive one with 0.05 g., but results did not follow the bacterial content. Practically all bread made in this country contains "rope" organisms, but is rarely stored under conditions sufficiently favourable for its rapid growth. Bacterial counts were also made on wheats, and washing and brushing processes were found to reduce the bacterial content about 60%.

D. G. HEWER.

Buffer intensities of water extracts and suspensions of various flours at different H-ion concentrations. G. E. HOLM and E. GREWE (Cereal Chem., 1930, 7, 49—58).—The hydrogen-ion titration curves of water extracts and suspensions of various grades of flour from hard and soft wheats are compared. By plotting against p_H the equivalent of acid or base required to change the p_H value by one unit, curves are obtained which show the variation of buffer intensity with p_H . The relation of buffer intensity to grade, ash content, and phosphorus content is discussed.

E. B. HUGHES.

Staling of bread. W. PLATT (Cereal Chem., 1930, 7, 1—34).—The changes taking place during the staling of bread are divided into three sections: (a) loss of volatile constituents, including water, alcohol, aldehydes, carbon dioxide, and traces of organic compounds; (b) the changes due to oxidation, considered to be of minor importance, and (c) the "inherent" staling, a term covering the complex physical and chemical changes occurring within the loaf. An historical survey with full references is given and a method of ascertaining the degree of staling by measurement of the compressibility of the crumb is described.

E. B. HUGHES.

Value of the viscosimeter in a commercial flour-mill laboratory. A. R. SASSE and J. T. PEARSON (Cereal Chem., 1930, 7, 79—82).—An account of the application of the MacMichael viscosimeter to measurement of

the viscosity of acidified (lactic acid) flour-water suspensions. The viscosity due to the starch was found to be negligible, the important factors being the amount and quality of the protein and amount of ash. The effect of variation in protein content was eliminated by adjusting the concentration of the suspension so as to keep the protein content constant. The authors conclude that the use of the viscosimeter is not sufficiently standardised to give results of use other than in the particular laboratory where obtained, and that viscosity tests add nothing to the information obtainable from the usual protein and ash tests.

E. B. HUGHES.

New aids in the ashing of flour. F. M. WALTERS (Cereal Chem., 1930, 7, 83—87).—Addition of oxides of lanthanum, yttrium, cerium, and thorium accelerates the combustion of flours, and the time may be reduced from 5 hrs. to 25—30 min. The flour is made into a paste with a solution of lanthanum nitrate giving a known weight of the oxide on incineration, and this is subtracted from the final ash. Duplicate results are obtained, but they do not agree with those obtained using the official A.O.A.C. method. The hygroscopicity of the ash is not altered by the addition of the oxides.

E. B. HUGHES.

Determination of ash [of flour] by direct weighing. J. L. SPALDING (Cereal Chem., 1930, 7, 88—91).—This method, in which the flour is incinerated in an unweighed crucible and the ash knocked out on to the balance pan and weighed, is compared with the official A.O.A.C. method, and the accuracy of the former is found to be the greater. The maximum variation in duplicates by direct weighing is 0.003%, and by the A.O.A.C. method 0.010%. The greater variation in the A.O.A.C. method is due to moisture absorbed by the crucible.

E. B. HUGHES.

Quick ash determination [in flour] by magnesium acetate-alcohol method. J. L. SPALDING (Cereal Chem., 1930, 7, 93—97).—The sample of flour is moistened with an alcoholic solution of magnesium acetate and incinerated at a dull red heat in the presence of oxygen, ashing being complete in 15—20 min. An empty crucible and a crucible containing flour of known ash are incinerated at the same time for control purposes. Results agree well with those obtained by using the standard A.O.A.C. method.

E. B. HUGHES.

Metallic discoloration of Cheddar cheese. E. G. HOOD and A. H. WHITE (Sci. Agric., 1930, 10, 520—522).—Brownish discolorations in a cheese consignment were found to contain ferrous iron, and were traced to the presence of small particles of "steel wool" used in scouring the vats.

A. G. POLLARD.

Sample dryer. HOPPER.—See I. **Recovery of oils and fats.** BECKMAN.—See XII. **Caffeine in tea.** VON MIKO.—See XX.

PATENTS.

Improving the usefulness of feeding-stuffs. E. MISLIN (Austr.P. 107,279, 2.4.17. Addn. to Austr.P. 103,891; cf. U.S.P. 1,685,004; B., 1928, 912).—In the process described in the prior patent, the previously prepared material is heated with alkalis or acids at about 100° before sterilisation, then neutralised and treated with bacteria or yeasts. Only a portion

of the material may be treated with the organism, the remainder being later mixed intimately with the fermented product and the mixture allowed to undergo after-fermentation. W. J. BOYD.

Decomposition and improvement of feeding-stuffs and foods. E. MISLIN (Austr.P. 107,280—1, 2.5.19).—(A) Material of animal or vegetable origin is treated with micro-organisms from pentosan culture (Austr.P. 103,891; cf. preceding abstract) and then gradually warmed to 40—50°. (B) Feeding-stuffs obtained as described in the prior patent are soaked with vinasse, gradually mixed with bran, straw, etc., and kept at above 38° for some time. W. J. BOYD.

Souring of sap-containing vegetable matter in silos in preparation of a preserved feeding-stuff. GÄRTNER & AURICH (G.P. 447,063, 28.10.20).—Urea or an equivalent amount of a nitrogenous salt is added to the material as it is deposited, in such proportion that it serves as food for the lactic acid bacteria with economy of the plant proteins, until sufficient lactic acid has been produced to arrest further bacterial action. In this process an electric current may be passed through the mass and pressure may be applied. W. J. BOYD.

Production of soluble albumin. E. M. MEYER (U.S.P. 1,754,521, 15.4.30. Appl., 20.10.25).—See B.P. 260,224; B., 1927, 922.

Extraction of albumino-caseins of vegetable origin, and separation of such albumino-caseins from amylaceous matter. H. BEAUFOR (U.S.P. 1,755,531, 22.4.30. Appl., 11.10.26. Fr., 23.10.25).—See B.P. 260,242; B., 1927, 539.

Manufacture of a vegetable alimentary extract. J. PERINO (U.S.P. 1,754,434, 15.4.30. Appl., 18.10.27. Ger., 22.10.26).—See B.P. 279,487; B., 1929, 263.

Freezing of fish [by means of cooled brine spray]. STERILEX, LTD., and A. E. SHERMAN (B.P. 328,396, 19.3.29).

Concentration of liquids (Austr.P. 107,317).—See I.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Acid-base equilibrium of tincture of digitalis. J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1930, 19, 366—370).—Experiments on buffering the menstruum and on the buffer capacity of tincture of digitalis, and the effect of ageing on the p_H of the tincture indicate that the extractive material of the digitalis leaves possess sufficient buffer capacity to bring the p_H of each of the finished tinctures close to the p_H of the unbuffered tincture; the buffer influence of the tincture is apparently dependent on the organic extractive material from the drug. A very slow decrease in p_H occurs on keeping; one sample decreased from 5.88 to 5.38 after being kept for 2 years, but the change was more rapid in certain samples stored in direct light. The Van Slyke " β " for the tincture has been determined for strong acids and bases. E. H. SHARPLES.

Stability of infusion of digitalis, B.P. F. WOKES and G. K. ELPHICK (Quart. J. Pharm., 1930, 3, 73—75).—Infusion of digitalis, B.P., preserved by the addition of chloroform (0.3%) or thymol (0.064%), does not

decrease appreciably in physiological activity for at least 3 weeks (cf. Haag and Hatcher, B., 1929, 835).

H. E. F. NOTTON.

Stability of salts of ergotoxine and ergotamine. F. WOKES and G. K. ELPHICK (Quart. J. Pharm., 1930, 3, 59—72).—In continuation of previous work (B., 1929, 1031), the stability of solid and liquid preparations of ergot alkaloids has been examined. Ergotoxine phosphate in a vacuum over phosphoric oxide loses its specific physiological activity at the rate of about 10% yearly. In presence of air decomposition is more rapid. The deterioration, if any, of ergotoxine ethanesulphonate in air-filled closed tubes is less than 5% yearly. The phosphate in 0.01% solution at 0° loses half its activity in 6 months, and in 0.1% solution loses one third of its activity in 2—4 months. At 37° decomposition is several times more rapid. Commercial solutions of the phosphate and of ergotamine tartrate deteriorate at similar rates. H. E. F. NOTTON.

Manufacture of guaiacol and phenacetin. J. SCHWYZER (Pharm. Ztg., 1930, 75, 495—498, 509—510, 518—521).—Methods and apparatus are described for the manufacture of phosgene and methyl and ethyl chlorides; for the conversion of phenol into *o*- and *p*-nitrophenols, *p*-phenetidine, phenacetin, *o*-anisidine, guaiacol, guaiacol carbonate, benzoate, and orthophosphate, and potassium guaiacolsulphonate, and for the conversion of chlorobenzene into *o*- and *p*-chloronitrobenzenes, *o*-nitroanisole, and *p*-nitrophenetole.

H. E. F. NOTTON.

Evaluation of drugs containing caffeine. I. Tea. G. VON MUKÓ (Magyar Gyóg. Társas. Ert., 1929, 5, 384—399; Chem. Zentr., 1929, ii, 3045—3046).—A rapid macro-method, and micro-methods (refractometric and micro-Kjeldahl) for the determination of caffeine in tea are described. A. A. ELDRIDGE.

Technical preparation of tartar emetic. F. CHEMNITZ (Chem.-Ztg., 1930, 54, 214).—The manufacture by the interaction of a boiling solution of commercial tartar and excess of freshly melted and ground antimony oxide is described. After 2 hrs.' boiling the solution is filtered and crystallised on threads in leaden tanks. Vessels of wood or lead are used throughout the process. E. LEWKOWITSCH.

Stability of solutions of arsenious and mercuric iodide, U.S.P. X. W. J. HUSA and W. W. F. ENZ (J. Amer. Pharm. Assoc., 1930, 19, 328—341).—Donovan's solution is essentially a solution of arsenious acid, mercuric hydrogen iodide, and hydriodic acid, and deterioration results in oxidation to arsenic acid both by the air present in the bottle and by the oxidising action of iodine and water. The stability is increased (a) by storage in amber bottles, well-filled bottles, and in a refrigerator; (b) by replacement of the air in the bottle by an inert gas; and (c) by replacement of 25% of the water by honey or syrup. Changes in the proportions of the ingredients, the use of porcelain or Wedgwood mortars, or variations in the method of preparation have no influence on the stability; the presence of metallic mercury is also ineffective, and free arsenic dissolves and increases the arsenic content. The rate of deterioration increases with increasing

acidity or basicity, but the addition of calcium carbonate in sufficient amount to neutralise the acid present (p_H , when freshly prepared, is about 1.2) has a marked preservative influence; a neutral solution, having the same chemical composition as the official U.S.P. preparation, but varying in the method of preparation, was found to be much more stable. The addition of 0.4% of oxalic acid, "methenamine," terpin hydrate, or hypophosphorous acid retards the deterioration, but of these it is only practicable to use terpin hydrate. Apparently light of wave-length 3200–4600 Å. is responsible for most of the deterioration.

E. H. SHARPLES.

See also A., May, 599, **Derivatives of 6-amino-3-hydroxybenzoic acid** (PUNEDDU and SANNA). 609, **Constituents of *Arctium Lappa*** (SHINODA). **Rotenone from *Derris* root** (TAKEI and others). 617, **Reactions of antipyrine** (EKKERT). 623, **Microchemical reactions of pilocarpine and cocaine** (WAGENAAR). **Reactions of atropine and related compounds** (EKKERT). **Harmine** (KREITMAIR; WOLFES and IVERS). 624, ***Strychnos* alkaloids** (LEUCHS and HOFFMANN). 625, **Strychnine and brucine** (ASHLEY and others). **Microchemical reactions of apomorphine** (WAGENAAR). 629, **Microchemistry of cystine** (WAGENAAR). **Microchemical contributions** (VAN ZIJP). 639, **Detoxication of chloroform** (FÜHNER).

PATENTS.

Manufacture of ureides of dialkylacetic acids. F. HOFFMANN-LA ROCHE & Co. A.-G. (G.P. 459,903, 23.4.26. Switz., 20.1.26. Cf. B.P. 264,804; B., 1927, 573).—Further to the B.P., diethylbarbituric acid is heated with 3% aqueous ammonia to give diethylacetylcarbamide, m.p. 207°.

C. HOLLINS.

Tobacco poor in nicotine and its manufacture. GEN.-DIREKTION DER OESTERR. TABAK-RÉGIE (B.P. 307,703, 11.3.29. Ger., 10.3.28).—Nicotine-free tobacco prepared by extraction is mixed with untreated tobacco to produce a mixture of low nicotine content yet of good colour and flavour.

E. B. HUGHES.

Manufacture of cotarnine derivatives. E. MERCK CHEM. FABR., Ascees. of M. OBERLIN (G.P. 456,856, 23.10.25).—Narcotic, analgesic products similar in character to pyrimidone, but possessing also hæmostyptic properties, are prepared by condensing cotarnine base (or a salt with sodium ethoxide) with a reactive pyrazolone. *E.g.*, with 1-phenyl-3-methyl-5-pyrazolone there is obtained a product, m.p. 175–177° (decomp.) [*hydrochloride*, m.p. 188° (decomp.); *hydrobromide*, m.p. 171° (decomp.)], whilst that from 1-phenyl-2:3-dimethylpyrazolone has m.p. 220° (decomp.).

C. HOLLINS.

Manufacture of *ar*-tetrahydro- β -naphthol derivatives. CHEM. FABR. VON HEYDEN A.-G., Ascees. of D. LAMMERING (G.P. 457,060, 10.2.25. Addn. to G.P. 414,261; B., 1925, 865).—An anthelmintic is obtained by condensing the chloroformate of *ar*-tetrahydro- β -naphthol with anthranilic acid to give the *urethane*, m.p. 175° (decomp.).

C. HOLLINS.

Manufacture of therapeutical media [arsenicals]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,789, 13.11.28).—An aminoalkyl or alkylated amino-

alkyl group is introduced by the usual methods into the amino- or hydroxyl groups of aminophenolarsinic acids; the products may be reduced alone or with other arsinic acids to give arsenobenzenes. Arsanilic acid is dissolved in alkali and warmed with β -diethylaminoethyl chloride at 60° to give 4- β -diethylaminomethylaminobenzenearsinic acid, which is reduced with hyposulphite and magnesium chloride to the corresponding arsenobenzene.

C. HOLLINS.

Manufacture of derivatives of organic arsenic antimony compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,537, 13.12.28).—Arylamines carrying both arsinic and stibinic acid groups, or their reduction products, or amino-substituted arsenostibobenzenes, $R \cdot As : Sb \cdot R'$, are solubilised by conversion into their formaldehyde-bisulphite compounds. Products from 3:4'-diamino-4-hydroxyarsenostibinobenzene (prepared from the corresponding arsine and stibinous chloride), and from bis-(3-amino-4-hydroxybenzenearsenostibino)-3-amino-4-hydroxyphenylarsine, $(Ar \cdot As : Sb)_2 As \cdot Ar$, are described. The latter compound is obtained from 3-amino-4-hydroxyphenylarsine by treatment with the potassium salt of antimonyl-1:2-dihydroxybenzene-3:5-disulphonic acid.

C. HOLLINS.

Manufacture of soluble organic antimony compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,533, 12.12.28).—An *o*-dihydroxylated compound carrying sulphonic or carboxylic groups is treated with antimony pentoxide or an antimonate; examples are potassium pyrocatecholdisulphonate or pyrogalloldisulphonate with antimonic acid, or the diethylaminoethyl alcohol salt thereof. The products have low toxicity and high therapeutic value.

C. HOLLINS.

Metal containers for ether. MALLINCKRODT CHEM. WORKS (B.P. 300,641, 5.11.28. U.S., 17.11.27).—See U.S.P. 1,697,320; B., 1929, 453.

Aminoalkylamino-derivative of aromatic amino-hydroxy- or polyamino-compounds. W. SCHULEMANN and W. KROPP, Asses. to WINTHROP CHEM. CO., INC. (U.S.P. 1,757,394, 6.5.30. Appl., 30.6.27. Ger., 8.7.26).—See B.P. 303,093; B., 1929, 265.

Barbituric acid compound. F. HEFTI (U.S.P. 1,757,906, 6.5.30. Appl., 2.8.28. Switz., 9.3.28).—See B.P. 307,484; B., 1930, 264.

Iron organic compounds (B.P. 304,731).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Determination and separation of silver chloride [from photographic layers] by sedimentation. **Nature and origin of the high silver values.** H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 28, 35–40).—By a suitable treatment of the neutral emulsion with alcohol or by formation of a calcium oxalate precipitate therein a rapid sedimentation of the silver halide and a filtrate free of the latter are obtained, and a silver determination is thus easily effected. Rapid sedimentation also results when sulphosalicylic acid is added, this process being suitable when silver halide is

to be determined. Sodium sulphite dissolves 75% of the silver values, thus supporting the authors' theory of the presence of an organic silver compound. An organic silver compound which is soluble in sodium sulphite also appears to be formed by the action of silver nitrate on the gelatin, and these compounds are believed to be identical. The latter contains no nitrate, is difficultly soluble, and cannot be completely washed out of the jelly. It is reduced by the developer, and is the cause of the cloudiness produced by longer development of silver chloride or silver chloride-bromide paper emulsions since, with its removal by centrifuging or by dichromate-sulphuric acid treatment, this trouble is obviated. The observed variation in the silver values when using different halides is probably directly connected with the fact that the addition of potassium chloride replaces less silver halide from the compound than addition of potassium bromide or iodide.

H. I. DOWNES.

Gravimetric determination of excess silver in photographic layers. H. ARENS and W. JESSIEN (*Z. wiss. Phot.*, 1930, 27, 273—278).—The authors have repeated the work of Schmidt and Pretschner (*B.*, 1928, 625) and obtain similar results, but do not regard the silver found as having been present originally as free silver in the emulsion tested. The present authors find that if instead of nitric acid other agents (*e.g.*, caustic soda, chromic acid, ammonium persulphate) are employed to decompose the gelatin, no silver is detectable in the product. They conclude that the silver ions found by the method of Schmidt and Pretschner (method *A*) arise from the interaction of the silver halide with gelatin and nitric acid, with the formation of silver nitrate and of halogen derivatives of phenolic compounds known to be contained in the gelatin. This mechanism is suggested by the production of ionic silver when silver chloride is treated by method *A* in the presence of phenol, even if dextrin be employed in place of gelatin, or again in the entire absence of a protective colloid. No silver is produced in the last two cases in the absence of phenol. Again, if a chloride emulsion be centrifuged, the sum of the silver ions found in the solid and liquid phases by method *A* is not equal to that found in the uncentrifuged emulsion; this is to be expected from considerations of mass action if the silver were not originally present, but were formed during the process.

L. V. CHILTON.

Gravimetric determination of excess silver in photographic layers. H. H. SCHMIDT and F. PRETSCHNER (*Z. wiss. Phot.*, 1930, 28, 30—34).—The objections raised by Arens and Jessien (preceding abstract) and by Leszynski (*A.*, 1930, 305) to the authors' theory that the high silver values in photographic layers are due in part to the presence of metallic silver and in part to an organic silver compound, and the alternative theory of Arens and Jessien that during the nitric acid treatment silver halide is reduced, the halogen being organically bound by the gelatin and the reduced silver going into solution, are discussed. Evidence is set forth from the present authors' earlier work (*cf.* *B.*, 1928, 625; *A.*, 1929, 894, 1405) in support of their theory,

the experimental work on which Arens and Jessien base their objections being criticised.

H. I. DOWNES.

Determination of traces of silver in photographic layers. F. LÜHR (*Z. wiss. Phot.*, 1930, 27, 283—303).—

The differential electro-titration method of Cox (*A.*, 1925, ii, 999) is applied to dilute solutions of silver nitrate, full experimental details being given. In this method two titrations are performed simultaneously, one in advance of the other by a constant small amount; the end-point is signalled by a sharp maximum in the *P.D.* between the two cells, which are connected in series and contain equal quantities of the solution under test. The sharpness of the end-point increases as the "start" given to one titration is decreased; a 0.02-c.c. "start" gives, with the apparatus described, a very sharp end-point without plotting the results, but the same maximum is recognisable, on plotting, even with a 2.0-c.c. "start." The method is only slightly affected by the action of light, and least so when chloride or iodide is employed in titration. It will detect 0.001 mg. Ag with an error of less than 1%, when using 0.0001*N*-potassium iodide, and may be applied to emulsions if the gelatin be first broken down with nitric acid (*d* 1.2). The applicability of the method in the presence of gelatin is not greatly dependent on the gelatin concentration; 0.01 mg. Ag may be detected to within 100% in the presence of 0.35 g. of gelatin, and 0.005 mg. Ag to within 20%. The products of hydrolysis of the gelatin are neutralised with ammonia and acidified with glacial acetic acid before titration, to avoid corrosion of the silver electrodes and errors introduced thereby.

L. V. CHILTON.

Correlations between photographic characteristics in the normal and in the solarised regions of exposures. A. P. H. TRIVELLI and E. C. JENSEN (*J. Franklin Inst.*, 1930, 209, 37—81).—The sensitometric constants of 14 commercial brands of plate covering the complete range of speed from the fastest to the slowest were investigated in the normal and the over-exposure regions, the results being treated statistically by use of the Bravais-Pearson correlation coefficient. The correlations found, by normal exposures, between grain-size, maximum density, contrast, *H.* and *D.* speed, and threshold value do not extend into the region of solarisation; some correlation does appear, however, between (i) the *H.* and *D.* speed, the threshold value at which solarisation commences and that at which the first reversal of solarisation occurs, and (ii) the solarisation threshold and the exposure which produces a maximum ratio of density to exposure. The results are considered in relation to sensitivity theories and are shown to favour the "concentration speck" theory of Sheppard and others, on the one hand, and the regression theory of solarisation (Lüppo-Cramer) on the other.

L. V. CHILTON.

Reactions involved in the various methods employed for the dissolution of the silver image. A. SEYEWETZ (*Bull. Soc. Franç. Phot.*, 1930, 17, 17—24).—The investigations are mainly concerned with the ease and completeness of the removal of the reaction

products and with the effect of traces of these products and of subsidiary reactions with the gelatin on the stability of the treated film. In the first type of silver solvent, in which the silver is removed as a water-soluble salt by means of an oxidising agent in presence of sulphuric acid, ceric sulphate is satisfactory, but must be freed from chloride before use in order to avoid the formation of silver chloride by the chlorine liberated by the acid. Potassium permanganate, followed by bisulphite or sulphite to remove the manganese sesquioxide, is very satisfactory at low temperatures, but above 20° or in cases of prolonged treatment a rapid decomposition of the gelatin takes place. Concentrated solutions of alkali dichromates are recommended for rapid work on heavy silver deposits, but the silver dichromate formed from the reaction of silver sulphate with the excess dichromate must be removed by treatment with bisulphite. Benzoquinone or its sulphonate is more rapid in action than ammonium persulphate, but considerably slower than the above-mentioned solvents, probably due to its tendency to render the gelatin insoluble. With the second class of solvent, in which a water-insoluble silver compound is first formed and removed by sodium thiosulphate, the following substances in conjunction with sodium thiosulphate work satisfactorily: potassium ferricyanide, with or without the addition of potassium chloride or bromide, chromic acid, potassium dichromate and hydrochloric acid, potassium chlorochromate, iodine, and benzoquinone and hydrochloric acid. The chemical processes involved are represented by equations, and formulae suitable for use as silver solvents are given.

J. W. GLASSETT.

See also A., May, 555, Photochemical reduction of silver salts by pinachrome (STEIGMANN). 593, 1-Methylnaphthalene and derivatives (STEIGER; VESELY and others).

PATENTS.

Photographic films [for cinematography]. FILM OZAPHANE (B.P. 301,879, 6.12.28. Fr., 7.12.27).—A cellulose film (0.02–0.06 mm. thick) is sensitised by the "ozalid" process or by impregnation with a suitable diazo compound and a coupling agent. By using such thin film in conjunction with an impregnation method of sensitising, it is possible to obtain an image sufficiently sharp for projection purposes. [Stat. ref.]

J. W. GLASSETT.

Photographic sensitising. C. ROCHRIC (B.P. 304,597, 18.1.29. Ger., 21.1.28).—Colloid layers which have been treated with sensitising baths, *e.g.*, dichromate solutions, are finally treated, either before or after drying, with an aqueous solution of alcohol (10% of alcohol), which serves to prevent the crystallisation of the chemicals at the colloid surface.

J. W. GLASSETT.

Manufacture of light-sensitive materials. M. P. SCHMIDT and W. KRIEGER, Assrs. to KALLE & Co. A.-G. (U.S.P. 1,756,400, 29.4.30. Appl., 28.1.28. Ger., 22.2.26).—See B.P. 311,196; B., 1929, 624.

Desensitiser and desensitisation of light-sensitive photographic materials. B. WENDT, Assr. to AGFA ANSCO CORP. (U.S.P. 1,753,911, 8.4.30. Appl., 28.12.28. Ger., 3.2.28).—See B.P. 314,144; B., 1929, 738.

[Tri-colour filter-selector for] colour photography. SOC. FRANÇ. CINÉCHROMATIQUE (PROC. R. BERTHON), Assecs. of SOC. CIVILE POUR L'ETUDE DE LA PHOT. ET DE LA CINÉMAT. EN COULEURS (B.P. 314,995, 1.7.29. Fr., 6.7.28).

XXII.—EXPLOSIVES; MATCHES.

See also A., May, 560, Determination of water [in explosives] (NOTEVAP).

PATENTS.

Production of explosives. M. HAHN and M. WOLF (B.P. 326,974, 27.2.29).—Burnt lime is dissolved in nitric acid, and the solution diluted according to the desired explosive power. Cellulose in the form of powdered peat, sawdust, etc. is added to the solution until a consistent, non-sensitive mass is obtained; the product is then dried, rendered sensitive by further addition of the lime-acid solution, and again dried for use.

H. ROYAL-DAWSON.

Production of explosives. M. HAHN and M. WOLF (U.S.P. 1,751,326, 18.3.30. Appl., 20.3.29. Ger., 31.1.29).—See B.P. 326,974; preceding.

Denitration of waste [sulphuric] acid mixtures. W. BÜSCHING (U.S.P. 1,755,768, 22.4.30. Appl., 15.8.27. Ger., 3.9.26).—See B.P. 276,972; B., 1929, 75.

XXIII.—SANITATION; WATER PURIFICATION.

Air-conditioning in the [paper] press room. J. W. GRAHAM (Proc. Tech. Sect. Papermakers' Assoc., 1929, 10, i, 153–158).—A lecture, mainly on hygrometry.

T. T. PORTS.

Estimation of dust in mine air. J. BOYD (Third Empire Min. Met. Congr., Apr., 1930, 23 pp.).—Methods available for making the determination are reviewed, and the results obtained when applying them on the Witwatersrand are given.

C. W. GIBBY.

Sampling of waters for oxygen determination. E. MERKEL (Chem.-Ztg., 1930, 54, 214).—A device for drawing the sample rapidly without mixing air into the water is described: the bottle is closed by a cork carrying two tubes, and filled through the wider tube (which reaches to the bottom of the vessel), while the air is displaced quietly through the other tube the opening of which is just below the cork. A convenient all-metal device (Merkel-Hehl) has been made on the same principle.

E. LEWKOWITSCH.

Removal of phenols from effluents. HOROVITZ-VLASSOVA.—See II. **Determination of iodine in water.** ANDREW.—See XVI.

See also A., May, 645, Germicidal efficiency of "electrolytic chlorine" and formalin on bacterial spores (AYYAR).

PATENTS.

Manufacture of toilet material. T. HASHIMOTO (U.S.P. 1,756,152, 29.4.30. Appl., 17.11.28).—See B.P. 323,321; B., 1930, 218.

Disinfecting, insect-destroying, deodorising, or perfuming process and [vacuum-cleaner] apparatus therefor. ELECTROLUX, LTD. FROM INVENTIA PATENT-VERWERTUNGS-GES. (B.P. 328,242, 15.11.26).

B.—APPLIED CHEMISTRY

JULY 4 and 11, 1930.*

I.—GENERAL; PLANT; MACHINERY.

High-temperature furnace based on the surface-combustion principle. E. RYSCHKEWITSCH (Chem. Fabr., 1930, 61—63).—To operate a surface-combustion furnace necessitates the use of special mixing chambers for the gas and air and a higher gas velocity through the porous walls than the rate of flame propagation. It is not possible to preheat the air much, and it may be necessary to work with a reducing atmosphere. The furnace described avoids these disadvantages by the separate introduction of gas and air and the reduction of resistance to gas flow to a minimum. The combustion space lies between two concentric cylinders and contains vertical rods of refractory material parallel to the (upward) gas flow. The hot gases pass down outside the outer cylinder and then preheat the inlet air. Combustion is complete in about 0.01 sec., and it is possible to attain a temperature of 2100° with town's gas. At these temperatures the "contact effect" is independent of the chemical composition of the surfaces, and may be attributed to the liberation of free electrons rather than to adsorption. C. IRWIN.

Heat insulation. A. B. WINTERBOTTOM (Proc. Inst. Mech. Eng., 1929, 1075—1086).—The laws of the conduction, convection, and radiation of heat are summarised. In considering the transfer of heat through lagging it is convenient not to consider radiation and convection separately, but to treat each surface as having a "coefficient of emission or absorption" which under the conditions of many problems is substantially constant. A formula is given for the calculation of heat transference in terms of these quantities, surface, conductivity, and temperature difference. Apart from the use of a vacuum, the best insulating medium is stagnant air. This is usually obtained by using a substance having a cellular structure. For low temperatures compressed granulated cork is more reliable than slag wool. For temperatures from 8° to 20° hair felt is generally used, and for steam-plant insulation best results are obtained with kieselguhr plastic covered with slag wool or plastic magnesia and a setting composition. Removable insulating coverings are usually constructed of asbestos and magnesia. For superheated steam plastic magnesia cannot be applied direct owing to the possibility of dissociation, and kieselguhr must be used. A new cellular insulating substance of promise consists of crinkled aluminium foil. For high temperatures kieselguhr brick is usual. C. IRWIN.

Insulation of heated and cooled surfaces. J. F. S. GARD and R. S. ROBINSON (J.S.C.I., 1930, 49, 125—137 r).—Types of heat insulation for various conditions are discussed, and their outstanding advantages and disadvantages compared.

Information is given relating to the selection of suitable materials and their protection in service. Methods of testing are touched on, and the mathematical aspect of the subject is dealt with at length. Test results for different materials are compared and discussed. Examples of cost of application and fuel saving effected in specific cases of boilers, pipe lines, etc. are included. References are made to the further advantages to be derived from suitable insulation other than the primary object of fuel economy. A number of graphs and tables are included.

Multiple-bulb consistometer. W. H. HERSCHTEL (J. Rheology, 1929, 1, 68—75).—The instrument consists of a U-tube, one arm of which is formed of six bulbs of 5-c.c. capacity, enabling the rate of flow at six different pressures to be measured. Narrow necks between the bulbs increase the accuracy of the instrument, and opacity of the liquid causes no difficulty, as the bulbs are being filled, not emptied. C. W. GIBBY.

Modified plastometer for industrial use. D. V. GREGORY, G. M. RASSWEILER, and K. C. LAMPERT (J. Rheology, 1929, 1, 30—45).—A plastometer is described for use with clear or coloured liquids of viscosities varying between those of raw linseed oil and pastes containing 90% of pigment; it is quick in action, and requires only 5 c.c. of liquid. The rate of flow of the liquid through one of a set of calibrated capillary tubes is measured, suction being applied by a filter pump connected to a pressure regulator, manometer, and ballast tank which also serves as a trap. The tubes are easily cleaned *in situ* by drawing through suitable solvents. C. W. GIBBY.

Orsat gas-analysis apparatus. BAHR.—See II. **Heating mixers with blast-furnace gas.** D'HUART. **Steel for machines.** DUESING. **Alloy steels for locomotives.** JOHNSON. **Heat-treatment of locomotive parts.** STANIER.—See X.

PATENTS.

Oil-heating furnace. L. DE FLOREZ (B.P. 328,064, 8.2.29).—It is desired that the heat from the flame be transmitted to the oil-carrying tubes by radiation alone. To this end the tubes are arranged as a cylindrical cage close to the walls of the furnace and the flame passes upwards (or downwards), without obstruction, along the axis of the cage; to keep the gases away from the tubes as far as possible without using shields, both the burner chamber and outlet flue are of smaller cross-section than that of the combustion chamber. The oil may flow through the tubes in series, in parallel, or series-parallel, and the gases after leaving the combustion chamber may pass through an air preheater of known convective type. B. M. VENABLES.

* The remainder of this set of Abstracts will appear in next week's issue.

[Boiler] furnaces. J. F. O. STRATTON (B.P. 328,055, 28.1.29).—Means for supplying forced air through a jet formed between two grate-like carriages at the bottom of the combustion chamber are described.

B. M. VENABLES.

Centrifugal steam dryer or separator. W. II. TAYLOR and C. SHAW (B.P. 328,122, 30.4.29).—A stationary device suitable for placing inside a boiler or accumulator is described.

B. M. VENABLES.

Packing of tubular heat-exchangers. P. S. D. SMITH. From CRANE PACKING CO. (B.P. 328,066, 9.2.29).—The holes in the tube plates are rebated, the annular space between the tube and the larger part of the hole is filled with compressible packing, and the end of the tube is belled out.

B. M. VENABLES.

Heating and cooling of liquids and automatically controlling the temperature thereof. H. S. BENSON (B.P. 327,729, 9.1. and 28.3.29).—A heat-sensitive device (thermometer or thermostat) closes one of two electrical circuits which include solenoids at opposite ends of a tilting trough of mercury, the tilting of which closes other switches which operate the actual heat control.

B. M. VENABLES.

Refrigerant with low f.p. STRATMANN & WERNER (F.P. 637,898, 19.7.27).—A solution of crystallised calcium chloride (d 1.4) at 65° is mixed under pressure with a solution of magnesium chloride (d 1.3) at 18° which is circulating at high speed through a branched tube.

A. R. POWELL.

Desiccating apparatus. A. H. HARVEY, ASST. to BOWEN RES. CORP. (U.S.P. 1,745,168, 28.1.30. Appl., 8.5.28).—A desiccator, of the type in which the liquid is sprayed into a drying medium such as warm air, has the lower part of the apparatus, in which the dried material collects, arranged to telescope into the upper part so that the volume of the drying zone can be varied. The lower part is formed of pervious fabric, forms the outlet filter for the gases, and is preferably conical to collect the dried material; it is cooled by jets of cold air or other gas impinging on the outside, and the cooling and drying media flow away together.

B. M. VENABLES.

Homogenising or like mills for liquid-borne solids. W. EPPENBACH (B.P. 327,808, 13.2.29).—In a mill of the type in which discs rotate with relative velocity and small clearance, the working faces are provided with grooves which are saw-toothed in section, with the steep faces radial, and decrease in depth from the periphery. They may start from either the inner or outer periphery, according to whether the feed is supplied to the inside or outside, or they may be formed from both edges, but in every case an annular smooth zone is left to hinder the flow of the pulp; the shearing action of the teeth is believed to effect most of the reduction.

B. M. VENABLES.

Production of mixtures of finely-divided solids and gases. H. HARPER, R. SCOTT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 327,840, 20.3.29).—The powder is tumbled in a barrel provided with longitudinal lifters. The gas is blown in and the mixture out through pipes which are on the axis and preferably have their openings adjacent to each other. The apparatus is

suitable for feeding a gas producer, *e.g.*, water-gas may be the conveying medium and the steam may be mixed subsequently.

B. M. VENABLES.

Vapour fractionation. J. C. MORRELL, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,738,386, 3.12.29. Appl., 25.4.27).—Below the trays of a fractionating column are placed diaphragms having depressions formed in them immediately under the downflow pipes and forming liquid seals for the latter. The remaining area of the diaphragms is perforated.

B. M. VENABLES.

Purifiers for evaporating apparatus. APPAREILS ET EVAPORATEURS KESTNER (B.P. 328,092, 20.3.29. Fr., 23.11.28).—A scrubber which is inserted in the vapour outlet of any stage of an evaporator is of the trickling type, which does not throw any back-pressure in spite of the flow of liquid being countercurrent to the vapour.

B. M. VENABLES.

Fluids for use in fluid-pressure apparatus. HYDRAULIC BRAKE CO., ASSECS. of A. T. K. TSENG (B.P. 299,370, 19.10.28. U.S., 24.10.27).—A fluid which has a higher b.p., lower vapour pressure, and more uniform viscosity than alcoholic mixtures, and which remains liquid even at -35° , does not affect metals or rubber, and is unlikely to form acids, is made by mixing an oil (preferably castor oil) with diacetone alcohol, preferably in equal parts, with addition of a metallic hydroxide if desired.

B. M. VENABLES.

Apparatus for bringing liquids and gases, or different liquids, into intimate contact. LEVER BROS., LTD., P. PRITCHARD, and R. CRAIG (B.P. 327,748, 11.1.29).—D-Shaped transverse baffles are inserted in a trough or pipe and by means of ports alternately on either side of the axis the liquid is caused to flow in a zigzag manner; meanwhile the other fluid is forced into it through passages with outlet jets formed in the baffles, which are of cast material, and when a number are assembled in abutting relation a common inlet passage is formed.

B. M. VENABLES.

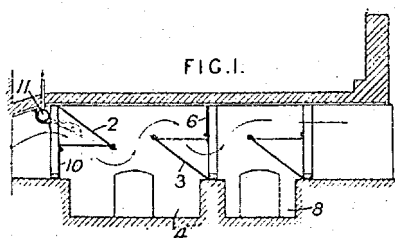
Centrifugal apparatus for separating and collecting dust or other solid particles from air and gases. C. H. W. CHELTNAM (B.P. 327,703, 5.12.28).—In a cyclone separator the lower, inverted, conical wall reverses to an upright cone and then reverts to an inverted cone so that a zone of increased diameter is formed, within which may be placed a baffle to prevent disturbance of the settled dust by the outgoing air. In the gas outlet is placed a diffuser in order to recover the dynamic head of the whirling gases; it comprises a double-conical thimble with sleeve, the outlet being through the annular space.

B. M. VENABLES.

Gas-washing towers. H. A. BRASSERT & CO. (B.P. 304,753, 3.12.28. U.S., 27.1.28).—The tower is constructed of a number of superposed cylindrical sections decreasing in diameter upwards and alternating with tapered or stepped sections which are not provided with filling. The lower edge of every cylindrical shell extends below the joint, so that any liquid running down the wall will fall clear and be dispersed on the hurdles or other filling next below. The area of the sections may be proportional to the volume of the gases as reduced by absorption and/or reduction of temperature.

B. M. VENABLES.

Device for separating unburnt particles from hot gases. G. KAMP (B.P. 309,045, 3.4.29. Holl., 3.4.28).—The gases are passed through a flue having



inclined baffles and doors for withdrawal of dust, as shown in the figure. The flue may encircle the base of a chimney.

B. M. VENABLES.

Re-activation of catalysts. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE DE L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 301,736, 1.12.28. Fr., 3.12.27).—Reduced metals, e.g., iron or copper, which have been used for removing oxides of nitrogen from gas mixtures, are re-activated by washing the catalyst with a solution of uncombined alkali or alkaline salts, including ammonia.

C. A. KING.

Effecting catalytic reactions between gases under pressure and at high temperature. S. G. S. DICKER. From M. CASALE-SACCHI (B.P. 328,139, 22.5.29).—All the operations of preheating, catalysing, recovery of heat, final cooling, condensation, and separation of products are carried out in one pressure-tight casing which is divided into annular compartments for the various functions, the innermost or catalytic zone being provided with an electric heater and the outermost with a cooling coil or other means of condensing the products; if necessary, the pressure-tight outer wall and covers may also be cooled. The recirculation of the residual gas is effected by an injector worked by the incoming fresh gas.

B. M. VENABLES.

Measurement of heat-quantities. SIEMENS & HALSKE A.-G. (B.P. 305,532, 16.1.29. Ger., 6.2.28).—A testing chamber is attached to the surface from which the flow of heat is to be measured, and in it are placed two temperature-sensitive devices, such as bimetallic coils, both approximately in the same place, but in line with the flow of heat, so that the second one will need a supplementary supply of heat to equalise the temperature of both. The supplementary supply is electrical and the bimetallic strips operate contacts, so that the current is on when there is any difference in temperature, and off (or reduced) when the temperature becomes equal. The supply of electrical heat is measured by a watt-hour meter, and is proportional to the integrated heat flow from the surface.

B. M. VENABLES.

[Recording] pyrometers adapted to the control of temperature. J. BRIDGES, and ELECTROFLO METERS Co., LTD. (B.P. 327,861, 12.4.29).—The pointer of such an instrument, while clamped by the printing bar, detains or allows to fall fully a tongue which drops in such a direction that no torque is exerted on the pointer. The tongue is adjustable longitudinally of the scale according to the temperature desired, and makes appropriate electrical contacts, which, though they operate at every recording period, only carry current

in the event of an adjustment to the heat supply being necessary.

B. M. VENABLES.

Thermostats. J. H. GRAYSON (B.P. 327,775, 21.1.29).—A tube and rod of different metals are connected together at one end, and the other end of the rod operates a plunger having a circular ridge on its face which engages with a thin convex disc of slightly larger diameter, and has its edge supported, but not clamped. The other side of the disc engages centrally with the stem of a poppet valve which controls the heating fluid. The valve may be arranged to open gradually or with a snap, the latter if the disc is allowed to buckle over the dead-centre; in either case the disc acts as a magnifying lever and the desired temperature may be adjusted by altering the length of the rod, it having a screwed joint for this purpose.

B. M. VENABLES.

Continuous absorption refrigerating apparatus. R. F. BOSSINI and G. MAIURI (U.S.P. 1,758,215, 13.5.30. Appl., 28.3.29. U.K., 11.4.28).—See B.P. 307,236; B., 1929, 501.

Centrifugal machines. SHARPLES SPECIALTY Co., ASSECS. of A. U. AYRES (B.P. 306,079, 29.1.26. U.S., 15.2.28).—See U.S.P. 1,737,137; B., 1930, 223.

Filter for liquids. A. GRONNING (U.S.P. 1,758,284, 13.5.30. Appl., 13.9.27. Denm., 25.7.27).—See B.P. 300,600; B., 1929, 80.

Apparatus for removing dust from gases or vapours. E. M. SALERNI, ASST. to E.M.S. INDUSTRIAL PROCESSES, LTD. (U.S.P. 1,758,624, 13.5.30. Appl., 31.10.25. U.K., 10.11.24).—See B.P. 247,274; B., 1926, 304.

Furnace walls. M. H. DETRICK Co. (B.P. 328,651, 8.2.29. U.S., 30.11.28).

Continuous production of cold by the compression method. DEUTS. GASGLÜHLICHT-AUER.-GES.M.B.H. (B.P. 310,474, 25.4.29. Ger., 26.4.28).

Jacketed metal drums. F. KRUPP A.-G. (B.P. 328,902, 30.9.29. Ger., 27.10.28. Addn. to B.P. 310,531).

Separation of particles from gases (B.P. 327,622).—See II. **Gas containers** (B.P. 304,775).—See VI. **Inert atmospheres** (U.S.P. 1,743,167).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Lignins of some peat-forming plants and of a sphagnum peat. II. G. STADNIKOV and A. BARYSCHEVA (Brennstoff-Chem., 1930, 11, 169—171; cf. B., 1930, 308).—Lignins isolated from samples of peat taken from a sphagnum peat bog at depths of 1.5 and 2 m. were similar in composition to the lignin of cotton grass (*Eriophorum vaginatum*) which has been shown to be represented by the formula $C_{50}H_{49}O_{11}(OMe)_2(CO_2H)(OH)_4$. It is concluded that the peat bog was originally formed from both sphagnum moss and cotton grass, but that the lignin of the former was transformed into humic acids more rapidly than that of the latter. The slow rate of transformation of wood and cotton-grass lignin is attributed to its colloidal nature, rather than to the antiseptic action of the accompanying resins.

One of the first stages in the transformation of lignin into humic acid, as shown by a comparison of lignins isolated from peat samples taken from different depths in the bog, is the loss of methoxyl groups.

A. B. MANNING.

Brown coal as a raw material for gas production.

W. HAUSWALD (Brennstoff-Chem., 1930, 11, 151—152, 172—174).—The possibility of producing town's gas from brown coal is discussed. High-temperature carbonisation has disadvantages, particularly the action of the ash on the retort walls, the somewhat low calorific value of the gas, and the difficulty of disposing of the coke. Low-temperature carbonisation yields a gas which when diluted with water-gas to the desired calorific value has still too high a sp. gr. and carbon monoxide content. Other suggested processes depend on subjecting the gases and tar vapours issuing from the retort to further heat-treatment. A gas of the desired properties could probably be produced by gasifying the brown coal in a suitably designed generator.

A. B. MANNING.

Combustion of powdered fuel. A. L. GODBERT (Fuel, 1930, 9, 57—75).—For the most efficient utilisation of powdered fuel in a furnace, the thermal intensity of the combustion should be maintained at its maximum value. According to Rosin (cf. following abstract) the thermal intensity is inversely proportional to the combustion period of the particles. Combustion, however, does not begin immediately the dust enters the chamber, and the ignition period is as important a factor as the combustion period in determining the effective use made of the combustion chamber. Since heat is absorbed in the ignition space from the combustion space, the former is detrimental to the furnace load; it can be reduced by preheating the air for combustion, injecting the dust into the furnace at as low a speed as possible, and supplying the minimum amount of air to the dust at the burner to suspend it. The ease of ignition of coal dusts is related to their rational analyses, low-rank coals being more easily ignited than high-rank coals, and durainous coals than bright coals. The relationship between combustibility and rank is less definite. The combustion periods of individual particles of coal have been investigated by a photographic method (cf. Griffin, Adams, and Smith, B., 1929, 877), but the results are of limited applicability to the combustion of powdered fuel in the plant. The combustion period decreases with increase of the specific surface of the dust, but this is limited in practice by the cost of pulverisation. The combustion period at first decreases with increasing proportion of excess air, then, after passing through a minimum, increases again (cf. Audibert, Rev. de l'Ind. Min., 1924, 73, 1). The system in which the whole of the air is mixed with the dust when it begins to burn is more favourable to rapid combustion than that in which part only is supplied at first, and the remainder when the "volatile matter" has burnt. By inducing turbulence in the dust cloud, the thermal intensity of combustion is improved; the effect is probably due more to the better dispersion of the dust than to the higher relative motion between the dust and the air. A high temperature of the combustion

space favours a high rate of combustion. To prevent destruction of the chamber lining when high temperatures are used, water-cooling of the walls is essential; such cooling should be just sufficient to cool the burnt gases to the fusion point of the ash before they come into contact with the walls and before they enter the boiler (cf. Lölöfs, Trans. Fuel Conf., 1928, 3, 362).

A. B. MANNING.

Thermodynamics of the combustion of powdered coal. P. ROSIN (Proc. II Int. Conf. Bit. Coal, 1928, 1, 838—864).—Formulae correlate the heat generated per unit volume of combustion space; shape, size, and temperature of particles; combustion time, calorific value, and composition of the fuel. In most power plants the combustion time of the largest particle is 1.5—3.6 sec., and in locomotives 0.17—0.4 sec.

CHEMICAL ABSTRACTS.

Control of vertical retorts [for continuous carbonisation]. H. B. KENDRICK (Gas J., 1930, 190, 306—309).—Results obtained in working Glover-West retorts since their construction in 1922 are discussed, and attention is drawn to the ease of control of calorific value in the gas made. Factors such as the voltage of current supplied to extractor-gear motors, or the quantity of steam admitted to the charge, must be carefully regulated, and it is suggested that by operating producers under pressure much better temperature control would be possible. Leakage of gas to or from the retorts would be diminished by similar practice. R. H. GRIFFITH.

Low-temperature carbonisation [of coal] in thin layers. H. MANTEL (Brennstoff-Chem., 1930, 11, 150—151).—A process has recently been developed for carbonising coal in layers 1 mm. or less in thickness (cf. B.P. 301,907; B., 1929, 968). Owing to the consequent rapidity with which the heat is transmitted through the material the required throughput is attained without raising the temperature of the retort itself anywhere much above the desired temperature of carbonisation. The tars produced therefore undergo a minimum of cracking. The apparatus, which consists of a ring-shaped retort through which the material is conveyed on a rotating plate, is briefly described.

A. B. MANNING.

Distribution of sulphur in the combustion of coal and coke. I. TRIFONOV and E. RASCHEVA-TRIFONOVA (Brennstoff-Chem., 1930, 11, 165—169).—A gas coal was finely powdered and the bulk of the ash separated therefrom by flotation in a mixture of carbon tetrachloride and xylene of d 1.30. The remainder of the inorganic sulphur was then removed from the coal by treatment with cold nitric acid (d 1.20) and subsequent washing. The purified coal contained about 1% of ash and 1.1% of organic sulphur. Coke free from inorganic sulphur was prepared by carbonising some of this coal and treating the coke with nitric acid. The coal and coke were burnt in an electrically heated muffle at 900°, alone and with 10—15% of various forms of inorganic sulphur and other ash constituents; the distribution of the sulphur between the ash and the gaseous products of combustion was determined. Pyrites lost its total sulphur as sulphur dioxide. Calcium sulphide lost more sulphur than the sulphate, whereas

the reverse was true for the corresponding magnesium compounds. The sulphur dioxide evolved from the organic and pyritic sulphur was partly retained by certain other ash constituents, more especially by calcium carbonate, and to some extent by basic silicates. Magnesium carbonate absorbed sulphur dioxide at temperatures below 800°, but gave up the greater part of it again at 900°. Alumina, silica, and kaolin were without effect on the distribution of the sulphur. The results were the same whether the fuel burnt was coal or coke. If the mixture of coal and ash is burned before being coked the sulphur distribution is affected only when calcium sulphate or carbonate is present; this is attributed partly to the reduction of calcium sulphate to sulphide and partly to the volatilisation of some of the sulphur during carbonisation.

A. B. MANNING.

Preparation of active carbon. E. BERL and H. BURKHARDT (Z. angew. Chem., 1930, 43, 330—333).—Numerous samples of active carbon have been prepared by dropping various pure organic compounds into a steel cylinder heated at 1000° and quenching the carbonised product in water. When the compound contained potassium a vigorous evolution of hydrogen occurred during heating, but the corresponding sodium compound did not exhibit this phenomenon. After washing the carbons with hot dilute hydrochloric acid, then with water, their adsorptive capacity for methylene-blue and heat of moistening with benzene were determined. The tabulated results show that the presence of potassium in the molecule of the organic compound used causes a highly active form of carbon to be obtained, whilst the simultaneous presence of potassium and sulphur in the compound leads to the production of a much more highly active form of carbon than any hitherto prepared. Thus from potassium naphthalenedisulphonate the carbon has an effective surface area of 1080 m.²/g., and will absorb 46% of its weight of ether from air containing 4.125 g./m.³

A. R. POWELL.

Volumetric determination of carbon in graphite.

E. SCHWARZ VON BERGKAMPF and L. HANANT (Z. angew. Chem., 1930, 43, 333—335).—The finely-powdered sample (0.1 g.) is heated at 1200° in a current of oxygen and the gases are collected and measured in the usual gas-analysis apparatus with a burette and automatic pipette of 600 c.c. capacity. The carbon dioxide is then absorbed in potassium hydroxide and the decrease in volume measured. A correction must be applied if the original substance contained carbonates; these are determined in a separate trial in the usual way. A nomograph for calculating the weight of carbon dioxide from the volume at various temperatures and pressures is included.

A. R. POWELL.

Determination of volatile matter [in fuels etc.].

A. P. SHAKHNO and M. D. ZHOOKOVSKAIA (Izv. Tepl. Inst., 1929, 7, 38—50).—The use of a new platinum crucible gives high values, probably owing to catalytic action.

CHEMICAL ABSTRACTS.

Determination of thiocyanates in coke-oven liquors. TRAVERS and AVENET (Compt. rend., 1930, 190, 1128—1129).—Sulphides are removed by precipitation as zinc or cadmium sulphide, the remaining solution is saturated with sulphur dioxide, and the

CNS ion precipitated as cuprous thiocyanate by means of copper sulphate. If more than 0.1% of thiosulphate is present from oxidation of polysulphides, the solution should be cooled. After 3 hrs. the precipitate is washed, dissolved in an excess of ammonia, oxidised by addition of a few drops of hydrogen peroxide, the excess of which is boiled off, and the cupric compound titrated with potassium permanganate solution in the cold in the presence of a 15% excess of free sulphuric acid. The CNS ion is decomposed into the CN ion, which does not react, and sulphur, which is oxidised to sulphuric acid. The accuracy is 1%.

J. GRANT.

Fuel gas technique. Some modern developments.

L. H. SENSICLE (J.S.C.I., 1930, 49, 57—66 T).—More than half the gas used in industry is still made at the consumer's works. The constant quality demanded in modern applications is rendered possible by the new accurate recording calorimeters. In the Thomas calorimeter variations in temperature and humidity of the calorimetric air affect the accuracy of the instrument. Calculations are given. Modern methods of gas storage such as waterless piston holders and high-pressure spherical holders are discussed. In the combustion of gas the three types of air admixture, viz., (1) when no primary air is mixed with the gas before combustion, (2) part of the air is added before combustion, (3) all the air required is supplied before combustion, are discussed in relation to recent developments. In particular, submerged combustion, belonging to category (3), has important uses in chemical industry for concentrating corrosive liquids such as ammonium chloride solutions. Recent work in relation to gas fires indicates that radiation in the main shorter than 2.0×10^{-3} mm. wave-length is desirable owing to the scorching effect produced by absorption of infra-red rays longer than this. In large-scale gas burners automatic adjustment of the air supply is necessary for high efficiency. Types of apparatus are described. The corrosion of mains is discussed, and the author's method for rapidly testing pipe coverings is outlined and results are given. A few weeks' controlled electrolysis in wet ground is adequate to determine the liability to corrosion of protected pipes. Problems awaiting solution are discussed.

Purification of illuminating gas by the dry method.

C. MAZZETTI (Annali Chim. Appl., 1930, 20, 139—147).—The volume of gas that can be freed from hydrogen sulphide by a given quantity of ferric oxide is rigorously constant within the limits of experimental error, so that the principal reaction must be a true chemical reaction. The presence of hydrogen or carbon monoxide in the gas has no influence on the purification, provided that oxygen is absent. Addition of oxygen to mixtures of hydrogen sulphide with nitrogen greatly increases the volume purified by a definite amount of the purifying agent, this effect being diminished considerably if hydrogen is also present, but remaining unaffected by carbon monoxide. The added oxygen does not revivify the mass *in situ*, but determines the reaction, $2\text{H}_2\text{S} + \text{O}_2 = 2\text{H}_2\text{O} + 2\text{S} + 110,000$ g.-cal., which is activated catalytically by the ferric oxide. Increased concentration of the hydrogen in the

gas exerts an inhibiting function on this reaction owing to adsorption phenomena. T. H. POPE.

[Coal] gas drying. R. MEZGER and F. PISTOR (*Gas- u. Wasserfach*, 1930, **73**, 193—200).—The principles involved in the drying of coal gas are reviewed, and the chief advantages and drawbacks of the process are discussed. It is shown that although the volume of the dried gas is smaller, its calorific value is higher than that of the moist gas, so that a slightly larger proportion of water-gas can be added to it. Methods for adapting water-sealed gas holders for storage of dry gas, by use of a suitable oil film, are mentioned and the various types of dehydration plant are described. These may depend on compression, cooling, adsorption, or washing with hygroscopic liquids, and it appears that drying by means of glycerin or calcium chloride can be most cheaply operated. A bibliography is included in the paper. R. H. GRIFFITH.

Determination of the explosion limits of gases and gas mixtures. II. LÖFFLER (*Brennstoff-Chem.*, 1930, **11**, 145—146).—A micro-gas calorimeter is described which permits the determination of the calorific value of a gas, of which only 10 c.c. are available, with an error not greater than 0.33%. It can be used to determine also the limiting explosive mixtures with air or oxygen, the limiting mixtures giving complete combustion, the carbon dioxide content of the gases before and after combustion, and the diminution in pressure due to combustion. It consists essentially of a vacuum-jacketed, glass, explosion vessel at the centre of which is the bulb of a sensitive mercury thermometer. The vessel is connected by capillary tubes to a water-jacketed gas burette and a mercury reservoir; a carbon dioxide absorption tube, through which the gas can be passed when desired, is also provided. A spark gap in the explosion vessel, in series with which is a small argon vacuum tube to act as an indicator, is connected to an induction coil actuated by a pocket-lamp battery. A. B. MANNING.

Determination of the water-gas equilibrium under the influence of the electric discharge under reduced pressure. K. PETERS and H. KÜSTER (*Brennstoff-Chem.*, 1930, **11**, 171—172; cf. *B.*, 1930, 44).—Mixtures of carbon dioxide and hydrogen were passed through a tube wherein they were subjected to an electric discharge under pressures of 20—80 mm. of mercury, and the issuing gases were analysed. It was impossible to correlate the observed equilibrium constants with either the electrical measurements or determinations of the gas temperature. With a high electrical energy input, particularly in the range of high initial carbon dioxide concentrations, the equilibria were comparable with those obtained in the purely thermal reaction. Equilibrium is attained in less than 0.15 sec., and is unaffected by variations of pressure from 20 to 60 mm. of mercury. A. B. MANNING.

Orsat apparatus for quick and accurate gas analysis. H. A. BAHR (*Gas- u. Wasserfach*, 1930, **73**, 440—444).—A modified Orsat apparatus for the analysis of coke-oven and similar gases is described. The apparatus is in two portions, one portion being used for absorption and the other for combustion analysis.

Errors due to the capacity of the connecting capillaries are eliminated by keeping these filled with the confining liquid (saturated sodium chloride solution acidified and coloured red). Karlsruhe three-way taps placed in the bridge itself above the absorption pipettes facilitate this procedure. Accidental loss of gas is prevented by placing safety pipettes of more than 100-c.c. capacity, and containing the sodium chloride solution, at the ends of the capillary and absorption train. The pipettes used are of a modified valve type, and have been found satisfactory in use. They are sealed to the atmosphere by connecting to pipettes containing either water (in the case of cuprous chloride pipettes) or sulphuric acid (for the fuming sulphuric acid pipette). The glowing-wire combustion pipette is provided with electrodes consisting of glass tubes expanded at the ends and filled with mercury covered with saturated sodium chloride solution. The platinum wire is energised from the electric mains through either a transformer or resistance. Provision is made for the control of the temperature of the wire. When methane or one of its homologues is present in the gas sample the hydrogen is determined by passing the gas over copper oxide at 300° and the methane etc. is determined afterwards by combustion in a quartz tube maintained at 800—1000°. The apparatus is flushed out with carbon dioxide when hydrogen is being determined, and with oxygen in the case of methane determinations, the cleansing gases being absorbed after combustion. The carbon dioxide is generated by a small Kipp's apparatus and the oxygen by electrolysis. Recently it has been found that the carbon dioxide reduced the activity of the copper oxide and the use of oxygen as cleansing gas leads to simplification of the apparatus. A coke-oven gas can be analysed with an accuracy of 0.1—0.2% in the apparatus in 20 min. H. E. BLAYDEN.

Determination of sulphur in gas. J. H. STEINKAMP (*Chem. Weekblad*, 1930, **27**, 271—272).—The absorption apparatus in which the products of combustion are brought into contact with hydrogen peroxide solution is provided with a cooling coil, to avoid decomposition of the hydrogen peroxide. S. I. LEVY.

Determination of naphthalene in oils and coal gas. S. TWEEDY (*Gas World*, 1930, **92**, 497).—Oil containing naphthalene is heated slowly, in a U-tube immersed in an oil-bath, to 150°, and a stream of air is passed over it to a 10-bulb Meyer absorption tube containing picric acid solution. The naphthalene picrate formed is separated by means of a sintered glass filter, and the picric acid liberated by its decomposition is titrated. R. H. GRIFFITH.

Cracking of tar acids from coal. J. C. MORRELL and G. EGLOFF (*J.S.C.I.*, 1930, **49**, 263—271 r).—The economic success of low-temperature carbonisation depends on the profitable disposal or utilisation of low-temperature coal tar, and this in turn depends in a large measure on the profitable utilisation of the tar acids. In the present work the problem is approached from the viewpoint of converting, by the cracking process, the high-boiling tar acids into tar acids or phenols of lower mol. wt. and lower boiling range which possess greater toxicity as antiseptics and have greater utility for other

uses, such as wood preservatives, animal dip, phenol-aldehyde condensation products, mineral flotation, etc. High yields of low-boiling tar acids were obtained. The percentage of phenols and cresols made by the cracking process was determined. The germicidal efficiency of the low-boiling phenols was also determined. A study of the effect of water vapour on the cracking of tar acids was made.

Ultra-violet ray analysis as applied to coal-tar pitch and bitumen. W. TEUSCHER (Chem. Fabr., 1930, 53).—Solutions of pitch, bitumen, and mixtures of the two can be distinguished by observation from above of the fluorescence colour as shown in three reagent vessels mounted in a block of wood transversely to the rays of a Heraeus lamp. C. IRWIN.

Substitute for amalgamation in testing bituminous materials for m.p., ductility, and float test. H. EISNER (Ind. Eng. Chem. [Anal.], 1930, 2, 176).—The use of an amalgamated plate on which excess mercury may be left is open to objection on account of the danger of mercury poisoning. It cannot be replaced by a highly polished plate, to which bitumen sticks, but a rough plate of brass or stainless steel cleaned and covered with a layer of glycerin is satisfactory. C. IRWIN.

Disintegration of kukkersite in organic liquids. K. LUTS (Brennstoff-Chem., 1930, 11, 148—150. Cf. Craig, also Kogerman, B., 1922, 799 A).—Pieces of kukkersite placed in ether or acetone undergo considerable swelling, principally in a direction perpendicular to the bedding plane, and numerous cracks appear in the material which rapidly breaks down into small plates. At the same time a marked evolution of gas occurs. The material behaves similarly in chloroform, pyridine, or carbon disulphide; it softens, but does not break down, in alcohol or aniline, and petroleum or benzene is without effect on it. The rapidity with which the material breaks down in different liquids is roughly proportional to the heat of wetting of the material by the liquid, and is also related to the surface tension of the liquid. The breakdown appears to be due to the gas pressure set up by the penetration of the liquid into the capillaries. Some other oil shales exhibit a similar phenomenon, but to a lesser degree. A. B. MANNING.

Generation of oil in rocks by shearing processes. J. E. HAWLEY (Bull. Amer. Assoc. Petr. Geol., 1929, 13, 303—366).—The determination of soluble organic material in oil shales is considered. Oxidation renders soluble some of the insoluble organic material. Shales which have been subjected to high shearing pressures give an extract containing more volatile hydrocarbons than those not sheared. Experiments on the effect of shearing indicate that high shearing pressures at low temperatures and during short periods of time are unimportant in the generation of oil.

CHEMICAL ABSTRACTS.

Oil and water content of Grozni oil sands. N. T. LINDTROP and V. M. NIKOLAEV (Bull. Amer. Soc. Petr. Geol., 1929, 13, 811—822).—A study of the displacement of oil by water in sands of different grain sizes leads to consideration of the large amount of water retained

in reservoir sand and of questions of production and recovery.

CHEMICAL ABSTRACTS.

Paraffin content of Baku crude oils. L. GUKHMAN and D. GOLDBERG (Azerbaij. Neft. Choz., 1929, No. 10, 74—76).—Removal of resins with fuller's earth, silica gel, or activated carbon yielded from the same sample paraffin in quantity and m.p., respectively, 2.1%, 43°; 3.45%, 53°; 3.05%, 53°. The wax was extracted from the absorbent with a loss of 30%. The wax is preferably separated from acid sludge heated at 65—75° with a low-cold test gas oil as diluent.

CHEMICAL ABSTRACTS.

Fergana crude oils. V. FOMENKO (Neft. Choz., 1929, 17, 560—569).—Characteristics of crude and fractionated oils from the Chimion, Santo, and Shor-Su districts are recorded.

CHEMICAL ABSTRACTS.

Perm petroleum. D. M. MARKO (Bull. inst. rech. biol. univ. Perm, 1929, 6, 524).—The petroleum from Tschussovski Gorodki had $d_{40}^{15.0}$ 0.948, viscosity at 50° 1.52, ignition temperature below 5°, congealing temperature below -20°, S 3.1%, wax 32%, decomp. 270°.

CHEMICAL ABSTRACTS.

Ural crude oil. M. M. FAINGARD and E. M. BRANDO (Neft. Choz., 1929, 17, 83—85).—The oil (I. V. Kosior) had d 0.955, E_{30} 2.67, Abel-Pensky flash point 10°, SO_3 4.5%, S 1.2%, initial b.p. 66°, aromatic compounds 7%. Other data are also recorded.

CHEMICAL ABSTRACTS.

Cracking of Ural crude oil. M. D. TILITSCHÉEV (Neft. Choz., 1929, 17, 82—83).—The crude oil, d 0.938, E_{50} 1.49, was stripped of gasoline and cracked at 425° at 10—20 atm., yielding 10% of gasoline, 86% of residue (d 1.04, E_{50} 7), and 4% of gases etc. In a second experiment, with recycling, the gas-oil fraction yielded 22% of gasoline and 71% of residue (d 1.104, E_{100} 9.2).

CHEMICAL ABSTRACTS.

Refining of crude oil. J. C. COLDHAM (Chem. Eng. Min. Rev., 1930, 22, 261—268).—The plant and lay-out at the works of the Commonwealth Oil Refineries, Laverton, Victoria, is described and illustrated by diagrams. Anglo-Persian crude oil is handled exclusively, the monthly output of the plant being approx. 10,000 tons, and consisting of motor spirit 25, kerosene 10, fuel oil 62 vol.-%. Exhausted bauxite from the treatment of the crude kerosene is steamed and revived by roasting at 600°. A plant for the manufacture of sodium hypochlorite (from purchased caustic soda and chlorine) for washing the various distillates is described.

H. S. GARLICK.

Decolorising efficiency of Russian and foreign clays. V. TOKMANOV (Azerbaij. Neft. Choz., 1930, No. 1, 58—65).—Experiments with a solution of ozokerite in kerosene showed that Glukhov clays, activated with sulphuric acid, are effective. The adsorptive power is greatly increased by drying at 100—105°; further heating up to 350° has no effect on raw clays.

CHEMICAL ABSTRACTS.

Recracking [of kerosene]. B. K. TARASSOV and V. V. RUDENKO (Neft. Choz., 1929, 17, 530—532).—A kerosene (aromatics 13.2, naphthenes 35.2, paraffins 51.6%) was passed 24 times through an iron tube at 600—610°, the bottoms being mixed with fresh stock

to give the same charge. The total yield of gasoline (final b.p. 200°) was 45%, the content of aromatic hydrocarbons being increased by each cracking operation. Characteristics of the kerosene fraction are recorded.

CHEMICAL ABSTRACTS.

Action of potassium permanganate on unsaturated hydrocarbons. D. A. HOWES and A. W. NASH (J.S.C.I., 1930, 49, 113—119 r).—The action of solutions of potassium permanganate on a commercial liquid-phase cracked petrol and on pure unsaturated and saturated hydrocarbons was studied. As experimental controls, the determination of bromine values and aniline points was investigated. The effects of concentration, time, and temperature on these permanganate oxidations were studied. It was found that the use of 2% alkaline permanganate solution at 0° constituted the best conditions to obtain oxidation of unsaturated compounds, without affecting the other series of hydrocarbons possibly present, although this concentration of the oxidising agent only lowered the bromine value of the cracked spirit from 24.1 to 22.0 in 6 hrs. The oxidation of synthetic mixtures of olefinic hydrocarbons in *n*-heptane or light petroleum showed the olefines to vary in ease of oxidisability. It was attempted to correlate the ease of oxidation with anti-knock value, and generally speaking the olefines more easily oxidised were the poorer anti-knocks. Diamylene samples, however, did not fall into line with the other olefines in this respect.

Knock ratings of aromatic hydrocarbons. D. A. HOWES and A. W. NASH (J.S.C.I., 1930, 49, 16 r).—Following the investigation of a number of pure hydrocarbons (B., 1929, 272), a series of pure aromatic hydrocarbons (20% by volume in a standard benzine) were rated as regards anti-knock property in the Delco testing unit. Toluene has a better knock rating than benzene, and the three xylenes are more effective than toluene. Of the xylenes, *p*-xylene is the most effective and *o*-xylene the least. In general, the *p*-disubstituted benzenes are the best anti-knocks, *e.g.*, *p*-cymene, *p*-xylene, and *p*-*tert*-butyltoluene. Tertiary groupings appear to have a better effect than secondary groups; *e.g.*, *tert*-butylbenzene is more effective in reducing knocking than *sec*-butylbenzene. Only one of the aromatic hydrocarbons tested was found to have a lower anti-knock value than benzene, namely, *sec*-butylbenzene.

Polymerising action of sulphuric acid on unsaturated hydrocarbons contained in light cracked oils. I. E. BESPOLOV (Neft. Choz., 1929, 16, 730—736).—Addition of acid in small portions increases the amount of sludge. The action of 90—100% acid differs from that of 70—80% acid. Oil partly polymerised by storage, when treated with acid, gives unusually dark polymerides which are similar to those obtained by direct action of acid. Heating during treatment with acids produces a smaller quantity of polymerides of different appearance and properties.

CHEMICAL ABSTRACTS.

Composition of cracked gas oil. I. E. BESPOLOV (Azerbaid. Neft. Choz., 1929, No. 10, 57—71).—Gas-oil fractions from the Vickers coil were similar in composition to straight-run gas-oil fractions.

CHEMICAL ABSTRACTS.

Radioactivity of ash from certain crude oils. L. BOGOYAVLENSKI (Neft. Choz., 1929, 17, 91—92).—Ukhta borehole water contains 7.5×10^{-9} g. of radium per litre, as well as some mesothorium and its decomposition products. The radioactivity of the mineral matter is approximately inversely proportional to the amount.

CHEMICAL ABSTRACTS.

Natural crude-oil emulsions from the Boryslav-Tustanovic territory. III. T. KRZYŃSKI (Przemysł Chem., 1927, 11, 605—614).—The conductivities of the crude oil and of emulsions with water have been determined.

CHEMICAL ABSTRACTS.

Fractional analysis [of oils and gases]. F. L. KALLAM, G. W. DECKERT, and L. J. COULTHURST (Oil Gas J., 1929, 28, 209, 212, 370, 373).—A discussion of requirements for accuracy.

CHEMICAL ABSTRACTS.

Determination of sulphur [in oils] by the bomb method. A. MALYATSKI and B. NAKASHIDZE (Azerbaid. Neft. Choz., 1930, No. 1, 110—114).—The U.S. standard bomb method is satisfactory for cracked gasoline. The pressure of oxygen should be (for 0.60—0.65 g.) 16—18 atm. for gasoline, 25 atm. for gas oil, and 30—35 atm. for heavy oils.

CHEMICAL ABSTRACTS.

Conradson carbon determination. N. VASILIEV (Azerbaid. Neft. Choz., 1929, No. 4, 90—93).—Oils which are not properly washed after treatment with sulphuric acid show a high coke content. The amount of resins can be calculated from the values for coke and asphaltenes.

CHEMICAL ABSTRACTS.

Comparison of American and Russian paraffin waxes. S. S. NAMETKIN, A. S. VELIKOVSKI, and S. S. NIFONTOVA (Neft. Choz., 1929, 17, 533—549).—Grozni oil affords paraffins of higher quality than American, but contains much resin and is insufficiently sweated. The determination of the qualities of wax is discussed.

CHEMICAL ABSTRACTS.

Acids of montan wax. D. HOLDE, W. BLEYBERG, and H. VOHRER (Brennstoff-Chem., 1930, 11, 128—132, 146—148. Cf. 1929, B., 1929, 1005).—The acid, $C_{28}H_{56}O_2$, of m.p. 84.4°, previously separated from montan wax and then believed to be the *iso*acid, has been further purified by micro-distillation under very low pressure, and a product obtained the m.p. of which agrees closely with that of *n*-octacosic acid (89°). That this is the normal acid has been confirmed by X-ray examination. In a similar manner, from a fraction of lower m.p., *n*-hexacosic acid has been isolated in a sufficiently pure state to be identified by a mixed m.p. determination. Evidence was found of the presence of *n*-behenic acid ($C_{22}H_{44}O_2$) in the fraction of lowest m.p. It is concluded that the fatty acids derived from montan wax, like those from arachis oil, beeswax, etc., consist exclusively of the normal acids containing an even number of carbon atoms.

A. B. MANNING.

Fergana ceresin. V. E. TOKMANOV (Neft. Choz., 1929, 17, 556—559).—Ceresins are harder than paraffins, do not sweat, and usually have a fine crystalline structure. On distillation ceresins afford paraffins, but the latter have not yet been converted into the former.

CHEMICAL ABSTRACTS.

Petrolatums from Surakhani crude oil. A. VELIKOVSKI and S. S. NIFONTOZA (*Neft. Choz.*, 1929, 17, 71—78).

Carbon ratios and oil gravities in the Rocky Mountain region of the United States. C. E. DOBBIN (*Bull. Amer. Assoc. Petr. Geol.*, 1929, 13, 1247—1256).

PATENTS.

Treatment of coal [slurry]. B. NORTON (B.P. 327,845, 21.3.29).—The slurry from a coal-washing plant is delivered on to a vibrating draining screen and the water containing fine pyrites, clay, etc., which first drains away, is separated from the cleaner water, which drains away subsequently, by arranging two or more transverse gutters or troughs below the screen. The cleaner water is returned to the circulating system of the washer. A clean-water sprayer may be arranged above the screen to aid in washing the dense impurities from the coal. By arranging a valve in the gutter for the dense water a certain amount of sedimentation may be brought about therein, and the cleaner water may be allowed to flow over into the second gutter for recirculation with the other clean water.

A. B. MANNING.

Treatment of fine coal dust to render it suitable for transport in open vessels. S. and W. L. TULIP (B.P. 327,813, 16.2.29).—The fine dust is conveyed in a current of air to a vertical cylinder wherein it is brought into contact with low-pressure steam. The coal, with a uniform moisture content of about 10%, is delivered from the cylinder directly to the wagon used for transport.

A. B. MANNING.

Removal of liquid from peat, coal-mud, or like watery substances. A. TEN BOSCH (B.P. 304,329, 19.1.29. *Holl.*, 19.1.28).—The peat is provided with an increased draining or de-watering surface by perforating the outer surface of the mass, *e.g.*, by passing the material below a spiked roller. The increased surface is rendered more permeable by the application of heat, which may be generated by passing an electric current through the mass, the means used for producing the perforations serving as electrodes. In another modification the peat is perforated in a mould between spiked plungers heated preferably to 150—200°.

A. B. MANNING.

Vertical retort oven. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,738,743, 10.12.29. *Appl.*, 20.3.26).—A series of vertical coking chambers alternating with vertical heating flues extend along one side of a battery and the regenerators along the other. The structure is made up of sections, each comprising a set of vertical flues with the heating walls on either side and the corresponding regenerators. Between adjacent sections are expansion joints extending from the coking chamber to each of the outer ends of the sections, the joints being so arranged that any leak therein may be readily stopped. The joints in the outer wall adjacent to the coking chambers are staggered, and the adjacent surfaces in the wall between the chambers and regenerators are connected by vertically-extending tongue-and-groove joints. A. B. MANNING.

Apparatus for treating carbonaceous material. W. RHOADES (U.S.P. 1,743,394, 14.1.30. *Appl.*, 24.3.26).—Oil shale is treated in a retort the lower part of which forms a combustion chamber. The formation of clinker in the lower part of the combustion chamber is prevented by the rotation of two cylinders provided with curved teeth. These cylinders serve also to support the column of shale. Below them are two other rotatable cylinders provided with undercut pockets and adapted to discharge the spent shale as it is produced. The shale is fed continuously into the upper end of the retort, and a continuous updraft is maintained through the combustion chamber, the hot gases from which bring about the distillation of the material in the upper part of the retort.

A. B. MANNING.

[Steaming in] horizontal chamber ovens, especially coke ovens. O. Y. IMRAY. From N.V. SILICA EN OVENBOUW MIJ. (B.P. 327,442, 7.12. and 15.12.28).—Provision is made for introducing steam into the ovens through openings in the lower part of the heating walls. The steam ducts are so arranged that the steam enters the chamber in a downward direction, or recesses are provided in the chamber walls into which the steam ducts open. The ratio height: width of these recesses is less than corresponds with the angle of repose of the coal. The recesses preferably extend the whole length of the wall. The steam ducts are placed either in the partition walls between the regenerators or in the walls between the heating flues.

A. B. MANNING.

Dry-cooling of coke and the like. O. PIETTE (B.P. 327,819, 22.2.29).—The hot coke is delivered into a number of adjacent, inclined chambers having floors formed of shutter-like plates upon which the coke is spread in sufficiently thin layers. Between the floor plates are spaces through which a continuous current of non-oxidising gases is circulated in such a manner that they pass up through the layer of coke in the lower part and down through that in the upper part of each chamber. After passing through a dust-collecting receptacle, the now hot gases are brought into contact with the heating surface of a steam generator, and are then returned to the first cooling chamber.

A. B. MANNING.

Activated charcoal. F. MIK, Assr. to HOLZVER-KOHLUNGSIND. A.-G. (U.S.P. 1,743,975, 14.1.30. *Appl.*, 6.2.29. *Ger.*, 27.12.27).—A highly-activated charcoal is produced by carbonising hard wood when in the condition of "white rot," and subsequently activating the product by known means.

A. B. MANNING.

Preparation of highly active carbon. A.-G. F. STICKSTOFFDÜNGER (B.P. 315,810, 18.7.29. *Ger.*, 18.7.28. *Addn.* to B.P. 301,330; B., 1930, 449).—In the process of the prior patent, the sorting is effected by an elutriation process, either alone or in combination with a sifting process.

A. B. MANNING.

Manufacture of carbon-black from carbon monoxide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,548, 11.3.29).—In the production of carbon-black by the thermal decomposition of carbon monoxide

in the presence of a solid catalyst, about 3% by wt. of water vapour is added to the gas.

A. B. MANNING.

Manufacture of a material applicable as an adsorbent, comprising carbon, iron, and iron oxide. C. J. G. AARTS (B.P. 308,351, 14.3.29. Holl., 22.3.28).—The product, which can also be used as a pigment, a carburising agent, and for the manufacture of electrodes, is prepared by passing a gas containing carbon monoxide, but practically free from water vapour, over iron oxide distributed over dried carbonaceous material (charcoal, wood-wool, hay, etc.) at 350–450° and under conditions such that carbon dioxide forms 46–65% of the combined carbon monoxide and dioxide content of the issuing gas and the product contains, *e.g.*, 80–90% of carbon.

L. A. COLES.

Destructive hydrogenation. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 327,443, 31.12.28).—In the destructive hydrogenation of carbonaceous materials in the liquid phase the temperature of the hydrogenation vessel is regulated by circulation of part of the liquid material being treated through an external heater, without substantial release of the pressure and without the addition of hydrogen.

A. B. MANNING.

Production of acetic acid, wood naphtha, and other products of the distillation of wood and similar carbonaceous substances. P. POORE (B.P. 327,415, 2.1.29).—The yield of liquid products is increased by distributing superheated steam into the charge in the upper part of the retort. The steam is superheated by passing through a coil of piping within the retort, and adjacent to the wall thereof.

A. B. MANNING.

Combustible gas generator. W. J. MURDOCK, E. E. LUNGREN, and O. B. EVANS, ASSRS. to PIER PROCESS CORP. (U.S.P. 1,743,724, 14.1.30. Appl., 2.5.25).—The generator is rectangular or elliptical in horizontal cross-section, the width being not greater than 3–4 ft. The formation of a central inactive core of fuel is thereby avoided.

A. B. MANNING.

Gas producers. BAMAG-MEGUIN A.-G., and O. HELLER (B.P. 306,143, 14.2.29. Ger., 17.2.28).—Pulverised fuel is gasified in a current of preheated steam and gas, the apparatus comprising a heater for the current of steam and gas, and a reaction chamber. The mixture of fuel, steam, and gas is injected vertically up the centre of the reaction chamber from a nozzle in a hollow beam traversing the lower part of the chamber; the gases produced and the ash pass out through an exit at the bottom of the chamber. A relatively small volume of compressed gas is used to convey the fuel to the nozzle where it mixes with the steam and gas from the heater. The latter is of the regenerator type, and is heated intermittently by the combustion of producer gas therein. The fuel is supplied intermittently, but the current of conveying gas continuously, to the reaction chamber. The gases produced pass through a dry dust separator, and, except for the part which is by-passed for re-circulation, through a scrubber to a holder.

A. B. MANNING.

Manufacture of producer gas. W. J. MURDOCK, E. E. LUNGREN, and O. B. EVANS, ASSRS. to PIER PROCESS CORP. (U.S.P. 1,743,726, 14.1.30. Appl., 28.8.26. Renewed 7.9.29. Cf. U.S.P. 1,602,242; B., 1927, 35).—The producer is so designed that the fuel is disposed in an annular column between inner and outer refractory walls, all portions of the fuel being within an effective radiating distance, *e.g.*, 18–24 in., of the walls. Formation of a central impermeable mass of fuel is thereby avoided, and the entire fuel bed is blasted and processed uniformly with a minimum of channelling. The producer may be used with coals of relatively high volatile matter content.

A. B. MANNING.

Separation of solid particles from combustion gases. A. R. V. KEARSLEY (B.P. 327,622, 21.5.29).—The combustion gases pass through a casing in which the solid particles are thrown radially outward by a fan rotating about a horizontal axis. A receiver for the particles consists of a series of peripheral ducts formed by the spaces between wedge-shaped deflectors. A baffle plate behind each duct causes the particles to fall into a groove behind each wedge-shaped deflector, whence they pass to a convenient receptacle below. A fluid-spraying means may be provided at each duct to keep it clear.

A. B. MANNING.

Miner's safety lamp indicating the amount of firedamp in the air. G. SCHAULY (U.S.P. 1,744,416, 21.1.30. Appl., 10.8.27. Fr., 10.8.26).—A nickel-chromium thermocouple is supported above the flame of a miner's lamp. The elongation of the flame due to the presence of methane raises the temperature of the thermocouple, the potential of which is used to indicate the concentration of methane in the air.

A. B. MANNING.

Production of asphalt and road oil. J. F. FABER and M. L. CHAPPELL, ASSRS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,745,155, 28.1.30. Appl., 20.12.26).—Asphalts of high ductility at low temperatures are produced by distilling asphaltic-base oils in the presence of acid sludge obtained by treating lamp oil with fuming sulphuric acid or lubricating oil with acid of d 1.83. The oil should not contain more than 3–4% of wax and light distillates should be removed before adding the acid sludge. The sludge-oil mixture (5–30% of sludge) is heated at 285–340° under a pressure of 1–3 in. of mercury, with admission of steam. The product is free from sulphuric acid. An asphalt having a penetration of 50 at 25° is produced. By the admission of air during distillation higher-melting asphalts are produced.

T. A. SMITH.

Improvement of asphalt and bituminous masses. K. WINKLER (B.P. 307,466, 8.3.29. Ger., 8.3.28. Addn. to B.P. 307,465; B., 1930, 559).—The process described in the prior patent is modified by substituting fresh or used rubber, in solution if necessary, for a part of the drying oils.

A. B. MANNING.

Obtaining products from low-temperature and other tars and pitches. G. T. MORGAN and D. D. PRATT (B.P. 327,797, 4.2.29).—“Resinoic acids,” “resinamines,” and “resinenes” are isolated from tars and pitches by extracting the crude carboxylic acid, basic

and neutral fractions, respectively, with petrol, dissolving the residues in an organic solvent, *e.g.*, ether, benzene, or low-temperature tar spirit, and reprecipitating the desired products from the solution by the addition of petroleum. In treating the residues with the organic solvent part may remain insoluble; this is separately collected and forms a resinous material of high m.p. The products may be used to form the basis of stains, lacquers, etc.

A. B. MANNING.

Production of refined hydrocarbon oils and the like. I. G. FARBENIND. A.-G. (B.P. 327,463, 6.11.28. Addn. to B.P. 300,900; B., 1930, 499).—The impure oils are purified by treatment with hydrogen or gases containing hydrogen at temperatures above 300° and pressures above 10 atm. The reaction is carried out in the presence of catalysts of group VI which are immune from sulphur poisoning and do not contain iron, nickel, or cobalt. By the use of high pressures a greater throughput can be attained in a reaction vessel of given size. The temperature and pressure are also governed by the nature of the oil and its sulphur content. The tendency to produce low-boiling products is increased with the duration of the treatment. A crude lubricating oil, obtained from low-temperature tar and giving a high refining loss by the ordinary methods, is treated with hydrogen for 50 min. at 400° and 200 atm. in the presence of a zinc oxide, magnesia, and molybdic acid catalyst. Very little decomposition to low-boiling products takes place and little further refining is required. Motor fuels and burning oils may be similarly refined.

T. A. SMITH.

Removal of petrolatum from hydrocarbon oils. R. S. LANE and S. A. MONTGOMERY, ASSRS. to STANDARD OIL Co. (U.S.P. 1,744,648, 21.1.30. Appl., 6.3.25).—Wax-containing oils, such as cylinder stocks and lubricating oil distillates, are mixed with 1–2 times their vol. of a mixture of naphtha (35–60%), butyl alcohol (15–30%), and benzol (18–35%) and cooled to –6°. The layer containing wax settles out and may be separated either centrifugally or by gravity. The wax-free oil is freed from naphtha by distillation.

T. A. SMITH.

Treatment of [lubricating] oil. W. D. HARRIS and R. V. AYCOCK, ASSRS. to REFINOIL MANUFACTURING CORP. (U.S.P. 1,745,167, 28.1.30. Appl., 8.3.29).—Hot lubricating oil drawn from a still is passed upwards through a bed of fuller's earth. It is then allowed to settle and filtered downwards through the same bed. The sludge produced by the action of the fuller's earth on the oil forms a very efficient filter and further treatment is unnecessary. If the oil is passed only once through the earth the advantage of the improved filtering capacity of the earth is lost.

T. A. SMITH.

Distillation of refined, cracked oils without avoidable decomposition or discoloration. F. C. AXTELL, ASSR. to AXTELL RES. LABS., INC. (U.S.P. 1,738,518, 10.12.29. Appl., 8.2.27).—Acid-treated cracked spirits are distilled in the presence of soda ash (5–6 lb. per barrel). With this addition it is possible to distil out more gasoline without producing discoloration of the products or of the residue in the still.

T. A. SMITH.

Treatment of hydrocarbons. TAR & PETROLEUM PROCESS Co. (B.P. 311,689, 21.9.28. U.S., 14.5.28).—Hydrocarbons in liquid form, *e.g.*, tars, pitches, oil residues, are deposited at a controlled rate on to an externally heated horizontal surface and are coked thereon, the deposition being continued until a solid layer of metallurgical coke of substantial thickness has been formed. The surface may be made of heat-resistant glass or of a refractory material containing at least 60% Al_2O_3 . The hydrocarbons may first be distilled in the upper part of the chamber by causing them to flow in thin films over heated surfaces, *e.g.*, by delivering them into a rotating basket containing loose material, preferably coke. The volatile products are withdrawn from the chamber to a condensing system, and the liquid residue is coked as described above. The layer of coke is periodically discharged.

A. B. MANNING.

Treatment of vapours produced by cracking hydrocarbons. ANGLO-PERSIAN OIL Co., LTD., and A. E. DUNSTAN (B.P. 327,421, 4.10.28).—The vapours obtained in cracking processes, especially vapour-phase cracking, are passed through a filter charged with pumice which has been steeped in an alcoholic solution of zinc chloride. The filtering material is maintained at 200° and the gum-forming constituents of the spirit are polymerised. The polymerides are removed at the bottom of the filter and at the base of an adjacent dephlegmating tower through which the vapours leaving the filter are led. A distillate of good colour and odour and free from unstable compounds is obtained. For the treatment of 40 gals. of spirit 1 lb. of reagent is required, and the highest useful compression ratio is not materially altered. The filter is maintained at the required temperature by surrounding it with a jacket through which the vapours to be treated are passed, and the contents of the filter are occasionally flushed with refined spirit to maintain the activity of the filtering material.

T. A. SMITH.

Oxidation of organic compounds of high mol. wt. [paraffin wax]. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 327,707, 5.1.29).—In the oxidation of paraffin wax, montan wax, etc. to acids by air or oxygen and/or nitrogen oxides, the formation of hydroxyacids is avoided by gradually making the conditions milder as oxidation proceeds, *e.g.*, by lowering the temperature, diluting the oxidant, or decreasing the pressure or velocity of the gases.

C. HOLLINS.

[Paraffin] wax sweating and crystallising apparatus. H. L. ALLAN and J. MOORE, ASSRS. to BURMAH OIL Co., LTD. (U.S.P. 1,743,746. 14.1.30. Appl., 9.2.28. U.K., 26.3.27).—The apparatus consists of an upright vessel divided by transverse plates into a number of wax compartments. Each compartment is heated or cooled by a series of transverse coils of close pitch (not more than 2 in.) which act as a support for the wax during sweating. During cooling wax mixture is admitted to the upper portion of each compartment to compensate for the contraction of the mass and air is excluded. The wax mixture admitted at the top of each cell keeps fluid the wax at the base of the next cell above until sweating commences. Steam-pipes for steaming out the wax after sweating are fitted to the base of each cell.

T. A. SMITH.

Washing of coal and other minerals. A. FRANCE (U.S.P. 1,758,035, 13.5.30. Appl., 3.11.25. Belg., 23.4.25).—See B.P. 251,246; B., 1926, 971.

Generation of gases for power development. Improving the flowing characteristics of coal. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,758,630—1, 13.5.30. Appl., 25.10.26).—See B.P. 299,718; B., 1929, 311.

Production of liquid fuels. A. JOSEPH, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME HOUDRY (U.S.P. 1,758,796, 13.5.30. Appl., 11.7.27. Fr., 21.7.26).—See F.P. 632,360; B., 1930, 230.

Obtaining peat [briquettes]. M. N. KORELIN (B.P. 328,658, 30.10.28).

Installations for dry-cooling of hot coke. SULZER FRÈRES SOC. ANON. (B.P. 317,392, 16.3.29. Switz., 15.8.28. Addn. to B.P. 312,033).

Apparatus for cooling coke. HUMPHREYS & GLASGOW, LTD., Assees. of W. T. BOSLER (B.P. 328,887, 23.8.29. U.S., 4.10.28).

Burners for fuel in pulverulent condition. P. W. GRIFFIN (B.P. 328,682, 4.2.29).

Mixtures of solids and gases (B.P. 327,840). Fractionation (U.S.P. 1,738,386).—See I. Condensation of aromatic compounds (B.P. 327,382).—See III. Decolorising clays (U.S.P. 1,739,734). Hydrogen from methane (B.P. 328,048).—See VII. Roads, paths, etc. (B.P. 328,166).—See IX. Carbon black (B.P. 307,743).—See XIV.

III.—ORGANIC INTERMEDIATES.

Eutectic mixtures of alcohols and acetic esters. E. ALINARI (Annali Chim. Appl., 1930, 20, 159—167).—Owing to the formation of eutectic mixtures, fractional distillation at the ordinary pressure does not separate an ester (acetate) from the corresponding alcohol, except when the ester is in excess of the proportion present in the eutectic mixture, and thus forms a tail-product of the distillation. For the first three members of the homologous series of alcohols, separation from the acetates may be effected by washing the mixture with very concentrated calcium chloride solution, in which the alcohol is soluble but the acetate insoluble. In the first treatment with the chloride solution small quantities of the ester may be dissolved if a large proportion of the alcohol has passed into the calcium chloride, as this alcohol acts as a solvent for the acetate. In the successive treatments, however, the losses of ester are very small, so that a good yield of high-grade ester is obtainable. The treatment is continued until the volume of the residual ester remains practically unchanged. For mixtures of the higher alcohols, such as propyl, and their acetates treatment with calcium chloride solution is useless, but four successive washings of the eutectic mixture, containing about 55% of propyl acetate, with water yields about 50% by vol. of a product containing about 88% of the ester. B.p. curves are given for mixtures of methyl, ethyl, propyl, *n*-butyl, and isoamyl alcohols with their respective acetates. The b.p. of methyl iodide (lit. 44—45°,

42.3—42.8°) is lowered by the presence of small proportions of methyl alcohol, owing to the formation of the compound 3MeI.MeOH (b.p. 40°). T. H. POPE.

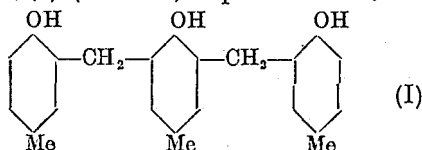
Amination of 2:5-dichloronitrobenzene. L. McMASTER and A. STEINER (Ind. Eng. Chem., 1930, 22, 547—548).—In the formation of *p*-chloro-*o*-nitroaniline (m.p. 115—116°) from 2:5-dichloronitrobenzene (25 pts.) and aqueous ammonia (140 pts. of 28% concentration) a maximum yield of 93% was obtained by heating with constant stirring for 2 hrs. at 170° and 1627 cm. pressure without catalysts such as vanadium pentoxide or copper salts. D. F. TWISS.

Preparation of *o*- and *p*-xenylamines (amino-diphenyls). G. T. MORGAN and L. P. WALLS (J.S.C.I., 1930, 49, 15 t).—When required for various synthetic purposes *o*-aminodiphenyl has generally been prepared by means of the Heusler-Hirsch reaction, which involves a pyrolysis of diazoaminobenzene in presence of aniline. This method, which gives a small yield, is unduly laborious when large quantities of the base are required, and accordingly the following processes have been employed, by means of which both *o*- and *p*-aminodiphenyls (*o*- and *p*-xenylamines) can be readily prepared from technically available diphenyl. Nitration to a mixture of *o*- and *p*-nitrodiphenyls was effected in glacial acetic acid followed by a separation of these isomerides based on difference in solubility in the nitrating medium and distillation of the *o*-compound under reduced pressure. The two xenylamines (*o*- and *p*-aminodiphenyls) were prepared by reducing the corresponding nitrodiphenyls with iron and acidified water and purified by distillation under reduced pressure.

Formaldehyde condensations with aromatic compounds. G. T. MORGAN (J.S.C.I., 1930, 49, 245—251 t).—The formaldehyde condensations with aromatic amines, phenols, and aromatic hydrocarbons are reviewed. The reactions whereby the methylene radical becomes attached to the aromatic nuclei of the amines are utilised in the manufacture of such dyes as the magentas, acridine-orange, and auramine, and in the production of certain synthetic drugs including proflavine and acriflavine. The processes of preparing ionamine dyes and indigo from phenylglycine also involve the intervention of a formaldehyde condensation. In the basic series the introduction of the methylene group is more useful industrially than the addition of the radical CH.OH, but in the phenolic group both these types of condensations are employed; the former is more characteristic of acidic condensations, the latter of alkaline condensations. Resins of the Novolak type are obtained in the former case, those of Bakelite type in the latter. The case of *m*-cresol was studied in detail. The phenolic constituents of low-temperature tar are readily amenable to formaldehyde condensations, furnishing both soluble and insoluble resinoids. The aromatic hydrocarbons, and especially acenaphthene, are generally capable of condensing with formaldehyde in acidic media.

Formaldehyde condensations with phenol and its homologues. N. J. L. MEGSON and A. A. DRUMMOND (J.S.C.I., 1930, 49, 251—257 t).—Resinous condensations of phenols with formaldehyde are performed

in volatile organic solvents as controlling media, using both acidic and alkaline condensing agents. With hydrochloric acid in alcoholic solution, and subsequent steam extraction, phenol and paraformaldehyde yield 4:4'- and 2:4'-dihydroxydiphenylmethane (identified by the diacetate and diethyl ether). The yield of the 2:4'-isomeride is shown to decrease with increase of resin, that of the 4:4'-isomeride remaining constant. Bromination of the 2:4'-isomeride gives 3:5:3':5'-tetrabromo-2:4'-dihydroxydiphenylmethane, m.p. 199°. Under similar conditions, *o*-cresol yields 4:4'-dihydroxy-3:3'-dimethyldiphenylmethane (identified by dibromo-derivative), *m*-cresol yields needles, m.p. 161°, probably 4:4'-dihydroxy-2:2'-dimethyldiphenylmethane, in minute amount. *p*-Cresol yields needles, m.p. 124°, 2:2'-dihydroxy-5:5'-dimethyldiphenylmethane (diacetate, m.p. 124°; bromo-derivative, m.p. 202°), and prisms, m.p. 214°, insoluble in benzene, 3:5-bis-2-hydroxy-5-methylbenzyl-*p*-cresol (I) (triacetate, m.p. 117–119°; dibromo-



derivative, m.p. 175°), in addition to a resin soluble in benzene, and an amorphous powder of unknown constitution. The yields of needles and prisms are compared with resin formation, both increasing to a maximum, and then falling off. Diethylal is isolated from distillation of the alcoholic solvent in all the above cases. Using various controlling solvents, phenol and formaldehyde in presence of alkali under varying conditions of concentration yield saligenin and *p*-hydroxybenzyl alcohol. *m*-Cresol yields a homosaligenin, m.p. 108°, probably 2-hydroxy-4-methylbenzyl alcohol. 1:3:5-Xylenol yields xylenol dialcohol. All these phenols give heat-sensitive resins, caustic alkalis causing their formation more readily than mild alkalis. A method of removing condensing agent from a solution of resin consists in repeated washings with saturated aqueous solutions of certain inorganic salts (*e.g.*, potassium carbonate, sodium carbonate, sodium thiosulphate). Purified resinous products are thus prepared for application to electrical insulation. Fractionation of the heat-sensitive resins is brought about through precipitation of the resin from solution, by a non-solvent for the resins (*e.g.*, benzene, chloroform), which retains crystalline material in solution. Such precipitation may take place on to an inert pulverulent substrate (*e.g.*, sand, powdered glass, anhydrous sodium sulphate), and the latter may be extracted with successive quantities of ethyl acetate and acetone. From a *m*-cresol resin ethyl acetate removes a syrup hardening at 100°, and acetone removes an amorphous powder hardening after some hours at 50–60°.

Friedel-Crafts reactions, naphthalene series. I. Preparation of naphthanthraquinone. P. H. GROGINS and H. P. NEWTON (Ind. Eng. Chem., 1930, 22, 157–159).—A detailed study of the effects of the following factors: (1) reaction solvent and temperature, (2) ratio of naphthalene to phthalic anhydride, (3) ratio of anhydrous aluminium chloride to phthalic anhydride,

(4) conditions of hydrolysis of the reaction product, and (5) the conditions of formation of the ammonium salt and precipitation of α -naphthoylbenzoic acid, on the purity and yield of the product in the preparation of 1:2-benzanthraquinone from phthalic anhydride and naphthoic acid and subsequent dehydration of the α -naphthoylbenzoic acid so formed, is described. On the basis of the results the following technique is developed. A 97.5% yield of α -naphthoylbenzoic acid, m.p. 168–170° (purity 99.2%) [pure acid has m.p. 174.4° (corr.)] is obtained by condensing equimolecular proportions of naphthalene and phthalic anhydride in 3 pts. of *o*-dichlorobenzene with a 10% excess of aluminium chloride at 0°. Hydrolysis of the reaction mixture is effected by adding it gradually to dilute sulphuric acid with non-swirling agitation at 0°. After steam-distillation of the solvent (99% recovery) the crude acid is filtered, dissolved in dilute ammonia, the solution heated with vegetable carbon and filtered, and the cool filtrate fed slowly into cold dilute sulphuric acid with continuous agitation. Coagulation of the precipitated acid occurs if the ammonium salt solution is added too rapidly, or is too warm, or if the sulphuric acid is too concentrated. The only effective reagent to bring about ring-closure of the α -naphthoylbenzoic acid to benzantraquinone is 6 pts. of 95% sulphuric acid for 7 hrs. at 80°, at which temperature considerable sulphonation occurs normally, but is prevented by addition of boric acid (equal weight to that of the α -naphthoylbenzoic acid) as an inhibitor. Sulphonation is increased by use of a higher acid ratio or by a decrease in the proportion of boric acid used, whilst a lower acid ratio tends to result in incomplete ring-closure. The reaction mixture is diluted with 20% sulphuric acid to a residual acidity of 50%, cooled to 25°, and the crude product filtered, washed successively with 50% sulphuric acid and hot water, digested with 2% sodium hydroxide solution, washed, and dried. Under these conditions a 94% yield of benzantraquinone, m.p. 168°, is obtained, the degree of purity being unaffected by the use of either pure or the crude α -naphthoylbenzoic acid.

J. W. BAKER.

Cracking of tar acids from coal. MORRELL and EGLOFF.—See II. **Analysis of formic esters.** TOCCO and NYSENS.—See V. **Tests for peroxides in ethyl ether.** PINES. **Testing etc. of paraldehyde.** SCHULEK.—See XX.

PATENTS.

Manufacture of condensation products [flotation agents containing nitrogen and sulphur] from acetylene. I. G. FARBENIND. A.-G. (B.P. 316,282, 30.1.29. Ger., 27.7.28).—Acetylene and ammonia are passed at 300–400° with hydrogen sulphide or sulphur over a metal or metal sulphide (copper sulphide on coke, copper and cobalt sulphides on zinc oxide) to give products containing nitrogen and sulphur. A product of b.p. 120–300° is suitable for ore flotation.

C. HOLLINS.

Manufacture of formaldehyde. A. J. WEITH, V. E. MEHARG, and H. W. AHLBECK, Assrs. to BAKELITE CORP. (U.S.P. 1,738,745, 10.12.29. Appl., 29.10.27).—Ammonia is added to the mixture of methyl alcohol

vapour and air to prevent an acid reaction; or the product may be neutralised before distillation.

C. HOLLINS.

Manufacture of alcohols [from aldehydes or ketones]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,573, 6.4.29).—Aldehydes or ketones are reduced with gas mixtures not rich in hydrogen (less than 80%), freed from carbon monoxide, sulphur, etc., at 20–30 atm. in presence of an appropriate catalyst. *E.g.*, acetaldehyde at 30–35° and 20 atm. is reduced to ethyl alcohol by 80% hydrogen in presence of nickel or copper on kieselguhr; aldol gives *n*-butyl alcohol and a little γ -butylene glycol.

C. HOLLINS.

Catalysts for production of ketones. HOLZVERKOHLLUNGS-IND. A.-G. (B.P. 315,818, 10.1.29. Ger., 19.7.28).—Sponge iron or iron turnings are allowed to rust in the presence of acetic acid or calcium acetate and the product is mixed with a paste of manganese carbonate or of chromic or aluminium hydroxide and the mixture is heated at 500–550°. The mass is an efficient catalyst for the conversion of unsaturated aliphatic hydrocarbons and of aldehydes and alcohols into ketones in the presence of steam at 450–550°. [Stat. ref.]

A. R. POWELL.

Carrying out condensation reactions with non-aromatic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,411, 27.11.28).—A non-aromatic hydrocarbon is condensed with an organic compound containing halogen, alkoxyl, or carbalkoxyl substituents in presence of aluminium chloride etc., the reaction being performed at raised pressures. Polymethylene ketones are obtained from petroleum of b.p. 60–70°, carbonyl chloride, and aluminium chloride at 100°; *n*-butane similarly gives an oil, b.p. 120–150°/15 mm. Hexane is condensed with acetyl chloride or ethyl chloride, ethylene with methyl chloride or with the double compound of ether and boron fluoride, cyclohexane with benzoyl chloride.

C. HOLLINS.

Separation of anhydrous acetic acid from its aqueous solutions. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 317,462, 21.12.28. Fr., 17.8.28).—An accessory liquid (petroleum fraction of b.p. 114–116°) which forms an azeotropic mixture with acetic acid is added to the anhydrous mixture of solvent (amyl acetate) and acetic acid obtained by the method of B.P. 296,974 (B., 1929, 349), and on distillation a mixture of anhydrous acid and accessory liquid, separable by decantation (especially after addition of about 1% of water), is collected, whilst the extracting solvent remains behind. The solution of acid in amyl acetate may, *e.g.*, be introduced at the middle of a heated tower down which flows the petroleum. Vapours of acetic acid and petroleum pass from the top of the tower to a condenser and receiver, where the desired amount of water is added and the acid decanted. The amyl acetate, unvaporised, is collected at the foot of the tower.

C. HOLLINS.

Removal of water from aqueous acetic acid. KODAK, LTD. From EASTMAN KODAK Co. (B.P. 327,444, 3.1.29).—Ethylene dichloride is used as entraining liquid in the distillation of water from aqueous acetic acid. The hot aqueous acid may be supplied to a

fractionating column, ethylene dichloride being introduced at the top in large excess (9 pts. for each pt. of water to be removed); water and ethylene dichloride are separated in the receiver after condensation, whilst the mixture of acetic acid and excess ethylene dichloride drawn from the base of the column is rectified separately. Suitable apparatus is figured.

C. HOLLINS.

Manufacture of methylamine [from hydrogen cyanide]. G. BARSKY, ASSR. to AMER. CYANAMID Co. (U.S.P. 1,736,872, 26.11.29. Appl., 30.3.26).—Hydrogen cyanide and large excess of hydrogen are passed over platinum-black on asbestos at 150–180° and at 50–100 lb./in.², the warm exit gases being scrubbed with cold acid and returned to the circuit. After about 10 days' working, during which the temperature should be raised 1½° per day from 165° to 180°, the catalyst is re-activated by washing it with 30% nitric acid. The gas mixture is conveniently produced by passing hydrogen through liquid hydrogen cyanide cooled in ice-water. Very little tar formation occurs. A suitable plant is described.

C. HOLLINS.

Manufacture of ethyl acetate. I. G. FARBENIND. A.-G. (B.P. 307,471, 8.3.29. Ger., 8.3.28).—A mixture of ethyl alcohol, excess of acetic acid, and a little sulphuric acid is distilled through a column. The lower (aqueous) layer in the receiver is run off, and the upper (alcohol and ester) layer is returned to the top of the column until its content of ester attains a maximum. Thereafter alcohol is run into the still gradually and a corresponding quantity of ester is drawn off from the receiver, the remainder being returned to the column as before. Finally the small residue of acetic acid is esterified by adding excess of alcohol and the distillate from this is added to the next batch.

C. HOLLINS.

Production of isopropyl alcohol. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 327,224, 25.1.20).—Acetone vapour is hydrogenated at 180° in presence of a copper-nickel catalyst prepared as in B.P. 316,399 (B., 1929, 806).

C. HOLLINS.

Esterification of polyvalent alcohols or their derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 327,165, 22.11.28).—The products (red compounds of unknown constitution) obtained by treating organic acid chlorides with pyridine to remove hydrogen chloride are used as esterifying agents for cellulose, glycerol, etc. Acid chlorides mentioned are lauryl, acetyl, naphthenic, and phenylacetyl.

C. HOLLINS.

Manufacture of urea [carbamide]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,026, 15.4.29).—Ammonia gas (7 pts.) and carbon oxysulphide (4 pts.) are liquefied in a pressure vessel and heated at 120° (pressure 18–36 atm.). Carbamide is obtained in 70–78% yield without isolation of the thiocarbamate.

C. HOLLINS.

Condensation of aromatic compounds with olefinic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,382, 28.11.28).—Olefines condense with aromatic, hydroaromatic, or aliphatic compounds at 300–400° and 15–50 atm. pressure in presence of cerium metaphosphate or other phosphate,

borate, or arsenate not reduced by hydrogen under these conditions. Products from ethylene and naphthalene, toluene, soft paraffin wax, stearin, aniline (giving higher arylamines and heterocyclic bases), phenol, chlorobenzene, tetra- and deca-hydronaphthalenes, gas oil, anthracene, of propylene with toluene, and of styrene with naphthalene, are described.

C. HOLLINS.

Manufacture of *m*-2-xylidine. IMPERIAL CHEM. INDUSTRIES, LTD., and J. KENNER (B.P. 327,830, 5.3.29).—*m*-2-Xylidine nitrate separates out substantially free from isomerides when the mixed xylidines remaining after removal of most of the *m*-4-xylidine as sulphate are dissolved in hot dilute nitric acid and the solution is allowed to cool.

C. HOLLINS.

Manufacture of pure benzoic acid. I. G. FARBENIND. A.-G. (B.P. 307,343, 18.2.29. Ger., 5.3.28).—Crude benzoic acid from phthalic acid or anhydride is pasted with water and stirred with sodium hydrogen sulphite at 40–50° to reduce the naphthaquinones and hydrate the phthalic anhydride present; the reduction products etc. are removed by washing with water, leaving benzoic acid of 99% purity. Other reducing agents may be used.

C. HOLLINS.

Preparation of *o*-(*p*-sulphobenzoyl)benzoic acid derivatives and anthraquinone compounds derivable therefrom. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN (B.P. 327,130, 24.9.28).—*o*-(*p*-Sulphobenzoyl)benzoic acid is nitrated in sulphuric acid at 35–38° to give 4-nitro-4'-sulphobenzophenone-2-carboxylic acid, which is reduced with iron and acetic acid to the 4-amino-compound. The latter on cyclisation in sulphuric acid yields 2-aminoanthraquinone-7-sulphonic acid, from which by the action of ammonia in presence of ammonium arsenate at 180° 2:7-diaminoanthraquinone, m.p. 330–332°, is obtained. 2-Nitroanthraquinone-7-sulphonic acid, from the nitro-ketone, is converted into 7-chloro-2-nitroanthraquinone, m.p. 251–252°, reducible to 7-chloro-2-aminoanthraquinone, m.p. 302–303°.

C. HOLLINS.

Manufacture of *o*-(aminoaroyl)benzoic acids and inner anhydrides thereof. I. G. FARBENIND. A.-G. (B.P. 305,593, 7.2.29. Ger., 7.2.28).—*N*-Arylphthalimides are transformed by heating with sodium aluminium chloride etc. into lactams of 2-*o*-aminobenzoylbenzoic acids, from which the free acids are readily obtained by hydrolysis. The preparation of 2-(*o*-aminobenzoyl)benzoic acid, m.p. 195° (decomp.) (lactam, m.p. 245–246°); 2-(4'-chloro-2'-aminobenzoyl)benzoic acid, m.p. 204–205° (decomp.) (lactam, m.p. about 300°); and the corresponding 3'-chloro- (acid, m.p. 176°; lactam, m.p. 293°), 6'-chloro- (acid, m.p. 188°; lactam), 3(?)-chloro- [acid, m.p. 168° (decomp.)], 4'-bromo- [acid, m.p. 170° (decomp.)]; lactam, m.p. 285°], and 4'-methyl [m.p. 194° (decomp.)]; lactam, m.p. 238–240°] derivatives is described.

C. HOLLINS.

Manufacture of esters of *O*-arylated or *O*-aralkylated bivalent [dihydric] alcohols [perfumes]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 327,705, 29.12.28).—Ethylene or γ -propylene glycol monoaryl or monoaralkyl ethers are treated with acylating agents derived from acids other than formic

and acetic. The following compounds are described: β -phenoxyethyl propionate, b.p. 121°/4 mm. (odour resembling hyacinth), butyrate, m.p. 129–131°/4 mm., isobutyrate, b.p. 125–127°/4 mm., isovalerate, b.p. 135–136°/4 mm., benzoate, m.p. 64°, and cinnamate, m.p. 64°; β -*p*-tolylloxyethyl isobutyrate, b.p. 124–125°/3 mm.; β -*o*-tolylloxyethyl isobutyrate, b.p. 128–130°/4 mm.; β -benzylloxyethyl isobutyrate, b.p. 134–137°/5 mm.; γ -phenoxypropyl isobutyrate, b.p. 138–141°. The products find application in perfumes.

C. HOLLINS.

Manufacture of derivatives of aldehyde-amine reaction products [vulcanisation accelerators]. W. SCOTT, ASSR. to RUBBER SERVICE LABS. CO. (U.S.P. 1,737,391, 26.11.29. Appl., 18.5.26).—Dibenzylamine is condensed with formaldehyde, and the product is treated with carbon disulphide to give a vulcanisation accelerator.

C. HOLLINS.

Manufacture of aldehyde-amine reaction products [vulcanisation accelerators]. C. O. NORTH and W. SCOTT, ASSRS. to RUBBER SERVICE LABS. CO. (U.S.P. 1,737,384, 26.11.29. Appl., 8.4.26).—Butyldenedianiline, $\text{CHPr}(\text{NHPH})_2$, is condensed with heptaldehyde (1 mol.) below 50° to give a vulcanisation accelerator. Other alkylidenedianilines may be used.

C. HOLLINS.

Manufacture of heterocyclic bases of the amino-iminazoline [aminodihydroglyoxaline] series. I. G. FARBENIND. A.-G. (B.P. 310,534, 24.4.29. Ger., 27.4.28).—2-Thiol-4:5-dihydroglyoxaline ("ethylenethiourea") is treated with methyl iodide or sulphate and the product is heated with a primary or secondary amine (e.g., *p*-toluidine) at 130°, with or without a solvent (amyl alcohol). The *p*-toluidino- (m.p. 133–134°), methylanilino- (m.p. 131°), and anilino- (m.p. 122°) derivatives are described. They are vulcanisation accelerators.

C. HOLLINS.

Manufacture of [hydroxy-]carbazole derivatives. I. G. FARBENIND. A.-G. (B.P. 303,520, 4.1.29. Ger., 5.1.28).—Unlike 3-aminocarbazole, the 1- and 2-isomerides may be converted by the usual method (boiling the diazo compounds with water) into the corresponding hydroxycarbazoles. [Stat. ref.] C. HOLLINS.

Manufacture of hydroxycarboxylic acids of carbazole. I. G. FARBENIND. A.-G. (B.P. 303,901, 11.1.29. Ger., 12.1.28).—Hydroxycarbazoles are treated with alkali and carbon dioxide under pressure. 2-Hydroxycarbazole, potassium hydrogen carbonate, and carbon dioxide under 25 atm. pressure at 275° yield a separable mixture of 2-hydroxycarbazole-3-carboxylic acid, m.p. 273–274° (decomp.) (acetyl derivative, m.p. 227°; *o*-anisidide, m.p. 204–206°), and the isomeric 1-carboxylic acid, m.p. 271–272° (*o*-anisidide, m.p. 192–193°). The latter gives an indophenol with *p*-nitrosophenol.

C. HOLLINS.

Manufacture of nitrohalogenoquinazolines [2- or 4-halogeno-*Bz*-nitroquinazolines]. A. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,450, 4.1.29).—A *Bz*-nitro-2- or -4-hydroxyquinazoline is treated with phosphorus penta-chloride or -bromide, or with phosphoryl chloride. The preparation of 4-chloro-6-nitro- (m.p. 128°), 4-chloro-7-nitro- (m.p.

148°, sublims 200—230°, 4-chloro-6-nitro-7-methyl- (m.p. 100°, b.p. about 190°), and 4-chloro-7-nitro-2-phenyl-quinazolines, and of 6-nitro-4-hydroxy-7-methyl-quinazoline, m.p. 278°, and 7-nitro-4-hydroxy-2-phenyl-quinazoline, m.p. 240—250°, is described. The products have therapeutical application and serve as agents for combating insect pests. C. HOLLINS.

Manufacture of perylenetetracarboxylic anhydride. F. BENSA (B.P. 309,163, 11.2.29. Austr., 6.4.28).—The dyes of B.P. 278,325 (B., 1928, 399), obtained by the action of cuprous cyanide on diacetyl or dipropionyl derivatives of 3:9-dichloroperylene, are oxidised almost quantitatively to perylenetetracarboxylic anhydride by dichromate or permanganate. C. HOLLINS.

Preparation of thymol and its isomerides. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 308,681, 14.2.29. Ger., 26.3.28. Addn. to B.P. 326,215; B., 1930, 532).—*iso*Propylated or propylated *m*-cresols are isomerised without catalysts at 330—400°, the chief products being thymol, m.p. 51°, b.p. 232°, and an isomeride, m.p. 114°, b.p. 245—246°, which itself undergoes partial conversion into thymol at 380°. C. HOLLINS.

Manufacture of menthane. SCHERING-KAHLBAUM A.-G. (B.P. 327,924, 16.7.29. Ger., 8.6.29).—Dipentene is hydrogenated in presence of a nickel catalyst at elevated temperatures and pressure. C. HOLLINS.

Manufacture of derivatives of 2:3-hydroxy-naphthoic acid. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,971, 26.2.29).—2:6-Dihydroxy-3-naphthoic acid is treated with ammonia or a primary or secondary amine to give 6-amino-2-hydroxy-3-naphthoic acids. The 6-amino- (m.p. 295°), 6-methylamino-, 6-anilino- (m.p. 222—223°), 6-methylanilino-, and 6-benzylamino-compounds are described. C. HOLLINS.

Purification of crude aromatic hydrocarbons [anthracene, phenanthrene]. SELDEN Co., Assees. of A. O. JAEGER (B.P. 299,419, 8.10.28. U.S., 26.10.27).—Crude anthracene is mixed with air and passed over heated catalysts for preferential oxidation of carbazole and aliphatics; the phenanthrene may subsequently be removed by solvents. Suitable catalyst mixtures are: ilmenite and potassium hydroxide on pumice; ceria and potassium nitrate on pumice or aluminium; precipitated ferric oxide and titania with potassium hydroxide on pumice; etc. [Stat. ref.] C. HOLLINS.

Manufacture of anthraquinone derivatives. CHEM. WORKS, FORMERLY SANDOZ (CHEM. FABR. VORM. SANDOZ) (B.P. 308,713, 13.2.29. Ger., 27.3.28).—Diamino-anthra-rufin- and -chrysazin-2-sulphonic acids are tetramethylated by treatment with formaldehyde (3.5 mols.) in presence of a little sulphuric acid; formic acid is unnecessary. Excess of formaldehyde tends to split off the sulphonic acid group and should be avoided. C. HOLLINS.

Manufacture of aralkylbenzanthrones. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 327,526, 27.2.29).—Benzanthrones containing a free 3-position are treated with benzyl chlorides in presence of copper and a diluent or with a *p*-substituted benzyl alcohol in

presence of sulphuric acid. 3-Benzyl (m.p. 187°), 3-*p*-nitrobenzyl, 6:11-dichloro-3-benzyl (m.p. 207°), and 3-*m*-nitro-*p*-methoxybenzyl (m.p. 227°) derivatives of benzanthrone are described. C. HOLLINS.

Manufacture of stable leuco-indigo preparations. IMPERIAL CHEM. INDUSTRIES, LTD., A. DAVIDSON, A. J. HAILWOOD, F. HENESEY, and A. SHEPHERDSON (B.P. 327,864, 15.4.29).—Dispersed indigo of particle size 3 μ or less is reduced with 0.6—1.0 mol. of dextrose and 4—7 mols. of caustic alkali. The leuco-indigo paste so prepared is stable and is suitable for feeding fermentation vats. C. HOLLINS.

Manufacture of substituted arylsulphonic acids. K. DAIMLER and G. BALLE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,758,277, 13.5.30. Appl., 18.3.26. Ger., 1.4.25).—See B.P. 250,241; B., 1927, 470.

Preparation of nuclear-substituted arylsulphonic acids. K. DAIMLER and G. BALLE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,758,356, 13.5.30. Appl., 16.3.27. Ger., 27.3.26).—See B.P. 268,375; B., 1927, 743.

Diazo salt preparation. F. KELLER and K. SCHNITZSPAHN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,912, 13.5.30. Appl., 8.12.25. Ger., 27.12.24).—See B.P. 273,352; B., 1927, 647.

Condensation products of the benzodiazine series. W. HENTRICH and M. HARDTMANN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,792, 13.5.30. Appl., 14.12.27. Ger., 13.12.26).—See B.P. 309,102; B., 1929, 935.

Catalytically reducing anthraquinone compounds and hydrogenated products derived therefrom. J. VON BRAUN and O. BAYER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,381—2, 13.5.30. Appl., 10.6.27. Ger., 3.3.25).—See B.P. 248,759; B., 1927, 597.

Manufacture of 2:7-dinitroanthraquinone. R. E. SCHMIDT and B. STEIN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,855, 13.5.30. Appl., 28.6.26. Ger., 25.6.25).—See B.P. 291,886; B., 1928, 598.

Acetic acid from wood etc. (B.P. 327,415). **Oxidation of organic compounds** (B.P. 327,707).—See II. **Condensation products from carbazoles and olefines** (B.P. 327,746).—See XIII. **Fermentation of starch etc.** (B.P. 328,723).—See XVIII. **Ketones** (B.P. 325,669).—See XX.

IV.—DYESTUFFS.

Formaldehyde condensations with aromatic compounds. MORGAN.—See III. **Arsenic in food-colouring materials.** ANON.—See XIX.

PATENTS.

Manufacture of basic dyes [of the acridine series]. DURAND & HUGUENIN A.-G. (B.P. 316,315, 26.7.29. Ger., 28.7.28).—In the nitration of tetramethyl- or tetraethyl-diaminodiphenylmethane for Acridine-orange, additional nitric acid (*e.g.*, 4 mols. in place of 2) is used, whereby an acridine is eventually obtained which gives red-brown or copper-red shades on tannin-mordanted cotton. The methyl groups appear to be attacked. C. HOLLINS.

Dyes and dyeing [soluble esters of leuco-vat dyes]. R. S. BARNES, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 327,672, 4.10.28).—The sodium salts of acid sulphuric esters of leuco-vat dyes or of other leuco-quinones are converted into quaternary ammonium salts by double decomposition, by acidification, followed by neutralisation with a quaternary ammonium hydroxide with or without isolation of the free acid ester, or by conversion into pyridine salt followed by treatment with alkyl halide.

C. HOLLINS.

Preparation of dyes of the anthraquinone series [for wool and acetate silk]. IMPERIAL CHEM. INDUSTRIES, LTD., F. LODGE, and W. W. TATUM (B.P. 326,949, 8.2.29).—1:4-Diaminoanthraquinone-2-sulphonic acids are heated with alkali and a polyhydric alcohol to give (probably) hydroxy-ethers. 1:4-Diaminoanthraquinone-2:3-disulphonic acid with glycerol gives a bluish-red dye for acetate silk; glycerol or glycol and 1-amino-4-anilinoanthraquinone-2-sulphonic acid (violet on wool), "triethanolamine" and 1-amino-4-anilinoanthraquinone-2-sulphonic acid (reddish-blue on acetate silk) are similarly condensed.

C. HOLLINS.

Manufacture of vat dyes [of the anthraquinone-acridone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,758, 17.12.28).—Anthraquinoneacridones containing both chlorine and bromine are bluish-red vat dyes fast to light. Products from brominated anthraquinone-2:1-acridone and sulphuryl chloride or chlorine, and from chloroanthraquinone-2:1-acridone and bromine are described. [Stat. ref.]

C. HOLLINS.

Manufacture of anthanthrone derivatives [vat dyes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,712, 9.10.28. Cf. B.P. 325,797; B., 1930, 501).—Diaminoanthanthrones are acylated or otherwise converted into *N*-substituted derivatives. Diaminoanthanthrone, synthesised from 5-nitro-8-amino-1-naphthoic acid, is dibenzoylated for a bluish-red vat dye; the diamine obtained by reducing dinitrated anthanthrone is condensed with benzoyl chloride (rose-red), *m*-methoxybenzoyl chloride, 1-chloroanthraquinone (grey), and 1-aminoanthraquinone-2-carboxyl chloride (blue-red) for vat dyes.

C. HOLLINS.

Manufacture of vat dyes [of the dibenzpyrene-quinone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,756, 18.10.28, 18.4. and 19.7.29).—1:2:6:7-Dibenzpyrene-3:8-quinone or a derivative is halogenated in chlorosulphonic acid etc. in presence of a carrier under mild conditions, e.g., with chlorine at 50°, bromine at 60°, or iodine at 110°. At the higher temperatures the time of reaction must be shortened by using excess of halogen or of carrier (e.g., 1.5% of sulphur or iodine). Thirty-three examples of special conditions for the production of clear, golden-yellow to orange vat dyes are described. C. HOLLINS.

Manufacture of vat dyes [of the isodibenzanthrone series] containing nitrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,141, 27.12.28).—Vat dyes, differing from the aminoisodibenzanthrones of B.P. 23,052 of 1910 (B., 1911, 739) in that they are stable to hypochlorite, are obtained by the action of

hydroxylamine or isodibenzanthrones, with or without simultaneous or subsequent alkylation, acylation, or halogenation. *iso*Dibenzanthrone and hydroxylamine sulphate in presence of sulphuric acid and ferrous sulphate at 165–170° give a dark blue vat dye completely fast to chlorine; the presence of methyl sulphate leads to a redder dye, whilst benzoylation gives a navy-blue. Dibromo- and dichloro-*isobenzanthrones* give blue vat dyes.

C. HOLLINS.

Manufacture of nitrogenous derivatives of pyranthrone [vat dyes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,734, 9.10.28. Cf. B.P. 325,250; B., 1930, 453).—*N*-Substituents are introduced into amino- or diamino-pyranthrone by condensation, especially with vatable compounds. Aminopyranthrone gives with 1-chloroanthraquinone an olive-brown vat dye, with monobrominated dibenzanthrone a blue-grey to blue-black; a blue-red vat dye is obtained from diaminopyranthrone and 1-amino-2-aldehydoanthraquinone.

C. HOLLINS.

Manufacture of a vat dye [sulphonated indanthrone]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,007, 25.3.29).—Pure indanthrone, obtained, e.g., by the process of B.P. 184,193 (B., 1924, 49), is monosulphonated with 100% sulphuric acid at 120° in presence of boric acid. [Stat. ref.]

C. HOLLINS.

Manufacture of vat dye preparations. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,087, 25.3.29. Cf. B.P. 320,397; B., 1930, 277).—Mixtures of a pure sulphonic acid of indanthrone and preferably pure indanthrone containing 3–60% of the former have good fastness properties and strength. C. HOLLINS.

Iodination of organic compounds capable of being vatted. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,702, 29.10.28 and 6.4.29).—Vatable compounds are iodinated with iodine, iodine monochloride, etc. in sulphuric acid (monohydrate or weak oleum), perchloric acid, periodic acid, or phosphoric acid, in presence of carriers if desired. Vat dyes are prepared by iodination of anthanthrone (orange), bromoanthanthrone (red), chlorobromoanthanthrone (orange-red), pyranthrone (reddish-orange), dibenzanthrone (blue-violet), isobenzanthrone (violet), bromo-2:3:6:7-dibenzpyrene-1:8-quinone (blue-red), indanthrone (greenish-blue), 1:2:6:7-diphthaloylacridone (red-orange), benzanthronepyrazolanthrone (navy-blue), *ms*-anthradianthrone (yellow-orange), *allo-ms*-naphthadianthrone (orange), 6:6'-dichloro-4:4'-dimethylthioindigo (red-violet), and perylene (grey). Anthraquinone is similarly iodinated.

C. HOLLINS.

Manufacture of organic iodo-halogen compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,711, 29.10.28 and 20.6.29. Cf. B.P. 327,702, preceding).—Vatable iodinated compounds are brominated or chlorinated especially in sulphuric or chlorosulphonic acid. Vat dyes are obtained by chlorination of iodoanthanthrone (scarlet-red), iodo-1:2:6:7-dibenzpyrene-3:8-quinone (orange-yellow), iodopyranthrone (orange), iododibenzanthrone (navy-blue), iodobenzanthronepyrazolanthrone (reddish-blue), and 7:7'-di-iodothioindigo (bluish-red); by bromination of iodoanthanthrone

(scarlet-red), iodo-1 : 2 : 6 : 7-dibenzpyrene-3 : 8-quinone (orange), iodopyranthrone (orange-red), iodobenzanthronepyrazolanthrone (blue), and 7 : 7'-di-iodothioindigo (violet); and by chlorination and bromination of iodopyranthrone (orange-red), iodoanthanthrone (reddish-orange), iododibenzanthrone (reddish-blue), and iodoisodibenzanthrone (bluish-violet). C. HOLLINS.

Manufacture of halogenated *allo*-ms-naphthodianthrones and condensation products thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,175, 27.12.28. Addn. to B.P. 303,184; B., 1929, 238).—The halogenation is performed in inorganic acids (especially chlorosulphonic acid or oleum) below 60°. Vat dyes are obtained by the bromination or chlorination of *allo*-ms-naphthodianthrone (orange to scarlet), its cyano- (orange), amino- (grey-green), methoxy- (red-brown), and diphenoxy- (blue-red) derivatives.

C. HOLLINS.

Manufacture of diazonium fluorosulphonates. I. G. FARBENIND. A.-G. (B.P. 303,527, 5.1.29. Ger., 6.1.28).—Diazo compounds are treated with fluorosulphonic acid to give moderately soluble diazonium fluorosulphonates, which are not sensitive to shock and decompose without explosion when heated. The products are used in dyeing and against insect pests. C. HOLLINS.

Manufacture of azo dyes and complex metal compounds thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,380, 1.10.28).—*o*-Aminophenols are diazotised and coupled with *N*-alkylated, -aralkylated, or -arylated 4-hydroxy-2-quinolones, at least one sulphonic group being present in one or both components. The dyes may be chromed in substance or on the fibre, or may be dyed on chromed material. Examples are: 4-chloro-*o*-aminophenol-6-sulphonic acid \rightarrow 4-hydroxy-1-phenyl-2-quinolone (chrome, bordeaux-red); 4-nitro-*o*-aminophenol-6-sulphonic acid \rightarrow 4-hydroxy-1-ethyl-2-quinolone (chrome, yellowish-red); picramic acid \rightarrow 4-hydroxy-1-methyl-2-quinolonesulphonic acid (chrome, red-brown); 1 : 2 : 4-aminonaphtholsulphonic acid \rightarrow 4-hydroxy-1-methyl-2-quinolone (chrome, violet); 4-chloro-*o*-aminophenol-6-sulphonic acid \rightarrow 4-hydroxy-1-*o*-anisyl-2-quinolone (chrome, bordeaux-red).

C. HOLLINS.

Manufacture of [direct] dis- and poly-azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,791, 17.11.28).—The coupling of *N*-acylated 1 : 8-aminonaphthol derivatives, or of 1 : 8-aminonaphthols carrying in the amino-group a heterocyclic (e.g., cyanuric) residue, with diazotised 1 : 8-aminonaphthol derivatives through one or more middle components proceeds smoothly in presence of pyridine. Where the end component carries an *N*-aminobenzoyl group etc., the external amino-group may be modified by treatment of the product with phosgene, cyanuric chloride, organic acid chlorides, etc., or by diazotisation and development on the fibre or in substance. The dye H-acid \rightarrow cresidine is coupled with: 1-anisamido-8-naphthol-6-sulphonic acid (blue); the product from H-acid, aniline, and 2 : 4-dichloroquinazalone or cyanuric chloride (greenish-blue); 1-*p*-aminobenzamido-8-naphthol-6-sulphonic acid (blue; developed on the fibre with phenylmethylpyrazolone, green); 1-*m*-acetamidobenzamido-8-naphthol-6-sulphonic

acid (blue); or the product from 1 : 8 : 6-aminonaphthol-sulphonic acid (2 mols.) and terephthaloyl chloride (bluish-green), etc. C. HOLLINS.

Sulphur dyes. E. KRAMER, L. ZEH, and B. BOLLWEG, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,324, 13.5.30. Appl., 13.6.28. Ger., 30.6.27).—See B.P. 315,910; B., 1929, 809.

Manufacture of dyes containing sulphur and chromium. F. STRAUB, H. MEYER, and H. SCHNEIDER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,758,865, 13.5.30. Appl., 2.5.28. Switz., 7.5.27).—See B.P. 290,179; B., 1929, 238.

Preparation of azo dyes. H. WAGNER, H. EICHWEDE, and E. FISCHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,383, 13.5.30. Appl., 16.3.27. Ger., 31.3.26).—See B.P. 268,807; B., 1928, 741.

Manufacture of azo dyes [ice colours and pigments]. H. HEYNA, W. KIRST, H. KRACKER, and K. MOLDAENKE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,746,652, 11.2.30. Appl., 13.3.28. Ger., 21.3.27).—See B.P. 287,479; B., 1929, 711.

Azo dyes and material dyed therewith. W. ECKERT and W. KIRST, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,758,313, 13.5.30. Appl., 3.12.28. Ger., 10.12.27).—See B.P. 302,173; B., 1930, 550.

Preventing the settling out of paste dyestuffs. W. H. CLUTTERBUCK (U.S.P. 1,758,145, 13.5.30. Appl., 2.1.29. U.K., 1.2.28).—See B.P. 310,830; B., 1929, 674.

Sodium tetraiodophenolphthalein (B.P. 304,589).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Reactivity of plain and mercerised, or other swollen, cottons. C. BIRTWELL, D. A. CLIBBENS, A. GEAKE, and B. P. RIDGE (J. Text. Inst., 1930, 21, T 85—104).—A measure of the chemical reactivity of cotton is afforded by determining its copper number (Braid) after oxidation with an alkaline hypobromite solution under conditions which are examined and standardised in this paper. The figure obtained is expressed as a ratio to that given by "plain" bleached cotton (*i.e.*, bleached cotton which has never been immersed in a liquid which causes swelling) and is called the "reactivity ratio" by analogy with the absorption (mercerisation) ratio (cf. B., 1925, 584; 1927, 293). The reactivity ratio of plain bleached cotton is therefore 1, whilst that of mercerised or other swollen cottons exceeds 1 by an amount which depends on the conditions of the swelling treatment. The method described has the following advantages over Schwalbe's "hydrolysis number" method (B., 1909, 216). The copper number obtained is almost independent of the time and temperature of treatment, all plain bleached cottons give approximately the same copper number irrespective of their geographical origin, mechanical form, and the nature of the bleaching process, and the measurement is unaffected by slight over-bleaching or acid attack. On the other hand, more care is required in the preparation of the hypobromite solution than in that of Schwalbe's 5% sulphuric acid. The reactivity ratios of

plain bleached cottons lie strictly within the range 0.95—1.12, whilst trade-mercerised materials have values of 1.3—1.6. When the reactivity ratios of cotton mercerised to different extents by treatment with sodium or potassium hydroxide solutions of different concentrations are plotted against concentration of alkali, curves are obtained which are exactly analogous to the corresponding absorption-ratio curves both at -10° and 18° , and the only divergence between the behaviours of the reactivity and absorption ratios is afforded by the fact that the reactivity ratio of mercerised cotton is not affected by drying the material at 110° . Values for cotton material treated with concentrated sulphuric acid may lie within or above the range for mercerised cotton, according to the nature of the material and the conditions of treatment, whilst the reactivity ratio is very sensitive to small changes of acid concentration within the range 64—69%. Two procedures for determination of the reactivity ratio are described, one for accurate analysis, and the other for use as a works' test to distinguish between chemically and mechanically finished materials, and the effects of various factors such as origin of the cotton, conditions of scouring and bleaching, attack by oxidising agents and acids, etc., are examined.

B. P. RIDGE.

Copper number of cotton. A. CREMONINI (Annali Chim. Appl., 1930, 20, 168—170).—A more rapid procedure is given for determining the copper number of cotton, auto-reduction of the Fehling solution and the use of the stirrer employed in Schwalbe's method being avoided. The air-dried cotton (drying at 100 — 105° sometimes increases the copper number) is heated in a boiling water-bath with diluted Fehling solution, and the cuprous oxide formed determined either by means of ferric ammonium alum and standard permanganate, or by means of standard thiosulphate and potassium iodide.

T. H. POPE.

Adsorption of water by wool. J. B. SPEAKMAN (J.S.C.I., 1930, 49, 209—213 r).—The amount of water adsorbed by seven different types of wool at 25° over a wide range of humidity has been determined, the existence of marked hysteresis between adsorption and desorption being revealed for the first time in the case of wool. The study of water adsorption by "diazotised" and formaldehyde-treated wools showed that free amino-groups play a relatively insignificant part in water adsorption by untreated wool. A comparison of the changes produced in the elastic properties and size-characteristics of wool by water adsorption suggests that the fibre is built up of long-chain protein molecules arranged preferentially about the long axis of the fibre. The polypeptide groups in these molecules are responsible for the adsorption of water which is so effective in reducing the rigidity of wool.

Determination of sulphur in wool and similar materials. C. RIMINGTON (J.S.C.I., 1930, 49, 139—140 r).—A rapid and convenient method for the determination of sulphur in materials such as wool and hair is described. The method is based on a process of wet oxidation, using copper nitrate as the oxidant. All the sulphur is converted into sulphuric acid, which is determined as barium sulphate in the usual way. The

necessity of using sealed tubes, as required in the Carius method, is dispensed with, thereby affording a considerable economy of time and labour.

Thermal conductivity of textile materials and fabrics. J. B. SPEAKMAN and N. H. CHAMBERLAIN (J. Text. Inst., 1930, 21, r 29—56).—Relations between thermal conductivity and thickness for compressed pads of loose wool and between thermal conductivity and density at constant thickness for compressed wool, cotton, and viscose rayon; the effects of various finishing processes (soap treatments etc.) on the thermal conductivity of wool fabrics; and the specific conductivities of a wide range of wool, cotton, linen, silk, rayon, and mixture fabrics have been determined. The warmth of all-wool fabrics is expressed by means of a general equation involving measurements of their thickness and density only.

B. P. RIDGE.

Swelling of sulphite-cellulose. V. I. SHARKOV (J. Appl. Chem., Russia, 1929, 2, 753—773).—No direct relationship exists between the increase in thickness and decrease in length of the cellulose fibres.

CHEMICAL ABSTRACTS.

Influence of the presence of γ -cellulose on swelling of a cellulose cardboard in alkalis. V. I. SHARKOV (J. Appl. Chem., Russia, 1929, 2, 749—752).— β - and γ -Celluloses differ in swelling properties in sodium hydroxide solutions.

CHEMICAL ABSTRACTS.

Desulphurisation of viscose [rayon] silk with ammonia solution. Y. KAMI and M. INUBUSHI (J. Cellulose Inst., Tokyo, 1930, 6, 99—106; cf. B., 1930, 96).—Raw viscose rayon was treated in a closed vessel with a solution of ammonia under controlled conditions, washed free from ammonia, and dried below 40° . Tearing strength, extensibility, and sulphur content were determined on the treated material. It is found that in this way the sulphur content may be reduced to a lower value (below 0.01%) than when sodium sulphide is used, but it is impossible to remove the whole of the sulphur. Conditions of treatment recommended are: for 1 hr. with a solution of 150 g. of ammonia per litre at 70° , or, for a quicker process, 30 min. with a solution of 250 g. per litre at 50° . Tearing strength and extensibility are increased, and the increase is roughly proportional to the time of treatment and inversely proportional to the temperature. The material is generally whiter, softer, and more lustrous, but a disadvantage is that the process must be carried out in airtight apparatus.

B. P. RIDGE.

Method of analysis of cellulose formate [or other formic esters] by oxidation. G. TOCCO and A. NYSSENS (Giorn. Chim. Ind. Appl., 1930, 12, 121—126).—This method is applicable only to cellulose formates, since with the acetate the proportions of oxygen required for the complete oxidation of the cellulose and acetic acid are too nearly equal to allow of accurate results. The ester (1 g.), rendered absolutely dry by heating at 100 — 105° for 2 hrs. or at a lower temperature in a vacuum, is treated with 135—150 c.c. of *N*-potassium dichromate and, slowly and with cooling, with 40 c.c. of sulphuric acid. If necessary, the evolution of carbon dioxide is restrained by cooling. After the lapse of

30 min. the beaker is left for 8 hrs. in a boiling water-bath, the excess of dichromate (which should be at least 10 c.c.) being determined by titration either with ferrous sulphate or iodometrically. The number of c.c. of *N*-dichromate required for 1 g. is 148.14, 136.78, 128.43, or 121.95 for cellulose and the mono-, di-, and tri-formates, respectively. This procedure, which may be used also for other formic esters, such as those of starch, has been employed to control methods of hydrolysis by means of alkali. Of the finely-powdered product, 1 g. is dissolved in 40% potassium thiocyanate solution and treated for 3 hrs. with excess (40 c.c.) of cold *N*-sodium hydroxide, the excess of which is then titrated (1 g. of the mono-, di-, or tri-formate requires 5.26, 9.18, or 12.19 c.c. of the alkali, respectively). If the ester is not finely powdered, the hydrolysis requires heat. For formic esters which are not readily attacked by normal sodium hydroxide, or are only slightly soluble in thiocyanates, or are of very abnormal composition, the oxidation method is recommended.

T. H. POPE.

Beating of [paper] pulp. VIII. Effect of various reagents on the stuff in beating. IX. Effect of beating on the viscosity of stuffs. M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 89—99, 106—107; cf. B., 1930, 552).—VIII. The effects of various reagents and of various pretreatments on the beating of stuff in the ball-mill have been investigated. Pretreatment with dilute solutions of strong acids causes wet-beating to be attained rapidly, owing to the reduction in the degree of aggregation of the cellulose, but increasing loss of minute fibres, or slime from the wire-cloth results and the strength of the paper sheets is greatly diminished. When such dilute solutions are used as beating media, the effect is the same as when water alone is used. Weakly xanthated pulps give very wet pulps which become very free when decomposed with acid. Xanthated and subsequently decomposed pulps can only be wet-beaten with difficulty. Addition of alcohol or glycerin to the water retards beating. Pulps pretreated with concentrated zinc chloride solutions give very free stuffs and very absorbent paper sheets. Those beaten in alkaline solutions also give free stuffs, but pulps pretreated with concentrated alkaline solutions can only be wet-beaten with difficulty. Kraft pulps become soft on bleaching, and are easily wet-beaten, but the effects of bleaching sulphite-pulps depend on the degree of bleaching. Addition of lactic acid to the water, or preliminary steeping of pulps in dilute lactic acid solutions does not accelerate beating, which is contrary to Schwalbe's claim (G.P. 379,996; B., 1923, 1172 A). Pulps for artificial silks are similar in behaviour to ordinary paper pulps, whilst those for nitrocellulose are difficult to beat to wetness.

IX. Surgical cotton was beaten for 7 hrs. until a very wet stuff was obtained and the viscosity of the solution obtained by dissolving the dried product in 0.5% cuprammonium solution containing 182 g. NH_3 and 11 g. Cu per litre was compared with that of the original cotton under the same conditions at 25°. The average relative viscosities were found to be: cotton 10.8, beaten stuff 6.7; this considerable fall is attributed to

a reduction in the size of the micelles in the cellulose fibres.

B. P. RIDGE.

Digestion of pine wood [*Pinus sylvestris*] by the sulphite process. K. BERNDT (Papier-Fabr., 1930, 28, 313—319).—A summary of suggested methods for the pretreatment of pine wood to enable use to be made of the sulphite process. The heart-wood in its natural state does not respond to this process; the sap-wood may be pulped by this process and the residual heart-wood dealt with separately.

T. T. PORTS.

Irregular distribution of acid in a sulphite [pulp] digester. A. ECKE (Papier-Fabr., 1930, 28, 297—303).—The concentration of acid in a sulphite digester is greater in the lower part than in the upper. It is suggested that under the conditions of digestion the acid behaves as a solution of sulphur dioxide in water, and as the hydrostatic pressure is greater in the lower part of the digester the solubility of the gas is higher. Alternatively, the phenomenon may be explained as due to preferential adsorption by the wood in the upper part of the digester during the addition of the liquor.

T. T. PORTS.

Soda recovery in the sulphate-pulp process. W. GÜNTHER and T. KLEINCKE (Papier-Fabr., 1930, 28, 281—288).—An analysis of the cost of counter-current washing of sulphate-pulp, followed by recovery of soda by means of multiple-effect evaporation of the washings, incineration in a rotary oven, and causticising, is given.

T. T. PORTS.

Modern pulp-sizing problems. B. WIEGER (Papier-Fabr., 1930, 8, 113—116).—The alkaline and acid processes of sizing are compared. The author's "Bewoid" process utilises a 100% free rosin size produced by mechanical means. 100 kg. of rosin are dispersed in 10—15 min. to a particle size of 0.5—1.0 μ , with a power consumption of 4 h.p. It is found that particle size is the controlling factor in free rosin sizing. With particles below 1 μ sizing is always good, above 1 μ it diminishes, and above 10 μ it is negligible. The particles of a 40% free rosin size were found to lie between 0.2 and 1 μ . An optical method is used to determine the particle size. The size of particle does not increase with the free rosin content. The coagulation of rosin size is a time reaction dependent on the free rosin content: the higher the rosin content the slower is the reaction. With the mechanical dispersions several days are required for flocculation. The size of particle is not increased, there being merely a coalescence. The large flocs caused by rapid coagulation can act as sizing agents only by virtue of filtration, i.e., they act as loadings. The size should be thoroughly distributed throughout the stuff before coagulation in order that the formation of large flocs be avoided, the largest surface of fibres be covered, and the optimum particle size be not exceeded.

T. T. PORTS.

Determination of cellulose in wood and pulp. K. KÜRSCHNER and A. HOFFER (Tech. u. Chem. Papier Zellstoff-Fabr., 1929, 26, 125—139; Chem. Zentr., 1930, i, 146).—The dry wood (1 kg.), in small shavings, is heated for 1 hr. on the water-bath under reflux with 25 c.c. of 20 vol.-% alcoholic nitric acid; after

filtration through a glass filter-crucible the residue is again similarly treated once or twice. Finally the pure white cellulose is washed with water and dried at a temperature gradually rising to 108°. Only 75% of the hemicellulose of wood is removed, but the quantity remaining in the crude cellulose of similar kinds of wood is constant. The method was also applied to bleached and unbleached pulps. A. A. ELDRIDGE.

Testing of paper half-stuffs for strength. W. HUMM (Papier-Fabr., 1930, 8, 116—119).—The method given is claimed to render tests on half-stuffs independent of the paper-making factors, such as beating, "shake," and pressing, attending the preparation of test sheets, and to give figures which represent the strength of the individual fibres. The tests are made with an attachment fitted between the jaws of the Schopper tensile tester, and consisting essentially of two steel plates with a central aperture and a steel punch (4.75 mm. diam.) giving a circumference of 15 mm. (the width of the standard strip for the tensile test). Test sheets of the substance (200 g./m.²) are prepared and clamped between the steel plates. The punch is brought slowly to bear on the exposed surface until the test-piece is punched through. The pressure required is read off in the usual way. The force is considered as acting only at the circumference, as distinct from the bursting test. The specific resistance of the fibres to punching is obtained by multiplying the "punching length" by the sp. gr. of the fibres. Beating in the Lampén mill increases the punching strength, independently of the length of the fibres, as does also the removal of incrustants by bleaching. The strength of the original untreated fibres may be obtained by extrapolation. It is suggested that the method may be applied to pulp-boards. T. T. POTTS.

Determination of moisture in paper. E. H. RIESENFELD and T. HAMBURGER (Papier-Fabr., 1930, 28, 288—289).—Drying paper in an oven at 105° does not remove water of adsorption or "hydration" (mechanical). Extraction for 1 hr. with carbon tetrachloride removes a maximum of water, which is determined volumetrically in the extract. An attempt to differentiate between mechanically-bound and chemically-bound water in parchment papers failed, as papers of different degrees of parchmentisation gave almost identical results. It is assumed that parchments do not contain chemical water of hydration, or, alternatively, that it is not removable by this method. On a wide range of papers, moisture contents differing by 0.5—1.5% are obtained when determined by oven-drying and extraction methods. T. T. POTTS.

Absorption, transmission, and reflection of radiant heat by fabrics. Transfer of moisture through fabrics. J. GREGORY (J. Text. Inst., 1930, 21, T 57—65, T 66—84).

Cellulose-decomposing organisms. SKINNER.—See XVI.

PATENTS.

Viscose spinning-bath solutions. C. F. M. VERSTYNNEN (B.P. 328,492, 18.6.29).—The concentration of the spinning bath is maintained by addition of the

salts (magnesium or zinc sulphate) in the substantially anhydrous state. F. R. ENNOS.

Manufacture of stable viscose solutions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,044, 24.12.28).—Phenols or naphthols are added during the production of the viscose. F. R. ENNOS.

Manufacture of artificial threads. ACETA GES. M.B.H. (B.P. 311,763, 24.4.29. Ger., 16.5.28).—Artificial threads of uniform individual titers are produced by the dry-spinning process by causing the spinning solution to flow in symmetrically-placed streams, and in as thin a layer as possible, through the narrow space between two metallic bodies of high heat capacity which are placed directly above the spinning nozzle. F. R. ENNOS.

Manufacture of artificial threads. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,671 and 327,737, 4.10.28).—(A) Viscose is spun through a nozzle either of metal or vitreous material having a straight, cylindrical bore (diam. less than 0.2 mm., length at least five times internal diam.) into the usual precipitating bath; the spun threads are directly stretched to at least five times (metal nozzle) or ten times (vitreous nozzle) their original length. (B) A solution of an organic derivative of cellulose is spun through a glass nozzle (dimensions as in A) into air heated to 50—55°, and the spun threads are stretched to at least five times their original length. F. R. ENNOS.

Treatment of freshly spun threads of artificial silk. SONDERMANN & Co. (B.P. 307,357, 5.3.29. Ger., 5.3.28).—Threads coming from the spinning bath are reeled without doubling in close set, quick-traverse turns free from gaps, and preferably in a thick layer; the resulting compact cheeses are removed without skeining and treated in perforated containers, centrifuges, etc. with the appropriate liquids. F. R. ENNOS.

Production of artificial filaments or threads by the dry-spinning method. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 327,740, 6.10.28).—A lustre-modifying liquid (xylene, water, etc.) is applied by means of a wick at an early stage in the drying of the filaments of organic cellulose derivatives while they contain a relatively high proportion of solvent; the effect produced may be in the direction of reduced or of enhanced lustre, depending on the liquid used and on the stage of drying reached by the filaments. F. R. ENNOS.

Manufacture of [artificial] threads, foils, etc. [from polymerised diolefines]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,164, 17.11. and 31.12.28).—A filtered solution of synthetic rubber (from butadiene) in cyclohexane, methylene dichloride, or other suitable solvent is spread on glass plates (for foil) or pressed through a nozzle (for threads), and after evaporation of the solvent the product is treated with sulphur chloride vapour until it has lost its extensibility. Thin transparent foils or threads suitable for photographic purposes or for packing of comestibles are obtained. [Stat. ref.] C. HOLLINS.

Manufacture of cellulose acetate. U.S. INDUSTRIAL ALCOHOL Co. (B.P. 306,531, 22.1.29. U.S., 24.2.28).—

Cellulose acetate, produced by acetylation of cellulosic material under pressure, with or without a catalyst, in a medium of liquid sulphur dioxide, is discharged from the reaction vessel so that the sulphur dioxide is abruptly liberated as gas, thus disrupting the ester with production of a light and fluffy product. F. R. ENNOS.

Manufacture of benzyl cellulose. IMPERIAL CHEM. INDUSTRIES, LTD., and D. TRAILL (B.P. 327,714, 3.10.28).—After mercerising cellulose with 18–20% caustic soda and pressing to $2\frac{1}{2}$ –4 times the weight of cellulose used, the product is matured at 22–35°, mixed with 50–100 pts. of solid caustic soda for each 100 pts. by wt. of raw cellulose, and benzylated; by maturing for different times and at different temperatures within the prescribed limits, products differing in viscosity and solubility are obtained. F. R. ENNOS.

Manufacture of cellulose ethers. H. DREYFUS (B.P. 327,157, 23.12.28).—Cellulose hydroxyalkyl ethers (cf. B.P. 277,721; B., 1927, 872) are etherified, e.g., with alkali and ethyl sulphate, to reduce the capacity for dissolving or swelling in water. C. HOLLINS.

Preparation of cellulose esters and ethers. BRIT. CELANESE, LTD. (B.P. 303,348, 12.3.29. U.S., 22.3.28).—Cotton, wood pulp, etc. is treated with hydrofluoric acid to remove silicious matter, with an aliphatic acid (formic or acetic) to remove oils, wax, etc., and is afterwards washed and converted into the required cellulose derivative. F. R. ENNOS.

Production of distended fibrous material. F. L. BRYANT, Assr. to S. L. SCHWARZ (U.S.P. 1,740,280, 17.12.29. Appl., 19.12.28).—A mixture of fibrous material, e.g., cellulose pulp, asbestos, wool, with a foam-forming substance, e.g., saponin, is thoroughly agitated and afterwards dried with the bubbles incorporated therein. F. R. ENNOS.

Production of cellulosic material. B. DORNER, Assr. to CORNSTALK PRODUCTS CO. (U.S.P. 1,758,655, 13.5.30. Appl., 17.1.27).—See B.P. 283,851; B., 1929, 593.

Apparatus for the gassing of textile yarns or threads. J. STUBBS, LTD., and J. H. STUBBS (B.P. 327,913, 20.6.29).

Process and apparatus for treating artificial threads. I. G. FARBENIND. A.-G. (B.P. 311,399, 17.4.29. Ger., 11.5.28).

Making bands of artificial fibres. I. G. FARBENIND. A.-G. (B.P. 304,667, 23.1.29. Ger., 23.1.28. Addn. to B.P. 286,603).

[Machine for] drying of cellulosic films. A. J. MAURER (B.P. 328,566, 28.1.29).

Wood substitute (G.P. 461,775).—See IX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Substantivity [of dyes]. E. JUSTIN-MUELLER (Bull. Soc. Ind. Mulhouse, 1930, 96, 215–216).—It is more difficult to obtain clear white discharges (using sodium hyposulphite) on sulphur than on indigo dyes; this is attributed to the greater substantivity of sulphur dyes on cotton. A. J. HALL.

Adsorption of sodium hydroxide by cellulose and mercerisation. M. M. CHILIKIN (J. Appl. Chem., Russia, 1929, 2, 739–747).—The phenomena of mercerisation (swelling accompanied by a change in colloidal state) and adsorption of sodium hydroxide by cellulose are explained by considering the cellulose to be a two-phase system, consisting of a crystalline network in an amorphous mass. Adsorption from dilute sodium hydroxide solutions follows the laws of adsorption, which hold also for adsorption by the mercerised cellulose until (at high concentrations) a ratio of approximately $C_6H_{10}O_5 : NaOH$ is reached. Mercerised cellulose adsorbs more sodium hydroxide from aqueous-alcoholic solutions than does ordinary cellulose. Experiments with mercerised cellulose show that a certain concentration of sodium hydroxide within the cellulose can be in equilibrium with various concentrations of sodium hydroxide in water or aqueous ethyl alcohol.

CHEMICAL ABSTRACTS.

Reactivity of mercerised cottons. BIRTWELL and others.—See V. **Dyeing of leather.** WOODROFFE and HILL.—See XV.

PATENTS.

Dyeing and printing of cellulose acetate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,394, 1.10.28).—Acetate silk is dyed by means of azo dyes formed by coupling a diazo compound of the benzene series free from sulphonie or carboxylic groups with an *N*-alkylated 4-hydroxy-2-quinolone. Examples are: *m*-nitroaniline \rightarrow 4-hydroxy-*N*-methyl-2-quinolone (greenish-yellow); *p*-aminodimethylaniline \rightarrow 4-hydroxy-*N*-methyl-2-quinolone (red-violet); *p*-chloroaniline \rightarrow 6-chloro-4-hydroxy-*N*-ethyl-2-quinolone (yellow). C. HOLLINS.

Wet treatment of yarn or the like. SONDERMANN & Co. (B.P. 314,402, 26.6.29. Ger., 26.6.28).—The threads on spools are disposed at an angle to the directions of flow of the treatment fluid.

Rendering textiles impermeable and rot-proof. M. and R. WALRAVE (F.P. 638,377, 3.12.26).—The fabric is soaked in a mixture of a mineral or vegetable oil and a metal soap, e.g., a mixture of 25% of rape oil, 35% of ozokerite, and 40% of zinc oleate. A. R. POWELL.

Manufacture of [coated fabric for use as] gas containers. GOODYEAR-ZEPPELIN CORP., Assees. of K. HUERTLE (B.P. 304,775, 22.1.29. U.S., 27.1.28).—The fabric is coated with a Bakelite varnish mixed with a softener (tritolyl phosphate, castor oil) and thinner (acetone), and is dried at 90–100°. F. R. ENNOS.

Dyeing apparatus. J. P. DE G. ANGLADA (B.P. 310,006, 17.4.29. Spain, 19.4.28).

[Machine for] ornamentation of fabrics, paper, etc. W. LOWE, and FLEURET FABRICS, LTD. (B.P. 328,730, 7.3.29).

Calendering of textile fabrics. C. H. WEISBACH KOMM.-GES. (B.P. 329,259, 19.10.29. Ger., 20.10.28).

Dyeing with leuco-vat dyes (B.P. 327,672).—See IV. **Coating compositions** (B.P. 297,681).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of nitrous acid in spent nitrating acids. F. HOLDEN (J.S.C.I., 1930, 49, 220—221 r).—Spent nitrating acids containing substances which interfere with the usual titration methods are treated with urea solution in a nitrometer. The volume of nitrogen and nitric oxide obtained is measured over sodium hydroxide solution in a second nitrometer. The nitric oxide is measured by absorption with potassium permanganate or sodium sulphite. A less accurate method requiring only one nitrometer is also described.

Apparatus for the continuous generation of very dilute mixtures of nitrogen peroxide and air of controlled humidity and temperature. II. J. POOLE and W. J. POWELL (J.S.C.I., 1930, 49, 29—34 r).—An apparatus designed primarily for testing the sensitivity and uniformity of indicator papers used for the detection of nitrogen peroxide, but also suitable for use with other gas mixtures and the general study of gas reactions at high dilutions, is described. The principle involved is that of mixing two uniformly moving streams, one a slow stream of known velocity containing nitrogen peroxide and the other a fast one of air, also of known velocity. Successive dilutions are obtained by addition of further measured air streams to the original stream. A number of accessories devised for the accurate control and measurement of the velocity of gas streams are described.

Production of sodium carbonate from sodium sulphate, with ammonia as by-product. H. MURA and R. HARA (Tech. Rep. Tôhoku, 1930, 9, 57—68).—The interaction of nitrogen in presence of an iron catalyst on the reactants of the Leblanc soda process, as represented by the equation: $\text{Na}_2\text{SO}_4 + \text{CaCO}_3 + 6\text{C} + \text{N}_2 = 2\text{NaCN} + \text{CaS} + 2\text{CO}_2 + 3\text{CO}$, was investigated (cf. B., 1925, 497). The sodium cyanide produced (yield about 70%) is somewhat difficult to separate in a pure state from the sulphur compounds present. It may, however, be practically quantitatively converted into ammonia by means of superheated steam; a solid residue is then left having the same composition as "black ash," except that it also contains iron as metal or oxide. On lixiviation of this residue and evaporation of the extract in presence of carbon dioxide, sodium carbonate of 99.96% purity, corresponding to 90—98% of the sodium sulphate originally used, is obtained.

S. K. TWEEDY.

Ammonium phosphates. E. V. BRITZKE, A. P. DUNAIEV, and E. P. POKHVALINSKAJA (Trans. Sci. Inst. Fertilisers, Moscow, 1928, No. 51, 5—79).—The dissociation pressure in the reaction $(\text{NH}_4)_2\text{HPO}_4 = \text{NH}_3 + \text{NH}_4\text{H}_2\text{PO}_4$ at 98—147° satisfied the Clausius-Clapryron equation $d \log p / dT = U / RT^2$, for $t = 117.5^\circ$, $U = -19.26$ g.-cal. per mol. Diammonium hydrogen phosphate can be obtained below 130° by using excess of ammonia. Since the concentration of phosphoric acid has no influence on the process, the low concentrations of this substance from the volatilisation process can be fully utilised. Solubility curves are reproduced.

CHEMICAL ABSTRACTS.

Production of ammonium phosphate. S. VOLFOVICH (Fertilisers and Yields, Russia, 1929, 30—37).—Britzke's method requires a 50% concentration of P_2O_5 . Phosphoric acid concentrations of 6—18% P_2O_5 can be used when the reactions are allowed to take place in two stages with intermediate formation of ammonium dihydrogen phosphate. The sesquioxides can be completely precipitated in the first stage. The triammonium phosphate is unstable, and may be treated with phosphoric or sulphuric acid or mixed with the product of the first stage. CHEMICAL ABSTRACTS.

Thermal method of obtaining potassium phosphates. E. V. BRITZKE, N. E. PESTOV, and E. P. POKHVALINSKAJA (Fertilisers and Yields, Russia, 1929, 69—71).—Potassium metaphosphate was obtained by interaction of potassium chloride and phosphoric acid at 250—500°.

CHEMICAL ABSTRACTS.

Formation and volumetric determination of potassium thiosulphate in the mother-liquors of potassium metabisulphite. F. DE BACCO (Giorn. Chim. Ind. Appl., 1930, 12, 121—122).—The formation of thiosulphate together with metabisulphite when potassium hydroxide or carbonate solution is treated with the mixed gases from sulphur burners is doubtless due to the action of entrained sulphur vapour or powder, but its appearance in small proportion when pure sulphur dioxide is used is not readily explainable. To determine the thiosulphate, 10 c.c. of the mother-liquor are made up to 500 c.c. with cold, recently boiled water, and 100 c.c. of this solution are shaken with 10 c.c. of formalin previously neutralised to phenolphthalein by means of *N*-sodium hydroxide, and left for about 10 min. The solution is then made slightly acid to methyl-orange with *N*-sulphuric acid and, after the lapse of 20 min. to allow of the formation of the bisulphite-formaldehyde compound, the thiosulphate is titrated with iodine solution in presence of starch paste. In another aliquot part of the solution the bisulphite, sulphite, and free sulphur dioxide (rarely present) are determined.

T. H. POPE.

Radioactive ash from crude oils. BOGOYAVLENSKI.—See II. **Russian phosphate deposits.** FIVEC and ROZANOV.—See XVI. **Detection of gold chloride.** SETTIMI.—See XXI. **Absorption of mercury chloride by charcoal.** RAKUSIN.—See XXIII.

PATENTS.

Manufacture of potassium nitrate. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,272, 12.12.28 and 8.4.29).—In the treatment of calcium nitrate with potassium chloride, a quantitative yield of potassium nitrate may be obtained if the separation of the salt is carried out in presence of ammonia. This may be introduced by using ammoniacal solutions of the original salts, or by adding it after mixing, or after separation of the potassium nitrate. A further improvement consists in cooling the mother-liquor to as low as -30° , and then treating it with gaseous ammonia, or with the gas mixtures obtained in the synthesis of ammonia, to separate calcium chloride.

W. J. WRIGHT.

Production of alkali nitrates. F. JOST (B.P. 306,046, 14.2.29. Ger., 14.2.28).—Alkali chlorides are

converted by means of phosphoric acid into alkali phosphates, which latter are then converted by reaction with calcium nitrate into alkali nitrate and calcium phosphate. The process may be carried out in a cyclic manner. Thus the calcium phosphate may be treated with sulphuric acid and converted into calcium sulphate and phosphoric acid, the latter being then used in the process and the former being converted with ammonia and carbon dioxide into calcium carbonate and ammonium sulphate. The calcium carbonate is then converted into calcium nitrate, which is used in the process, the carbon dioxide being recovered for further treating calcium sulphate. Alternatively, the calcium phosphate may be treated with nitric acid directly, the phosphoric acid being treated in the solution containing calcium nitrate with alkali chlorides. S. K. TWEEDY.

Removal of alkali chlorides from crude potassium carbonate solutions. I. G. FARBERIND. A.-G. (B.P. 327,938, 4.9.29. Ger., 16.10.28).—Ammonia gas is passed through the crude potassium carbonate solution, preferably to saturation point. The liquid is allowed to settle and the upper ammoniacal layer which forms, containing the bulk of the alkali chloride impurities, is removed. S. K. TWEEDY.

Manufacture of alkaline-earth chlorides from the corresponding sulphates. I. G. FARBERIND. A.-G., Assces. of F. LINDNER (G.P. 460,572, 16.12.24).—Mineral sulphates, *e.g.*, barytes, are heated at 800° in a current of water-gas or producer gas to which chlorine has been added. An extraction of 95% of the barium as chloride is claimed. A. R. POWELL.

Manufacture of alkali fluorides. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 328,211, 18.1. and 24.9.29).—By causing excess of gaseous hydrofluoric acid to act on solid alkali chloride at temperatures below 50°, liquid alkali fluoride is formed with some loosely combined hydrofluoric acid. By heating this compound, alkali difluoride and fluoride are obtained. If the alkali chloride is heated with the hydrofluoric acid at 80–120° the neutral fluoride is produced, or the latter may be obtained by heating sodium difluoride at temperatures above 100°. W. J. WRIGHT.

Manufacture of carbamates and conversion products of the same. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,005, 14.1.29).—Salts of metals other than potassium, *e.g.*, sodium chloride, calcium chloride, lead nitrate, are treated in solution in liquid ammonia with carbon dioxide, generally at the usual temperature; water or ammonium nitrate or acetate may be added to increase the solubility of the salts in the ammonia. The carbamates formed, after separation from the ammonia by filtration, may be converted by heat into other compounds, *e.g.*, calcium carbamate into the cyanamide. L. A. COLES.

Dehydration of (A) solid substances, (B) salts. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 327,481–2, 17.1.29).—Liquid ammonia is used for dehydrating, *e.g.*, (A) silica hydrogels, (B) crystalline magnesium chloride; the process is effected, *e.g.*, on the Soxhlet principle, and residual ammonia, whether retained mechanically or in chemical combination, as

in the case of magnesium chloride, is removed by heat. L. A. COLES.

Manufacture of rinsing, grease-removing, and cleaning compositions. HENKEL & Co., G.M.B.H. (B.P. 328,097, 23.3.29. Ger., 27.2.29).—Aqueous solutions containing alkali phosphates with an alkaline reaction and soluble silicates, together, if desired, with alkali hydroxides, are dried in atomising apparatus with the simultaneous addition of regulated quantities of powdered, calcined sodium carbonate in the atomising zone. L. A. COLES.

Manufacture of sodium glutamate. Y. S. FONG (B.P. 327,810, 14.2.29).—Glutamic acid hydrochloride, from hydrolysis of gluten with concentrated hydrochloric acid and a little nitric acid, is dissolved in hot water, sodium carbonate equivalent to the hydrochloric acid is added, and the glutamic acid which separates on cooling is washed with ice-water to remove salt and finally neutralised with sodium carbonate to give monosodium glutamate of purity suitable for flavouring matters. C. HOLLINS.

Recovering zinc sulphide from zinc-containing liquids. F. KAUBA (B.P. 327,596, 24.4.29).—Solutions containing zinc compounds obtained, *e.g.*, in metallurgical leaching processes are treated with a mixture of non-reacting gases containing hydrogen sulphide to remove copper, lead, cadmium, etc., and a little zinc as their sulphides and to saturate the solution with the non-reacting gases; the residual solution is treated with a gas rich in hydrogen sulphide so that the remaining zinc is precipitated completely as its sulphide. The first gas mixture is prepared by passing mixed gases containing hydrogen (*e.g.*, coke-oven gas, water-gas) over red-hot iron pyrites, and the second by treating with acids the ferrous sulphide obtained in preparing the first mixture. L. A. COLES.

Preparation of pure zirconium sulphate from zirconium ores decomposed by sulphuric acid. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (G.P. 434,987, 26.9.23).—The concentrated, slightly acid solution of zirconium sulphate obtained by digesting the ore with sulphuric acid is treated with sulphuric or hydrochloric acid to precipitate zirconium sulphate. This is redissolved in dilute acid, the solution neutralised and boiled to precipitate a basic sulphate, and this salt redissolved and re-precipitated by either of the above methods to eliminate the last traces of iron and titanium. A. R. POWELL.

Treatment of decolorising clays. W. A. RAINE and R. C. POLLOCK, Assrs. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,739,734, 17.12.29. Appl., 21.12.22).—The clay, *e.g.*, halloysite, is made into a slurry with water and heated until a thin colloidal paste is obtained. Strong sulphuric acid is added in a thin stream to the hot paste, and heating and stirring are continued for 2 hrs., soluble material is then removed by washing and decantation, and the activated residue is dried first at 120° then at 80–85°. A. R. POWELL.

Production of metal carbonyls. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 327,956, 14.12.28).—Metal compounds capable of reduction are converted

into the metal carbonyls by treatment with carbon monoxide at elevated temperatures and pressures, *e.g.*, nickel oxide, nickel sulphide, and iron oxide are treated with the gas at 250° under 350 atm., at 200° under 200 atm., or at 225° under 200 atm., respectively.

L. A. COLES.

Preparation of [colloidal] silica gel. A. P. OKATOFF (B.P. 328,241, 23.10.28).—Sodium silicate is mixed with hydrochloric acid, and when syneresis begins the gel is treated with solutions of alkali salts or ammonium salts of polybasic acids in presence of ammonia, after which the gel is washed, and activated by drying at a high temperature.

W. J. WRIGHT.

Production of hydrogen [from methane]. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,048, 25.1.29).—Methane or a gas mixture containing it is blown together, if desired, with steam into molten iron; the carbon liberated by the decomposition of the methane dissolves in the iron and is subsequently removed by blowing with air. Alternatively, the methane and air are blown continuously into the opposite ends of a reaction chamber provided with a partition dipping below the surface of the iron to prevent admixture of the gaseous products in the two sections; secondary air may be admitted above the iron in the oxidation section to oxidise the carbon monoxide formed therein.

L. A. COLES.

Manufacture of hydrogen peroxide. I. G. FARBENIND. A.-G., Assecs. of W. FRANKENBURGER and C. STEIGERWALD (G.P. 461,635, 7.1.27).—A mixture of gases containing hydrogen and preferably less than 5% of oxygen together with the vapour of a metal is subjected to the radiation from a metal-vapour lamp.

A. R. POWELL.

Producing ammonium phosphate or mixed fertilisers containing it by leaching of raw phosphate. F. G. LILJENROTH (U.S.P. 1,758,448, 13.5.30. Appl., 21.6.28. Swed., 12.7.27).—See B.P. 290,518; B., 1928, 539.

Production of phosphorus and alumina cement. R. SUCHY, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,758,241, 13.5.30. Appl., 24.3.28. Ger., 19.3.27).—See B.P. 287,036; B., 1928, 366.

Refrigerant (F.P. 637,898). Catalysts (B.P. 301,736).—See I. Acetic acid from wood etc. (B.P. 327,415).—See II. Anhydrous acetic acid (B.P. 317,462 and 327,444).—See III. Tin from tin salt solutions (B.P. 313,166).—See X. Carbon monoxide (U.S.P. 1,740,139).—See XVIII.

VIII.—GLASS; CERAMICS.

Glasses transparent to ultra-violet radiation. A. R. WOOD and M. N. LEATHWOOD (Nature, 1930, 125, 351).—Polemical against English (B., 1930, 239).

L. S. THEOBALD.

Raw and white ground-coat [for enamels]. A. MALINOVSKY (J. Amer. Ceram. Soc., 1930, 13, 277—284).—The use of lepidolite (*cf.* B.P. 307,259; B., 1929, 356) as a ground-coat for enamel is shown to be impracticable for cast iron, though it might be suitable for steel. Details are given of the composition of a

number of ground-coat mixtures. Cobalt oxide is not essential to give good adhesion of the ground-coat. Silica, boric oxide, and sodium oxide, in the proportions of 9—14SiO₂, 2B₂O₃, and Na₂O, are indispensable components. The comparative adhesion of fritted and raw ground-coats was studied and the results are illustrated.

F. SALT.

Opacifying effect of some fluorides in enamel mill additions. R. M. PEARCE and R. M. KING (J. Amer. Ceram. Soc., 1930, 13, 272—276).—A study was made of the effect of barium fluoride and zinc fluoride, when added in the mill after fritting, on four types of enamels—high-silica, high-borax, high-lead, and an antimony oxide enamel. The results, which are presented in triaxial diagrams, were entirely negative. In the high-silica enamel the fluoride decreased the opacity.

F. SALT.

Simplified practice of mixing coloured glazes. V. J. BOEHM (J. Amer. Ceram. Soc., 1930, 13, 285—286).—To facilitate the mixing of a number of coloured glazes, using one base glaze to which the different wet stains are added, curves are plotted relating the percentage of solids and ounces per pint in both the base glaze and the stains.

F. SALT.

Refractories and refractory cements for the non-ferrous foundry. H. E. WHITE (J. Amer. Ceram. Soc., 1930, 13, 219—236).—A review is presented of the recent developments in linings for open-flame, crucible, and electric furnaces, and in refractory cements. The use of fireclay refractories in this industry is being rapidly superseded by a series of new products and super-refractory cements. For crucible furnaces, preference is being shown for silicon carbide refractories and cements, which materials are finding increased favour for open-flame furnaces also, although in this latter class alumina and alumina-silica combinations are competing strongly. High-alumina and mullite-containing refractories are favoured for electric furnaces. The life of fireclay refractories is being considerably prolonged by the use of suitable refractory cements.

F. SALT.

Forming pressure of dry-pressed refractories. I. Effect of pressure variations on the properties of green and dry bodies. R. E. BIRCH (J. Amer. Ceram. Soc., 1930, 13, 242—255).—Tests have been made on 8 commercial dry-press mixtures, test bricks being formed in a hydraulic press giving a maximum pressure of 6000 lb./in.² on the flat. The bulk density of the dry and green samples increased with increasing forming pressure, the effect being most marked at the lower pressures. The same applied also to the transverse strength. Pressures applied in industrial practice are low, and could be increased by the use of materials of lower moisture content; a slight increase would greatly improve the green strength of the ware. Dry-press bodies approach a condition of maximum compactness, beyond which additional pressure produces no further increase in density.

F. SALT.

PATENTS.

Tunnel kiln. A. McD. DUCKHAM, Assr. to WOODALL-DUCKHAM (1920), LTD. (U.S.P. 1,758,785, 13.5.30. Appl., 8.4.27. U.K., 3.12.26).—See B.P. 280,044; B., 1928, 92.

Refractory [coatings for silicon carbide] articles and their protection. R. H. MARTIN, ASSR. to NORTON Co. (Re-issue 17,661, 13.5.30, of U.S.P. 1,653,918, 27.12.27).—See B., 1928, 158.

Preparation [drying etc.] of ceramic articles. W. and M. LENGERSDORFF (B.P. 328,777, 22.4.29).

Artificial masses (B.P. 327,722).—See XIII.

IX.—BUILDING MATERIALS.

Magnesium cement. N. S. KURNAKOV, S. F. SHEMTSCHUSHNI and V. A. AGEIEVA (J. Appl. Chem., Russia, 1929, 2, 651—661).—Cement containing magnesium chloride has three setting periods; the three breaks in the heating curve are due, respectively, to the boiling of the magnesium chloride solution, the dissociation of magnesium hydroxide, and the formation of a solid, possibly basic magnesium chloride. The region of maximum hardness is within the limits of the third break.

CHEMICAL ABSTRACTS.

Heat insulation. WINTERBOTTOM.—See I. Refractory cements. WHITE.—See VIII.

PATENTS.

Manufacture of concrete. J. S. MORGAN (B.P. 328,030, 17.11.28).—Adherent air is prevented or removed from concrete mixtures by agitating the cement with water in a high-speed disintegrator and then mixing the slurry with a wetted aggregate. The process may be conducted under reduced pressure.

C. A. KING.

Manufacture of roads, paths, etc. S. J. L. ROBINSON and W. T. COLLIS (B.P. 328,166, 5.7.29).—A suitable grade of aggregate (75—95%) is mixed with 5—20% of powdered pitch and sprayed with 0.5—5% of creosote. The process of mixing is continued until the creosote has softened the pitch and caused it to adhere to the aggregate.

C. A. KING.

Treatment of roads, paved surfaces, etc. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,968, 12.1. and 16.5.29).—The roads etc. are cleansed by treatment with solutions containing sulphonated organic compounds (e.g., aromatic or hydroaromatic hydrocarbons or their derivatives) or with other substances which induce the solubility of oils and rubber in water.

C. A. KING.

Manufacture of a wood substitute. H. BRANDT (G.P. 461,775, 26.3.25).—The product obtained by digesting paper with potassium hydroxide solution is treated with sodium hydroxide and materials containing tannin, the mixture is dried and pulverised, and the powder mixed with sodium bicarbonate, talc, antimony trichloride, and a binder, e.g., glue. Cement or other fillers can also be added.

A. R. POWELL.

Kiln for manufacture of fused cement. A. BAUCHERE and G. ARNOU (U.S.P. 1,758,778, 13.5.30. Appl., 26.3.25. Fr., 9.4.24).—See B.P. 232,155; B., 1925, 549.

Production of artificial stone. J. JAKOB, ASSR. to A. T. OTTO & SONS, INC. (U.S.P. 1,758,518, 13.5.30. Appl., 2.11.25. Ger., 22.12.24).—See G.P. 417,360; B., 1926, 130.

Flooring [composition]. C. PIA and G. POLINI (B.P. 327,950, 12.12.28).

[Photogravure process for] reproduction of the natural appearance of articles [e.g., wood, marble] on other surfaces. OXFORD VARNISH CORP., Assees. of L. V. CASTO (B.P. 301,042, 23.11.28. U.S., 23.11.27).

Road oil (U.S.P. 1,745,155). Asphalt etc. masses (B.P. 307,466).—See II. Artificial masses (B.P. 327,722).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Heating [pig-iron] mixers with blast-furnace gas. K. D'HUART (Stahl u. Eisen, 1930, 50, 585—588).—The construction of several types of mixer fired with blast-furnace flue gas is illustrated and a new direct-fired regenerative mixer and ancillary plant is briefly described with reference to diagrammatic plans.

A. R. POWELL.

Recent developments in corrosion- and heat-resisting steels. (Sir) R. HADFIELD, T. G. ELLIOT, and R. J. SARJANT (J.S.C.I., 1930, 49, 41—51 r).—Heat-resisting steels are similar in constitution to corrosion-resisting steels, though silicon and tungsten are further added to give resistance to heat. These various materials may also conveniently be classified according to whether or not they are hardened by quenching in water from high temperatures. The austenitic steels offer generally greater resistance to corrosion. Particulars are given of the resistance to various corroding agents, such as the atmosphere, sea-water, mineral and organic acids, and steam contaminated with salt spray. The significance of mechanical tests at high temperatures is discussed, and an alloy Era H.R.1 is described having a limiting creep stress of $2\frac{1}{2}$ tons per in.² at 800°. In regard to resistance to scaling, the heat-resisting steels give as good a performance at 950° in 4000 hrs. as the best cast irons at 800° in only 100 hrs. Tables are given showing the mechanical and physical qualities of the various materials, and their working properties are also described. Typical applications of the special steels made by the authors' firm are mentioned.

Ageing of steel castings. A. POMP (Stahl u. Eisen, 1930, 50, 440—441).—The impact strength of mild-steel castings (notched bars) containing 0.1—0.27% C, 0.16—0.46% Si, 0.45—1.37% Mn, 0.05—0.06% P, and 0.04% S decreases on ageing at 250° for 1 hr. after forging to a reduction in area of 7—10%, but not nearly to the same extent as it does after completely annealing. The values obtained for the annealed metal at 20° to —20° were consistently below 2 mkg./cm.², whereas those for the aged metal varied from about 6 mkg./cm.² at 20° to about 2—3 mkg./cm.² at —20°.

A. R. POWELL.

Alloy steels in theory and practice. A. RYS (Stahl u. Eisen, 1930, 50, 423—438).—A lecture delivered at the annual meeting of Society of German Steel Founders describing the mechanical, physical, and chemical properties of alloy steels and the effect of heat and mechanical treatment on the properties of forged steels.

A. R. POWELL.

Tensile strength at high temperatures of steel containing small quantities of nickel and molybdenum. W. LIESTMANN and C. SALZMANN (Stahl u. Eisen, 1930, 50, 442—446).—The tensile properties of steel with 0.2% C, 0.8% Mn, 0.25% Si, 0.4—2.2% Ni, and 0.2—0.6% Mo have been determined at 500° after annealing at 900—950°. Nickel and molybdenum both increase the tensile strength and yield point, but reduce the ductility, 0.1% Mo increasing the ultimate strength by 1 kg./mm.² and the yield point by 2 kg./mm.², and 0.5% Ni increasing the former by 2 kg./mm.² and the latter by 1.8 kg./mm.² With higher proportions of molybdenum the ratio ultimate strength/yield point is unfavourably affected, but further addition of nickel improves this ratio. The steel with 2.1% Ni and 0.5% Mo has a yield point of 25.5 kg./mm.², a tensile strength of 42 kg./mm.², an elongation of 11%, and a reduction in area of 20% at 500°. A. R. POWELL.

Cast steel as a constructional material for machines. F. W. DUESING (Stahl u. Eisen, 1930, 50, 438—440).—Mild-steel castings for machinery parts must be thoroughly annealed before being incorporated in the work; they should have an average yield point of 28 kg./mm.², a tensile strength of 46 kg./mm.², an elongation of 30%, and a reduction in area of 40—50%. These values are well above the German standard specifications, so that with reasonable care these should readily be fulfilled. A. R. POWELL.

Alloy steels for locomotive construction. W. A. JOHNSON (Proc. Inst. Mech. Eng., 1929, 1087—1097).—Carbon steels cannot be relied upon to give a tensile strength above about 55 tons/in.², and such steels have low impact resistance if the tensile strength is high. Alloy steels on the contrary may have good impact strength, a tensile strength of 40—110 tons/in.², and are more easily heat-treated. Hence the use of nickel steel, vanadium steel, and nickel-chromium-molybdenum steel facilitates a reduction in deadweight. A recently developed German steel of similar properties contains 0.1—0.15% C and 0.67—1.5% Si. No difficulty in machining usually occurs. The subject is discussed chiefly from the engineering point of view. It is concluded that the possible saving of weight on a locomotive is 7%. C. IRWIN.

Heat-treatment of locomotive parts. W. A. STANIER (Proc. Inst. Mech. Eng., 1929, 1069—1073).—A graph is given showing the appropriate temperature for the heat-treatment of steel forgings with increasing carbon content, together with photomicrographs of samples treated in various ways. C. IRWIN.

Destructive action of zinc, at and above galvanising temperatures, on metals and alloys. V. Cast iron, malleable iron, case-hardened and alloy steels. W. G. IMHOFF (Amer. Metal Market, 1930, 37, No. 29, 6).—Alloy steels containing higher contents of chromium and nickel are more readily dissolved by molten zinc; impurities in the steel favour dissolution.

CHEMICAL ABSTRACTS.

Gases in refined copper. A. E. WELLS and R. C. DALZIEL (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 270, 3—15).—It is considered that colloidal dis-

persed cuprous oxide adsorbs gases, part of which is liberated on agglomeration. Evolution of nitrogen, water, and carbon dioxide is the chief cause of porosity.

CHEMICAL ABSTRACTS.

Oxides in brass. O. W. ELLIS (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 283, 3—19).—Retention of the charge in the furnace between the first and second pours increases the "oxide count" only in the absence of flux. Poling has a beneficial effect on charges to which flux has been added. CHEMICAL ABSTRACTS.

α - β -Transformation in brass. A. J. PHILLIPS (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 288, 3—9).—Conversion of β - into α -brass takes place rapidly if there is no change in composition; the α -structure was produced by quenching brass containing 62% Cu. CHEMICAL ABSTRACTS.

Effects of oxidation and certain impurities in bronze. J. W. BOLTON and S. A. WEIGAND (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 281, 3—17).—Oxidation by the furnace atmosphere causes shrinkage, loss of strength, sluggishness of metal, and loss of zinc. The undesirable effects of silicon, sulphur, and aluminium on the properties of bronze melted in a neutral furnace atmosphere are discussed.

CHEMICAL ABSTRACTS.

α -Phase boundary of the ternary system copper-silicon-manganese. C. S. SMITH (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 292, 3—32).—Alloys containing more than 90% Cu were studied. The addition of manganese causes a fall in the temperatures of the reactions in the copper-silicon system; for 2.5% Mn the peritectic reaction is at 760°, at which temperature there is a quaternary reaction with Mn₂Si. The solubility of this compound diminishes rapidly with fall of temperature. CHEMICAL ABSTRACTS.

Thermal conductivity of copper alloys. C. S. SMITH (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 291, 3—24).—On passing from pure copper to the saturated α -solid solution (39% Zn) the thermal conductivity falls from 0.941 to 0.285 g.-cal. per cm.² per cm. per sec. per 1°. The appearance of the β -phase causes an increase in the conductivity and a rapid decrease in the temperature coefficient.

CHEMICAL ABSTRACTS.

Mechanism of the corrosion of duralumin by sea-water. E. HERZOG and G. CHAUDRON (Compt. rend., 1930, 190, 1189—1191).—The rate of corrosion of duralumin immersed in sea-water or 3% sodium chloride solution in an atmosphere of oxygen increases linearly with the gas pressure up to 90 atm., but only slowly thereafter (up to 130 atm.). The position of the test-piece plays an important part, and corrosion is greater for horizontal than for vertical immersions. Corrosion by this method, or by addition of hydrogen peroxide, is inhibited or arrested by addition of small quantities of phosphates, alkali borates, salts of magnesium, and especially of manganese, zinc, and titanium, which, presumably, fix or destroy the hydrogen peroxide added or formed during corrosion under pressure. J. GRANT.

Corrosion by superheated steam. J. K. RUMMEL (Iron Age, 1929, 124, 1525—1527).—By determining the

hydrogen evolved, the rates of corrosion of various steels in superheated steam at pressures of 220–3450 lb. per in.² were determined. In steam free from oxygen the corrosion is negligible up to 430°, but corrosion and evolution of hydrogen are, in general, accelerated by the presence of oxygen. Within limits, alkalinity does not stop corrosion due either to the action of oxygen or to the evolution of hydrogen. CHEMICAL ABSTRACTS.

Hydrogen-ion concentration [in water works' corrosion problems]. S. GOTTLIEB (J. Amer. Water Works' Assoc., 1930, 22, 543–544).—An error in Van Giesen's paper (cf. B., 1930, 286), relating to that author's explanation of the p_H range of soils causing corrosion, is pointed out and corrected. C. JERSON.

Determination of aluminium and magnesium in zinc-base die-casting alloys. C. M. CRAIGHEAD (Ind. Eng. Chem. [Anal.], 1930, 2, 188–190).—These alloys contain about 90% Zn, which creates difficulties in most methods for the determination of aluminium and magnesium. Electrolysis with a weakly acid solution and a mercury cathode, however, completely separates these constituents. The alloy (2 g.) is dissolved in 20 c.c. of 50% sulphuric acid and 100 c.c. of water are added. The solution is electrolysed for 5 hrs. with a current density of 1 amp./6.25 cm.², the aluminium is precipitated by Blum's method, and the magnesium as phosphate. C. IRWIN.

X-Ray determination of particle size. R. BRILL (Metall-Wirt., 1929, 8, 699–701; Chem. Zentr., 1930, i, 125).—The particle size of iron liberated by thermal decomposition of iron carbonyl is 10^{-6} cm. A. A. ELDRIDGE.

Application of microscopical analysis to mixtures of metals and alloys. W. F. WHITMORE and F. SCHNEIDER (Ind. Eng. Chem. [Anal.], 1930, 2, 173–176).—Microscopical tests up to the present have been used mainly for confirmatory purposes with previous separation of the different elements present. A new system of separation is given which omits the use of hydrogen sulphide and separates the more common metals into four groups. The different elements in each group can be identified by described methods of micro-analysis without further separation. Photomicrographs of the salts used for identification purposes are given. C. IRWIN.

Utility of the platinum metals in chemical industry. E. R. THEWS (Chem. Fabr., 1930, 49–53).—The chemical and physical properties of the six metals of the platinum group, together with various applications of the metals and of their alloys, such as for the resistors in electric furnaces, for crucibles, etc., are described. C. IRWIN.

Superconducting alloy with resistance-temperature hysteresis. J. C. McLENNAN (Nature, 1930, 125, 447).—The resistance of an alloy of bismuth, lead, and tin decreased slowly with temperature down to 9° Abs., at which point it suddenly fell to zero. On raising the temperature the alloy remained superconducting to 13.2° Abs. and then the resistance reappeared and rose to a steady value at 13.8° Abs. L. S. THEOBALD.

Properties of Permalloy-C. ANON. (Engineering, 1930, 129, 567).—An alloy consisting of about 80% Ni

and 20% Fe, known as Permalloy-A, has been displaced by a modified alloy known as Permalloy-C. Magnetically, this alloy is the softest material available, and it has a resistivity equal to that of silicon-steel. The alloy, produced in a high-frequency tilting furnace, can be rolled into sheet or rod, and it requires only a single annealing process, care being taken in the regulation of the heating and cooling conditions. The use of this alloy should result in marked improvement in the design of electrical relays and measuring instruments. C. A. KING.

Electrolytic preparation of zinc. G. EGER (Chem.-Ztg., 1929, 53, 857–858, 878–879).—Technical practice in the preparation of electrolytic zinc in Europe and America is briefly reviewed. G. E. WENTWORTH.

Hardness and polishing of electrodeposits. D. J. MACNAUGHTAN and A. W. HOTHERSALL (J. Electroplaters' Dep. Tech. Soc., 1930, 5, 63–82).—Scratch tests are untrustworthy; the Brinell test is accurate. As ordinarily deposited, metals have the following ranges of hardness: lead 3–5, cadmium 12–53, zinc 40–50, silver 61–130, copper 58–150, iron 167–350, nickel 155–420, cobalt 270–311, chromium 500–900. Abrasion hardness and the influence of the electrolyte are discussed. CHEMICAL ABSTRACTS.

Ground-coat for enamels. MALINOVSKY. **Refractories and cements.** WHITE.—See VIII. **Absorption of mercury by charcoal.** RAKUSIN.—See XXIII.

PATENTS.

Reduction of [iron] ores. J. HERRMANN, B. G. FRANZEN, L. MACB. HUBBARD, and E. R. ZACHARIAS (B.P. 327,687, 4.1.29).—Iron pyrites or other iron ore is melted and supplied to an electric furnace in which it is subjected to the action of carbon dioxide under pressure and to a magnetic field at 800–900°. It is claimed that steel is thus obtained which is drawn out of the slag by the action of the magnetic field. A. R. POWELL.

Smelting of oolitic granules and similar finely-divided ores or slimes obtained from minette. P. GREDT (B.P. 328,240, 15.10.28. Addn. to B.P. 240,165; B., 1925, 996).—The ore is fed into the top of a mechanically-rabbed, multiple-hearth furnace on the upper hearths of which it is preheated by the combustion of waste gases from the lower hearths in which the ore is reduced by hot gases supplied from a common producer to every hearth from a common pipe-line. A. R. POWELL.

Manufacture of shaped metal articles [pure sheet iron]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 327,955, 14.12.28).—Scrap iron, preferably slightly oxidised, is cut into small pieces which are mixed with iron powder obtained by the decomposition of iron carbonyl, the mixture is pressed into an ingot and sintered in hydrogen at 950°, and the mass is forged and rolled into sheet. A. R. POWELL.

Production of shaped bodies for tools of material difficult to work, such as carbides, their alloys, etc. F. KRUPP A.-G., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 313,619, 14.6.29, Ger., 15.6.28).—The material, e.g., a mixture of carbides

and nickel or cobalt powder, is mixed with a binding material which sets at the ordinary temperature, *e.g.*, shellac, gums, dextrin, or cellulose acetate, and the mixture is pressed into shapes which are allowed to harden. After grinding and polishing to obtain the more complicated shapes, if desired, they are heated to remove the binding material and sintered. A. R. POWELL.

Heat-treatment of steel. H. HANEMANN (B.P. 304,196, 10.1.29. Ger., 16.1.28).—The metal is heated in the ordinary way to a temperature just below the Ar3 point, then heated suddenly by electrical resistance or induction to a temperature about 50° above the Ar3 point, and allowed to cool. This procedure produces a uniform fine-grained structure throughout the metal. A. R. POWELL.

Improving the fatigue strength of metal work pieces. A. L. MOND. From I. G. FARBEIND. A.-G. (B.P. 327,660, 15.8.29).—Metal articles, *e.g.*, engine casings, which may be subjected to fatigue stresses in use, are subjected to continuous dynamic stresses, which at first are considerably lower than the initial fatigue limit and which later increase more nearly to, but are always less than, that value. C. A. KING.

Treatment of iron prior to coating with tin or lead. G. RADTKE (MATUSCHEK METALLIND. G. RADTKE) (B.P. 308,804, 26.3.29. Ger., 29.3.28).—After the usual pickling in acid baths, the metal is rinsed and immersed in a boiling dilute solution of a nickel or cobalt salt for 10–30 min., rinsed, and dipped immediately into the coating bath. A. R. POWELL.

Coating and heat-treating [metals, *e.g.*, iron to be galvanised or tinned]. A. O. AUSTIN, Assr. to OHIO BRASS Co. (U.S.P. 1,740,639, 24.12.29. Appl., 25.5.25).—The metal is dipped into the cooler end of a long galvanising, tinning, or heat-treating bath, covered with a layer of flux, and slowly drawn towards the hotter end through a series of baffles on the surface of the bath, which serve to retain the impurities at the cooler end. A. R. POWELL.

[Inhibitor for pickling] treatment of metals with acid liquids. SOC. CHEM. IND. IN BASLE (B.P. 313,135, 7.6.29. Switz., 7.6.28).—A small quantity of a base of the di- or tri-arylmethane series, with or without a mineral salt, sulphite-cellulose liquor, a sulphonated oil, mono- or (*as*) di-acylated diamines, or a sulphonated tar product, is added to a hydrochloric acid pickling bath for cleaning iron or steel; *e.g.*, 0.02% of 4:4'-tetramethyldiaminodiphenylmethane hydrochloride or 0.01% of 3:3'-diamino-4:4'-dimethyldiphenylmethane hydrochloride is added to 15% hydrochloric acid. A. R. POWELL.

Manufacture of nickel-iron alloys. GEN. ELECTRIC Co., LTD., S. V. WILLIAMS, and G. R. POLGREEN (B.P. 327,865, 18.4.29).—A solution containing nickel, iron, and, if desired, cobalt salts is treated with an excess of sodium hydroxide and the precipitate is collected, washed, dried, and heated at 900° in hydrogen. The resulting alloy powder is ground in a ball-mill and used for the manufacture of dust cores for loading coils. A. R. POWELL.

Manufacture of [nickel-iron alloy] metal dust. STANDARD TELEPHONES & CABLES, LTD., J. C. CHASTON,

and A. F. B. NORWOOD (B.P. 327,419, 4.10.28).—Nickel-iron alloys containing cobalt or other metals are partially oxidised to render them brittle then pulverised in a ball-mill. The fine dust is removed by screening and the oversize annealed at a temperature at which it recrystallises to a fine-grained structure; the resulting material may be readily pulverised to obtain powder for the manufacture of dust cores. A. R. POWELL.

Soldering or like methods of joining metal parts. G. W. CHRISTOPH (B.P. 327,742, 11.10.28).—The parts, *e.g.*, of a radiator, are fitted into position and the whole structure is dipped into a molten metal, which fills in the interstices between the various sections. Excess of the solder is then removed by making the article the anode in a suitable electrolytic bath. A. R. POWELL.

Coating for welding rods. H. R. PENNINGTON, Assr. to HOLLUP CORP. (U.S.P. 1,745,267, 28.1.30. Appl., 22.5.26).—The coating comprises a mixture of 70% of powdered carbon and 30% of iron powder, bonded with sodium silicate. A. R. POWELL.

Articles of high resistance to wear, well polishable, and neutral to chemical agents. F. HAUPTMEYER (B.P. 307,011, 5.1.29. Ger., 1.3.28).—Teeth fillings and objects used in the brewing industry are made from a mixture of nickel-chromium, nickel-chromium-iron, or nickel-chromium-steel alloy powder mixed with a suitable cement. A. R. POWELL.

Granulation of blende. SOC. ANON. LA NOUVELLE MONTAGNE (B.P. 328,162, 27.6.29. Belg., 7.6.29).—A mixture of raw blende and more or less completely roasted blende is fed on to a conveyor belt from a series of hoppers between each pair of which the mixture is sprayed with dilute sulphuric acid, zinc sulphate solution, or, if the mixture contains zinc sulphate, water. The moist powder sets rapidly to a granular mass owing to the formation of a basic zinc sulphate. A. R. POWELL.

Zinc-base alloys and articles made therefrom. NEW JERSEY ZINC Co., Assees. of W. MCG. PEIRCE and E. A. ANDERSON (B.P. 305,651, 31.12.28. U.S., 9.2.28).—The alloys contain a minimum of 92% Zn and preferably more than 95% Zn, 0.005–0.5% Mg, and 0.05–5% Cu; they are suitable for rolling into sheets of high strength and high resistance to corrosion. [Stat. ref.] A. R. POWELL.

Preparation of aluminium and aluminium alloys. A. PACZ (B.P. 303,755, 7.1.29. Ger., 7.1.28).—The alumina used in the cryolite bath is prepared by the use of two alkali hydroxides, *e.g.*, sodium and potassium or sodium and lithium hydroxides, or small quantities of the second alkali in the form of aluminate, carbonate, hydroxide, or borate are added to the cryolite bath. By the addition of titanium, zirconium, or silicon compounds to the bath alloys of aluminium with these metals are obtained. The products obtained by the process disclosed are claimed to be superior in mechanical properties to aluminium and its alloys obtained by the usual method. A. R. POWELL.

Coating aluminium, magnesium, and their alloys with oxides of manganese. O. SPRENGER PATENTVERWERTUNG JIROTKA M.B.H., and B. JIROTKA (B.P. 328,485,

4.6.29. Addn. to B.P. 314,769; B., 1929, 725).—Manganous, ferrous, or stannous salts are added to the baths claimed in the chief patent, *e.g.*, the bath comprises a solution of 10.8 g. of potassium permanganate, 32 g. of potassium dichromate, 5 g. of manganous chloride, and 10.4 c.c. of hydrofluoric acid per litre. A. R. POWELL.

Recovery of tin from scrap tin-plate and like tin-bearing materials. H. WADE. From W. B. BALANTINE and M. G. GILBERT (B.P. 327,997, 15.1.29).—The scrap is immersed in a boiling solution of lead acetate in sodium hydroxide, whereby the tin replaces the lead. When all the lead is precipitated the tin is recovered by precipitation with zinc or by treating the solution with carbon dioxide. In the latter case the alkali is regenerated by addition of milk of lime and the acetic acid recovered as calcium acetate by crystallisation. The lead-coated iron is pickled with dilute acetic acid to regenerate lead acetate for use again.

A. R. POWELL.

Electrolyser for separation of tin from tin salt solutions. L. U. CORSA (B.P. 313,166, 30.5.29. Italy, 9.6.28).—The apparatus comprises a semi-cylindrical trough with two side troughs of rectangular cross-section at its upper ends. The large trough acts as cathode and the smaller troughs serve for the collection of the sponge tin which is removed from the inside surface of large trough by means of a scraper attached to a rotating anode, which revolves around the horizontal axis of the trough.

A. R. POWELL.

Electrical annealing [of metals]. M. FARMER (U.S.P. 1,739,958, 17.12.29. Appl., 2.8.26).—The apparatus comprises a refractory base on which the metal articles are stacked, a gastight hood which can be lowered over the articles, and a second hood of refractory material lined internally with electrical resistors and adapted to fit over the first hood. Means are provided for filling the heating chamber with an inert or reducing gas.

A. R. POWELL.

Electroplating metals with chromium. J. BAUER (B.P. 327,911, 17.6.29).—To prevent spraying, to each litre of the bath 0.5 c.c. of light petroleum or gasoline is added, or the surface of the bath is heated by radiation from electrical resistors heated at 800°.

A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 304,668, 23.1.29. U.S., 23.1.28).—An electrolytic bath for cadmium plating contains, per gal. of water, 1—6 oz. of cadmium (as sulphate), 1—10 oz. of free sulphuric acid, 2—12 oz. of ammonium sulphate, and 1—10 g. of glue.

C. A. KING.

Production of gold and silver [from scrap iron]. V. VOLPATO (B.P. 306,048, 14.2.29. Italy, 14.2.28).—Scrap iron or steel that has been subjected to high stress is exposed to the prolonged action of a magnetic force that imparts a centripetal velocity to the electrons, and the treated metal is dissolved in cold hydrochloric acid. The colloidal precious metals which are said to result from this treatment may be coagulated by the addition of Fontainebleau sand.

A. R. POWELL.

Metal polish. R. J. THOMPSON (B.P. 327,479, 17.1.29).—A paste, claimed to give a lasting silver effect

to brass or copper and to render steel stainless, consists of tin, antimony, silver, and mercury, made into an amalgam and powdered; to this is added silver oxalate, together with whiting, putty powder, aluminium chloride, and the usual colouring agents and vehicles.

L. A. COLES.

Mechanical ore-roasting furnace. J. HARRIS (U.S.P. 1,758,154, 13.5.30. Appl., 21.5.28. U.K., 2.6.27).—See B.P. 287,356; B., 1928, 372.

Process and furnace for extracting metals from ores. S. C. G. EKELEND (U.S.P. 1,758,786, 13.5.30. Appl., 15.10.26. Swed., 19.10.25).—See B.P. 309,651; B., 1929, 522.

Apparatus for subjecting a mass of powdered or granular material to the action of gases, particularly applicable to the calcining and roasting of ores and similar materials. H. McK. RIDGE (U.S.P. 1,758,805, 13.5.30. Appl., 9.5.28).—See B.P. 305,883; B., 1929, 522.

Proofing of iron and steel against rust. W. H. COLE (B.P. 328,580, 29.1.29. Addn. to B.P. 289,906; B., 1929, 488).—See U.S.P. 1,719,464; B., 1929, 781.

Foam-producing process and material for acid metal-cleaning baths. J. H. GRAVELL, Assr. to AMER. CHEM. PAINT CO. (U.S.P. 1,757,959, 13.5.30. Appl., 29.3.27. Renewed 20.3.30).—See B.P. 287,911; B., 1929, 725.

Producing a [rubber] coating stable towards acids and alkalis on metallic articles. F. AHRENS, Assr. to HARZER ACHSENWERKE GES.M.B.H. (U.S.P. 1,758,420, 13.5.30. Appl., 7.7.27. Ger., 18.5.25).—See B.P. 283,049; B., 1928, 204.

Material containing carbon, iron, and iron oxide (B.P. 308,351).—See II. Flotation agents (B.P. 316,282). Catalysts for ketones (B.P. 315,818).—See III. Paint for iron surfaces (B.P. 305,218). "Alkyd" resins (B.P. 299,424).—See XIII. Coating metal with rubber (B.P. 327,452). Rubber-metal adhesive (U.S.P. 1,744,840).—See XIV.

XL—ELECTROTECHNICS.

Water-gas equilibrium. PETERS and KÜSTER.—See II. Refractories and cements. WHITE.—See VIII. Zinc-base alloys. CRAIGHEAD. Superconducting alloy. McLENNAN. Permalloy-C. ANON. Zinc. EGER. Hardness etc. of electrodeposits. MACNAUGHTAN and HOTHERSALL.—See X. Testing equipment for paints etc. GARDNER and SWARD.—See XIII. p_H of tan liquors. GOLDMAN.—See XV.

PATENTS.

Provision of inert atmospheres [in electrical apparatus etc.]. C. A. STYER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,743,167, 14.1.30. Appl., 29.12.23).—An inert atmosphere is formed above the oil in transformers, circuit breakers, etc. by providing an apparatus through which the air passing into the vessel, or already therein, can circulate, and is thereby brought into contact with hot carbonaceous material, *e.g.*, sugar-carbon. The latter is supported on or between gauze partitions and is heated electrically.

A. B. MANNING.

[Leading-in] electric insulators [for gas-purification apparatus]. STEATIT-MAGNESIA A.-G. (B.P. 319,317, 15.7.29. Ger., 20.9.28).

[Hydrometric device for testing] electric storage batteries. S. FRAIS (B.P. 328,774, 19.4. and 31.7.29).

Heating and cooling of liquids (B.P. 327,729). Measurement of heat quantities (B.P. 305,532). Recording pyrometers (B.P. 327,861).—See I. Drying of peat etc. (B.P. 304,329). Material containing carbon, iron, and iron oxide (B.P. 308,351). Miner's lamp (U.S.P. 1,744,416).—See II. Reduction of iron ores (B.P. 327,687). Nickel-iron alloys (B.P. 327,865 and 327,419). Soldering etc. (B.P. 327,742). Tin from tin salt solutions (B.P. 313,166). Annealing of metals (U.S.P. 1,739,958). Chromium (B.P. 327,911). Cadmium plating (B.P. 304,668).—See X. Coating metal with rubber (B.P. 327,452).—See XIV.

XII.—FATS; OILS; WAXES.

Component glycerides of Borneo (Illipé) tallow. T. P. HILDITCH and J. PRIESTMAN (J.S.C.I., 1930, 49, 197—200 r).—The mixed fatty acids of a sample of neutral Borneo (Illipé) tallow (m.p. 36—36.5°, setting point 28.8°, saponification equivalent 290.7, iodine value 32.3, and unsaponifiable matter 0.74%) consisted of myristic (1.5%), palmitic (21.5%), stearic (39.0%), and oleic (38.0%) acids. The fat contained only 4.5% of fully saturated glycerides, the acids present in which were palmitic (57%) and stearic (43%); the remainder of the fat (95.5%) therefore consisted of mixed saturated-unsaturated glycerides, with saturated and unsaturated acids in the molecular ratio of 1.55:1. This corresponds with a general glyceride structure of (1) fully-saturated glycerides (4.5%); (2) mono-oleodisaturated glycerides (78 to 87%); (3) dioleomonosaturated glycerides (17.5—0.0%); and (4) triolein (0—8.5%). If the triolein content (which cannot at present be directly determined) were 4%, the amount of these four classes in the fat would be (1) 4.5%, (2) 85%, (3) 6.5%, and (4) 4%. Experimental verification was obtained of the presence of oxidation products of mono-oleodisaturated glycerides equivalent to a content of over 63% of the latter in the original fat, whilst it was also indicated that oleopalmitostearins predominated over other glyceride forms. Borneo (Illipé) tallow is therefore very similar in general structure to cacao butter (Lea, B., 1929, 331), but contains somewhat less oleic acid and a higher ratio of stearic to palmitic acid than the latter; the total amount of mono-oleodisaturated glycerides is even higher in Borneo tallow than in cacao butter, but there is probably more oleodistearin present in the former fat by reason of its relatively greater stearic acid content.

Fatty acids of nutmeg (mace) butter and of expressed oil of laurel. G. COLLIN and T. P. HILDITCH (J.S.C.I., 1930, 49, 141—143 r).—The quantitative composition of the mixed fatty acids of nutmeg butter (from *Myristica officinalis*), of the fat from *M. malabarica* kernels, and of expressed oil of laurel have been determined with the following results: (1) Nutmeg butter: lauric (1.5%), myristic (76.6%), palmitic (10.1%), oleic (10.5%), and linoleic (1.3%) acids. (2) *Myristica*

malabarica fat: myristic (39%), palmitic (13%), other saturated acids (3%), oleic (44%), and linoleic (1%) acids. (3) Expressed oil of laurel: lauric (35.0%), palmitic (9.7%), oleic (36.6%), and linoleic (18.7%) acids. All the oils contained appreciable amounts of resinous compounds in addition to fats; the laurel oil was a genuine trade sample, and evidently contained fats from both the kernel and the pericarp. Examination of the individual fats from the kernels and pericarps of laurel berries is in progress.

Dika fat (*Irvingia butter*). G. COLLIN and T. P. HILDITCH (J.S.C.I., 1930, 49, 138—139 r).—The white solid fat from the kernels of *Irvingia Barteri* (Nigeria) had saponification equivalent 233.9, iodine value 9.1%, unsaponifiable 1%, and melts at 41—42°; its constituent fatty acids were lauric (38.8%), myristic (50.6%), and oleic (10.6%). The composition of the mixed fatty acids was thus similar to that given by previous workers for the kernel fats of other *Irvingia* species from West Africa and of *I. Oliveri* (Cay-Cay fat from Indo-China). Although quite suitable for edible purposes, the low fat content of the nuts (about 12%) and the difficulty of removing the massive shells from the kernels will probably restrict the commercial outlets for this fat.

Cholesterol of buffalo butter. E. DE'CONNO and L. FINELLI (Annali Chim. Appl., 1930, 20, 148—154).—The buffalo butter examined yielded 87% of fat having d_{40}^{20} 0.865, m.p. 32°, solidif. p. 20°, m.p. of insol. fatty acids 41°, solidif. point of insol. fatty acids 38°, acid value 1.11, saponif. value 228, iodine value 37, acid value of soluble volatile acids (Reichert) 27, acid value of insol. volatile acids (Polenske) 1.30, butyrefractometer number at 35° 45. The cholesterol, $C_{27}H_{45} \cdot OH, \frac{1}{2}H_2O$, separated by Bömer's method (A., 1899, ii, 191) gave all the colour reactions of ordinary cholesterol, had m.p. 136°, $[\alpha]_D^{20}$ —33° 3', and yielded a formate, m.p. 86°, $[\alpha]_D^{20}$ —56° 52', an acetate, m.p. 104°, $[\alpha]_D^{20}$ —51° 56', a benzoate, m.p. 134°, $[\alpha]_D^{20}$ —9° 51', and a dibromide, $C_{27}H_{46}OBr_2$, m.p. 101°. The cholesterol is probably a higher homologue of the cholesterol of cows' milk or bile. T. H. POPE.

Occurrence of arachidonic acid in lard. J. B. BROWN and E. M. DECK (J. Amer. Chem. Soc., 1930, 52, 1135—1138).—Four specimens of commercial lard have been shown to contain 0.31—0.40% of arachidonic acid (cf. Ellis and Isbell, A., 1926, 972), as shown by the isolation of methyl octobromoarachidonate from the methyl esters. Direct bromination of lard yielded ether-insoluble bromine additive products which were probably a mixture of α -palmitodistearin and a glyceride of octabromoarachidic acid. R. K. CALLOW.

Babassu fat. A. HEIDUSCHKA and R. AGSTEN (J. pr. Chem., 1930, [ii], 126, 53—64).—Babassu fat was found, on hydrolysis, to yield the following fatty acids: hexoic (0.1%), octoic (6.5%), decoic (2.7%), lauric (45.8%), myristic (19.9%), palmitic (6.9%), and oleic acid (18.1%). H. A. PIGGOTT.

Continuous working in the soap industry. W. PROSCH (Chem. Fabr., 1930, 3, 158—160, 166—168).—A general description is given of modern plant layout and machinery for continuous and automatic working

in the production of finished soap. The manufacture of milled toilet soaps (cooling on chilled rolls followed by oven-drying of the soap shreds; drying on hot rolls followed by shredding, milling, etc.), household soaps (cooling in blocks, cutting, continuous drying ovens, etc.), and soap powders (spray-drying) is considered.

E. LEWKOWITSCH.

Determining volatile hydrocarbons in soap. C. T. N. MARSH (Ind. Eng. Chem. [Anal.], 1930, 2, 166—167).—The standard U.S. method for the determination of hydrocarbons in naphtha soaps is modified as follows. Calcium chloride solution is added to the distillation flask to reduce foaming and allow quicker distillation. The naphtha is collected in a Mohr's burette connected to a levelling tube and coloured with a particle of oil-soluble dye. It is allowed to run quietly down the burette side, thereby avoiding emulsification.

C. IRWIN.

Lubricating greases made from soaps of phenylstearic acid. W. S. GILFOIL (Ind. Eng. Chem., 1930, 22, 487).—Calcium, sodium, and lead phenylstearates used as bases for lubricant greases show no advantage over the usual oleate soaps.

J. O. CUTTER.

Determination of fully-saturated glycerides as an aid in the analysis of fats. B. C. CHRISTIAN and T. P. HILDITCH (Analyst, 1930, 55, 75—90).—An application to ordinary analytical practice has been made of the general method of oxidation of neutral fats in acetone solution with potassium permanganate, whereby the unsaturated fatty acid groups combined with glycerol are quantitatively converted into acidic products and fully saturated glycerides are left untouched (cf. B., 1928, 791; 1929, 253). Not more than 50 g. of fat are used, and the results are arrived at in 2 days or less. The order of accuracy of the percentage of fully-saturated glycerides, and still more of their apparent saponification equivalents, is less than in the more elaborate process, but empirical correction factors are introduced. For details of procedure, the original paper must be consulted. The ether is evaporated from the main ethereal extract of neutral products of oxidation, and from the ethereal extract from the primary washings with ammonia and water, and, after drying and weighing, determinations are made of the iodine values, saponification equivalents, acid values, and m.p. It was found that the fully saturated glyceride contents of the nut oils and some other fats are probably almost constant for a given fat, but units of variation for such fats as butter, tallows, oleo and palm oils require further study. The method was found most useful for the detection of nut oils in butter fats, since the proportion of fully-saturated glycerides in nut oils is much higher than in butter fats, and an approximate estimation of the percentage of nut fat present can be made if its nature is known. Diminution in the saponification value of the fully-saturated glycerides of carcase fats and increases in the m.p., as compared with the figures for butter, make the detection of such fats in butter and ghee possible, though not so exactly as in the case of nut oils. The utility of the process for elucidating margarine fat mixtures is not so marked, but it is considered that by accumulation of sufficient data for

a large range of typical mixtures, and a consideration of other factors, the determination of the fully-saturated glyceride content will prove useful. D. G. HEWER.

Detection of fats in ultra-violet light. J. LENFELD (Z. Fleisch- Milch-hyg., 1929, 39, 451—454, 477—481; Chem. Zentr., 1929, ii, 2841).—Changes in appearance observed when various edible fats are exposed to the light of the analytical quartz lamp are recorded. Marked differences in fats from the same species were often observed.

A. A. ELDRIDGE.

Constitution and drying of tung oil. G. G. SWARD and H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 58—62).—A brief *résumé* of the present knowledge of the structure of tung oil.

S. S. WOOLF.

Removal of mucilage from linseed oil. F. FRITZ (Farben-Ztg., 1930, 35, 1408—1409).—A brief account is given of the available methods of freeing linseed oil from mucilage. Although various processes are patented, it would appear that a simple heating process is still widely used. The greening of linseed oil in these processes is not caused by the use of copper kettles, as has been suggested, but is due to a colour change of the natural yellow colouring matter of the oil.

S. S. WOOLF.

Hexabromide determination on linseed oil. F. FRITZ (Chem.-Ztg., 1930, 54, 383).—The ether solution of the fatty acids and the wash-ether may be conveniently cooled by the direct addition of carbon dioxide snow. A modified procedure for the determination is outlined, using 1 g. of fatty acids, and removing the ether and (small) excess bromine by a stream of dry air; ether saturated with hexabromide is used for the separation of the bromides. Hexabromide numbers found for a Baltic and a La Plata linseed oil were 57.8 and 50.5, respectively.

E. LEWKOWITSCH.

Treatment of the fruits of the oil palm. A. STELTJES (Bull. Mat. Grasses, 1929, 357—364).—From experiments conducted by the Institut Coloniale de Marseille it was found that palm fruit was satisfactorily depulped by the Sigg machine; from the pulp so produced an 85—90% yield of oil was obtained with only a single pressing at 40 kg. pressure (cf. Sumatran practice giving about the same yield from double pressing of whole fruit). As the small amounts of broken nut-shells which pass into the pulp would cause rapid wear of the "scourtins," the Estrayer-Gueidon press, which uses none, was employed. The oil produced contained not more than 5% of slimes, of which the bulk was deposited on leaving the press; the remaining 1—1.5% of impurities was easily removed on being passed through a supercentrifuge. The nuts from the depulper were free from fibre. It appeared that this procedure could be adopted with advantage on the large scale. Solvents are preferably to be used only after the bulk of the oil and water has been removed from the fruit by pressure. A rapid procedure for routine determinations of acid value is described, in which 7.8 g. of oil are dissolved in 30—40 c.c. of alcohol and titrated with 0.5*N*-caustic soda (to phenolphthalein); the burette reading indicates directly the free acidity (as % oleic acid).

E. LEWKOWITSCH.

Grape-fruit seed oil. G. S. JAMIESON, W. F. BAUGHMAN, and S. I. GERTLER (Oil & Fat Ind., 1930, 7, 181—183).—Air-dried grape-fruit seeds contain about 30% of a semi-drying oil with a bitter taste, which gives a medium hard, free-lathering sodium soap. An expressed oil (clear yellow) and an extracted oil (greenish-yellow, purple fluorescence by reflected light) had, respectively: d_{20}^{25} 0.9170, d_{15}^{15} 0.9207; n_D^{25} 1.4700, 1.4696; acid value 2.5, 3.3; iodine value (Hanus) 106.3, 100.4; saponif. value 194.1, 194.3; acetyl value 7.7, —; unsaponifiable matter 0.7%, 0.7%; saturated acids (corr.) 26.6, 27.63; unsaturated acids (corr.) 68.5%, 67.77%; iodine value of unsaturated acids 146.2 (155.2 calc.), —. The expressed oil contained the glycerides of oleic (20.5), linoleic (51), palmitic (20.1), stearic (7.6), and lignoceric acid (0.1%), together with 0.7% of unsaponifiable matter. The extracted seed-cake had a bitter taste and contained 21.4% of protein, 38.8% of nitrogen-free extract, and 22% of crude fibre; it would be a good cattle-feed if rendered palatable by admixture with other feeding-stuffs.

E. LEWKOWITSCH.

Canadian pilchard oil. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 43—44).—A sample of Canadian pilchard oil showed iodine value (Wijs) 191.1, saponif. value 192.1, n_D^{25} 1.482, octabromide value 32.7%. On addition of driers, the oil dries slightly faster than does linseed oil similarly treated, but the films produced are slightly harder in the latter case. Pilchard oil contains a considerable percentage of solid fats which separate out at ordinary temperatures, giving the oil a granular appearance and causing dullness in films. It is suggested that the oil could be used in oil paints where high gloss is not essential, and could possibly be worked up for varnish with other oils and resins.

S. S. WOOLF.

Sardine oil colour standards. P. W. TOMPKINS (Oil & Fat Ind., 1930, 7, 55—58).—The difficulty is discussed of finding simple colorimetric standards or reference liquids by which to control not only amber oils, with 5—25 red (Lovibond) and 20—100 yellow, but also oils with a greenish cast (due to the feeding habits of the fish), which have only 2—4 red and 60—150 yellow. Dichromate, iodine, and caramel standards do not accommodate the wide colour-range, and even over short ranges cannot be used to compare both amber and greenish oils. Provisional grading of oils not exceeding 14 red (yellow to match) as "light," and oils over 14 red as "dark" has been adopted in Monterey and San Pedro for the last three seasons.

E. LEWKOWITSCH.

Carnauba wax. ANON. (Riv. comm. ital.-brasil, 1929, No. 12, 33).—Values for carnauba (Brazilian), Japan, and candelilla waxes were, respectively: d_{15}^{15} 0.999, 0.997, 0.947; d_{98}^{98} 0.8422, —, —; m.p. 85°, 51°, 75.8—77.4°; saponif. value 79.8—88.3, 221.3, 105—106; iodine value 13.5, 4.5, 5.2—5.5; fatty acids 47.95, —, —.

CHEMICAL ABSTRACTS.

Acids of montan wax. HOLDE and others.—See II.
Natural fats of goatskins. INNES.—See XV. Fat in milk and cream. ELSDON and STUBBS.—See XIX.

PATENTS.

Manufacture of products resembling wax. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 327,162, 24.9.28. Cf. B.P. 324,631; B., 1930, 519).—A bleached montan wax (B.P. 305,552, 308,996, and 320,854; B., 1929, 273, 425; 1930, 156) is esterified with ricinoleic acid, e.g., by heating at 160° with castor oil (or with castor oil acids) in a current of hydrogen chloride.

C. HOLLINS.

Manufacture of esters of fatty acids and of mixtures containing fatty acids. E. WECKER (U.S.P. 1,758,634, 13.5.30. Appl., 20.6.27. Ger., 23.6.26).—See B.P. 273,276; B., 1928, 902.

Grease-removing compositions (B.P. 328,097).
Decolorising clays (U.S.P. 1,739,734).—See VII.
Oil paints (G.P. 461,383). **Waxes** (B.P. 327,417).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ultramicroscopical study of linseed oil containing metallic driers. J. M. PURDY, W. G. FRANCE, and W. L. EVANS (Ind. Eng. Chem., 1930, 22, 508—510).—Cobalt and lead acetates were separately and in admixture dispersed in refined linseed oil under arbitrarily defined standard conditions. Ultramicroscopical photographs were taken during the period of ageing subsequent to preparation. The photographs show that the cobalt soap particles rapidly dispersed in the media and that peptisation of the lead soap was delayed. The incorporation of cobalt acetate with lead acetate accelerates the dispersion of the lead soap. Acid values of the clear media after settlement of the undispersed soaps show that increasing the percentage of lead decreases the value, although the increase of acid value is greatest with the maximum lead content. Direct ultramicroscopical observation in an electric field shows the soap particles to be negatively charged.

J. O. CUTTER.

Turpentine and wood-turpentine: wood-turpentine of the root-resin of *Pinus sylvestris*. V. KRESTINSKI and F. SOLODKI (J. pr. Chem., 1930, [ii], 126, 1—23).—The wood-turpentine extracted from *Pinus sylvestris* (grown in northern and mid-Russia) by distillation either in steam (extract A) or in steam from an alkaline solution (extract B) has been analysed by determinations of physical constants, and the main constituents have been isolated by fractional distillation under reduced pressure. Extract B contained about 43% of α -pinene, 38.5% of Δ^3 -carene, 6.5% of terpinolene, 7% of two alcohols of empirical formula $C_{10}H_{18}O$ (b.p. 102°/12 mm. or 213—213.5°/760 mm., d_{20}^{20} 0.9371, n_D^{20} 1.48299; and b.p. 106°/12 mm., d_{20}^{20} 0.9371, n_D^{20} 1.48521), the constitution of which is unknown, 3.5% of the sesquiterpene cadinene, and 1.5% of uninvestigated residue. Extract A contains 42.5% of α -pinene, 32.5% of Δ^3 -carene, 4% of terpinolene, 14% of alcohols, 3% of cadinene, and 4% of residue. β -Pinene is absent from both products.

H. A. PIGGOTT.

Wood rosin in antifouling paints. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 25—27).—Parallel exposure tests on anti-corrosive and antifouling paints containing gum resin

and wood rosin show the latter to be equal or possibly slightly superior to the former from the point of view of toxicity to marine animals. The addition of 5% of zinc chromate improves resistance against corrosion. It is pointed out that a well-balanced antifouling paint should contain mercury oxide, cuprous oxide, zinc oxide, and pine oil. S. S. WOOLF.

Silicon esters and their application to the paint industry. G. KING (J. Oil & Colour Chem. Assoc., 1930, 13, 28—50).—The historical development of silicon esters is traced and their properties are described at length. By controlled hydrolysis of ethyl silicates, stone preservative and silicon-ester paint medium are obtained. The use of the latter is discussed, consideration being given to application, suitable pigmentation, disadvantages, etc. S. S. WOOLF.

Settling of paints containing acid, alkaline, or neutral pigment. G. G. SWARD (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 22—24).—Paints were prepared from samples of Titanox previously treated with aqueous sulphuric acid, sodium hydroxide, and ammonia, respectively, and dried. The oil absorptions of these treated pigments were 35.0, 35.5, and 37, respectively, whilst that of the untreated pigment was 36.5. Normal and accelerated settling tests on these paints indicate that the "acid" pigment had the greatest, and the "alkaline" pigment the least, tendency towards settling. No relation apparently existed between oil absorption and rate of settling in these tests. S. S. WOOLF.

Accelerated testing equipment [for paints etc.]. H. A. GARDNER and G. G. SWARD (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 64—69).—Types of accelerated testing equipments employing carbon-arc and mercury-quartz lamps are illustrated. For adhesion and brittleness tests, films are removed from the accelerated weathering cabinet and cooled very rapidly by use of liquid carbon dioxide and an expansion chamber. S. S. WOOLF.

[Exposures of] quick-drying house paints. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 2—11).—The results of two years' exposure tests give indications that the introduction of bodied oil or long-oil varnish into the vehicle of linseed oil paints greatly delays their weathering, but information as to the best type of heat-bodied oil or the optimum concentration is not yet available. S. S. WOOLF.

Rezyl exposures. A. W. VAN HEUCKEROTH and H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 47—57).—The results of 5 months' exposures of various nitrocellulose lacquers containing Rezyls, Rezyl balsam plasticiser, and various other resins, both synthetic and natural, and plasticisers, are detailed and illustrated. S. S. WOOLF.

New-type driers ["Soligenates"]. S. LEVY (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 41—42).—"Soligenate" driers are understood to be lead, manganese, and cobalt salts of naphthenic acids derived from the oxidation of petroleum. High metal content conducive to rapid drying, freedom from resin, absence of tendency to spontaneous com-

bustion, and very little tendency to discoloration of clear varnishes are advantages claimed for these driers. They are soluble in vegetable oils, turpentine, mineral spirits, benzol, and toluol, giving solutions initially clear. Preliminary tests on drying power do not indicate that "Soligenates" are superior to linoleates and resinates in equivalent metal content. S. S. WOOLF.

Red lead. H. GROHN (Farben-Ztg., 1930, 35, 1354—1356, 1406—1408, 1457—1459).—The influence on the red lead-linseed oil system of the following factors was studied: particle size and structure of the red lead; addition of linseed oil fatty acids, glycerin, litharge, and oxides of alkaline-earth metals; variation of type of oil; storing at normal and elevated temperatures; viscosity changes on storing. It was shown that particle size, true red-lead content, and sedimentation volume cannot be directly connected with the thickening process, and that the controlling factors are content of lead monoxide, and its form in the red-lead particles. Linseed oil fatty acids neither retard thickening nor disperse an already thickened system. Glycerin retards the thickening at ordinary temperatures, but is an accelerator at raised temperatures. Litharge accelerates the thickening as does magnesium oxide, but in the presence of litharge in active form lime is a retarder. With the exception of fish oil, the type of oil used appeared to exert no influence on the thickening. The slight fall in viscosity in the early stages of storing is explained by the progressive wetting of the pigment by the oil. These results are fully discussed in the light of previous work, and it is considered that phase-reversal will account for the observed phenomena, the lead glyceride-linseed oil system changing under suitable conditions to a more or less rigid linseed oil-lead glyceride system. S. S. WOOLF.

Determination of titania in titanium white. G. AGAMENNONE (Giorn. Chim. Ind. Appl., 1930, 12, 123—124).—The titanium is dissolved as sulphate, reduced to the titanous state, and titrated with ferric alum: 0.5 g. of the pigment is heated, at first gently and then more strongly, with 8—9 g. of anhydrous sodium sulphate and 25 c.c. of concentrated sulphuric acid until dissolved. The cold liquid is mixed with 150 c.c. of water and heated for a few moments to boiling, the precipitated barium sulphate and silica being subsequently filtered off, and the filtrate and the washing liquor (5% sulphuric acid solution, followed by water) made up to 250 c.c. 100 C.c., diluted with an equal volume of water, are heated to about 50°. Meanwhile 150 c.c. of 5% sulphuric acid are poured through a stop-cock burette (60 cm. long, 3 cm. wide) charged with amalgamated zinc and having a long outlet tube passing into a 750-c.c. vacuum flask. Suction is applied and the acid drawn into the flask at the rate of 30 c.c. per min. The titanium solution is similarly introduced and is followed by 150 c.c. of 5% sulphuric acid and 150 c.c. of water, both used to rinse out the beaker, which is finally washed out into the burette. The air in the flask is displaced by a current of carbon dioxide passed through a second tube traversing the stopper, the flask being then detached and the contents titrated with 0.1*N*-ferric alum after addition of 10 c.c. of 25% ammonium thiocyanate solution. (1 C.c. of ferric alum

solution corresponds with 0.00801 g. TiO_2 .) If the pigment does not dissolve completely in the hot mixture of sodium sulphate and sulphuric acid, it may be fused with potassium hydrogen sulphate. T. H. POPE.

Crystallising lacquers. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 358, Jan., 1930, 70—71).—If phthalic anhydride be dissolved in acetone and added to nitrocellulose compositions (a suggested ratio being 1 pt. of phthalic anhydride to 2 pts. of nitrocellulose) crystallising lacquers are obtained. These appear to crystallise more rapidly on bare than on primed metal surfaces. They are not suitable for exterior exposure. S. S. WOOLF.

Nitrocellulose. R. G. DANIELS (J. Oil and Colour Chem. Assoc., 1930, 13, 99—107).—Routine determination of alcohol content, nitrogen content, and stability of nitrocellulose yields information irrelevant from the point of view of lacquer manufacture, and the elimination of these tests is recommended. Undue significance is given to the individual boiling ranges of the volatile constituents of lacquers, when vapour tension at low temperatures of complex mixtures is the real issue. Legal regulations controlling the manufacture and use of nitrocellulose lacquers have not kept pace with the growth of the industry and thus present difficulties. It is considered that separate recommendations are necessary for manufacturer and consumer. The datum figure of 73° F. laid down by the Petroleum Act gives rise to anomalies when applied to lacquers, and specific legislation is considered desirable. S. S. WOOLF.

Flow relationships in nitrocellulose dispersions. D. R. WIGGAM (J. Rheology, 1929, 1, 48—69).—Viscosity measurements have been made at 25° on solutions of nitrocellulose in a number of solvents. The laws of viscous flow are not followed, but the results can be represented by Williamson's equation modified to take account of the radius of the capillary tube. C. W. GIBBY.

Purification of wood rosin. H. E. KAISER and R. S. HANCOCK (Ind. Eng. Chem., 1930, 22, 446—448).—Solvent-extracted wood rosin contains two colouring materials: (A) which produces the ruby-red coloration, and (B) which readily reacts with alkali to form coloured substances. Vacuum distillation improves the colour by the removal of A. By dissolution of the rosin in petrol and the addition of a second immiscible substance after adequate agitation, B can be removed. The secondary substance added may be a substituted ether, a phenol, aniline, furfuryl alcohol, ethylene chlorohydrin, or furfuraldehyde. A successful simple process of purification has been based upon the use of furfuraldehyde, since this liquid is practically immiscible with petrol. The rosin is dissolved in petrol, warmed, and agitated with furfuraldehyde. The two layers of liquid are separated after cooling, and the solvents recovered by evaporation. The undesirable colouring matter is concentrated in 20% of the rosin recovered. The process can also be modified to produce abietic esters of good colour. J. O. CUTTER.

Resins. V. Adulteration of sandarac. E. STOCK (Farben-Ztg., 1930, 35, 1459—1460; cf. B., 1930, 249).—

The presence of pistachio and pine resins as adulterants of sandarac may be detected by decrease in acid value, increase in saponif. value, and variation of the characteristic "capillary analysis" phenomena observed.

S. S. WOOLF.

Resins and resin substances. VII. Tolu balsam. A. ROLLETT [with O. SCHNEIDER] (Monatsh., 1930, 55, 151—157).—The residue from the ether-soluble extract of the resin is extracted with 20% acetone, whereby cinnamic acid is obtained. Further extraction of the residue from this with 20% alcohol affords a further amount of cinnamic acid, and the remainder of the product consists of impure tolueresinophenol (cf. Oberlaender, A., 1895, i, 188). Distillation of this gives an oil resembling clove oil. Reduction with zinc dust and acetic acid furnishes a product with an odour of eugenol, whilst oxidation with chromic acid affords vanillin. Hydrolysis with 5% potassium hydroxide solution yields cinnamic acid and a substance corresponding with a polymeric coniferyl alcohol. Successive extraction of the ether-insoluble portion of the resin with 20 and 50% acetone affords benzoic acid and vanillin, respectively. H. BURTON.

Acid values of dark-coloured resins. H. H. COBURN (Ind. Eng. Chem. [Anal.], 1930, 2, 181).—Determination of the acid value of a resin by titration with phenolphthalein is difficult if the resin is highly coloured. Good results are, however, given by the use of two layers, viz., a benzene-alcohol mixture and an aqueous layer saturated with sodium chloride. Excess of alkali is added with vigorous shaking and the excess titrated back. C. IRWIN.

Plastometer. GREGORY and others.—See I. **Formaldehyde condensations with aromatic compounds.** MORGAN; also MEGSON and DRUMMOND.—See III. **Pilchard oil.** GARDNER.—See XII. **White fillers for rubber.** DAWSON.—See XIV.

PATENTS.

Preparation of oil paints. A. HERMSDORF (G.P. 461,383, 11.4.26).—The oil used is prepared by heating linseed, poppy-seed, hempseed, tung, soya-bean, or whale oil with sulphur. When linseed oil is so treated 5—7% of a water-soluble sulpho-fatty acid is obtained which can be used for the preparation of fat-splitting compounds. A. R. POWELL.

Preparation of durable paints. CHEM. FABR. DR. J. WIERNIK & Co. A.-G., and BAKELITE GES.M.B.H., Assees. of J. SCHEIBER (B.P. 304,761, 27.12.28. Ger., 26.1.28. Addn. to B.P. 299,024; B., 1930, 469).—A deoxidant is added to the paint itself, in a primarily inert form, but which is adapted to be readily decomposed by the substances (acid degradation products, water, etc.) formed during the completion of the film, so that whereas the initial stages of drying are unaffected, the subsequent degradation is retarded. Suitable deoxidants are normal salts of lead, zinc, barium, magnesium, etc. with phenols or phenolic ethers, or amines combined with suitable metallic salts, e.g., zinc chloride, to form readily hydrolysable double salts. [Stat. ref.] S. S. WOOLF.

[Paint for] protection of iron surfaces against corrosion. I. G. FARBENIND. A.-G. (B.P. 305,218,

30.1.29. Ger., 2.2.28).—An anti-rust paint comprises a cellulose ester of a fatty, naphthenic, or resin acid of high mol. wt., a heavy metal salt of the same or similar acid, a cellulose ether insoluble in water, a drying oil, a colouring matter, and a softening or filling agent, *e.g.*, 20 pts. of cellulose trilaurate, 50 pts. of benzene, 50 pts. of xylene, 5 pts. of copper naphthenate, oleate, or similar compound, with or without 15 pts. of benzylcellulose.

A. R. POWELL.

Manufacture of coating compositions. ATLAS POWDER CO., Assees. of D. CARNEGIE, JUN. (B.P. 297,681, 24.9.28. U.S., 23.9.27).—A drying or semi-drying oil, *e.g.*, raw linseed oil, is oxidised without substantial polymerisation, *e.g.*, by blowing air therethrough below 250°, until it attains a viscosity at which a solid steel ball (diam., $\frac{1}{4}$ in.) requires 20–80 sec. (preferably 60–75 sec.) to fall under gravity a distance of 12 in. through the oil at 25–30°. Then 3–4 pts. of the oxidised oil are mixed with 1 pt. of a cellulose ester, *e.g.*, cellulose nitrate, to give a composition suitable for use in imitation leather manufacture.

S. S. WOOLF.

Manufacture of coating preparations and solutions of their constituents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,824, 15.11.28).—Cellulose derivatives soluble in organic solvents are dissolved in a solvent consisting of or containing an aliphatic homologue of 1:4-dioxan, and, if desired, other organic solvents, diluents, resins, pigments, dyes, etc.

S. S. WOOLF.

Graining composition. E. I. DU PONT DE NEMOURS & Co. (B.P. 315,214, 6.5.29. U.S., 9.7.28).—Dispersions of pigment in ethylene glycol and water with a small amount of a gum soluble in both solvents, *e.g.*, gum arabic, are claimed.

S. S. WOOLF.

Production of pictures in one or more colours on metallic bases. G. MANETTI, P. BENAGLIA, and M. LUCHSINGER (B.P. 303,419, 2.1.29. It., 2.1.28).—Metallic, gilded, or silvered surfaces etc. are covered with a layer of varnish (comprising a cellulose derivative) on which colours are subsequently printed. The metallic surface may be given a design in relief either before or after the colour-printing.

S. S. WOOLF.

Manufacture of a stable red lead paste. CHEMISCHES LABORATORIUM F. ANSTRICHSTOFFE GES.M.B.H. (G.P. 451,496, 10.1.25).—Red lead is ground in a mixture of linseed oil or linseed oil varnish with kieselguhr or a neutral or basic aluminium salt of a resin acid or a fatty acid.

A. R. POWELL.

Manufacture of condensation products of urea [carbamide] or derivatives thereof and formaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,673, 6.10.28).—Water-insoluble formaldehyde-urea resins, obtained by condensation in anhydrous mono- or poly-hydric alcohols (C_4 and higher) as solvents, are precipitated by the addition of low-boiling aliphatic, aromatic, or hydroaromatic hydrocarbons or ethers (excluding acetals) in a finely-pulverulent state which facilitates removal of solvent and excess formaldehyde by washing. *E.g.*, urea (2 pts.) in commercial alcohol (1.6 pts.) is treated with concentrated hydrochloric acid (0.024 pt.) and added to anhydrous formaldehyde (2.3 pts.) dissolved in amyl alcohol (9 pts.) at 110°;

the product is neutralised with trisodium phosphate, and the resin is precipitated by light petroleum (5–10 times the weight of mixture), washed with ether, and dried at 50°. This product, dissolved in *n*-propyl and ethyl alcohols, gives a water-resistant lacquer.

C. HOLLINS.

Manufacture of phenol-formaldehyde condensation products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,158, 28.12.28).—Less brittle and more easily workable resins are obtained by incorporating, during the condensation of phenol and formaldehyde, a polyalkylene glycol aryl ether, made, *e.g.*, by the action of ethylene oxide (3 mols.) on technical cresol.

C. HOLLINS.

Phenolic resin moulding mixtures. H. WADE. From BAKELITE CORP. (B.P. 326,840, 21.12.28).—Mixtures of a potentially-reactive phenolic resin and a cellulose ester, *e.g.*, cellulose acetate, the ester having a fibrous structure and being present in sufficient proportion to impart moulding capabilities to the mass, with the addition, if desired, of plasticisers, are claimed. A preferred mixture comprises approximately equal proportions of the two constituents.

S. S. WOOLF.

Manufacture of synthetic resins. H. WADE. From BAKELITE CORP. (B.P. 326,884, 24.12.28).—Cresol is condensed with furfuraldehyde in the presence of a base, *e.g.*, lime, until a fusible resin is formed substantially in the absence of free phenol; more lime and, if desired, a fusible resin, *e.g.*, a non-reactive phenol-methylene resin, are added and the reaction is continued.

S. S. WOOLF.

["Alkyd"] resinous compositions. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. H. KIENLE (B.P. 299,424, 19.10.28. U.S., 26.10.27).—Polybasic acids (phthalic acid) are heated with monohydric alcohols (amyl or butyl alcohol) to a reaction temperature until combination occurs, whereupon polyhydric alcohols (glycerin) are added and the mixture is heated to resinification. Monobasic acids, *e.g.*, oleic acid, may replace polybasic acids in part. The "alkyd" resins produced have high flexibility and water-resistance, and are suitable for coating wires and sheet metal.

S. S. WOOLF.

Manufacture of artificial masses [resins from polyhydric alcohols and dicarboxy-ethers or -thioethers, and their application]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,722, 10.12.28, 18.7., 19.7., 9.8., and 12.8.29).—A polyhydric alcohol or alcohol mixture, *e.g.*, glycol, glycerol, erythritol, pentaerythritol, mannitol, hydroxyalkyl esters and ethers, etc., are heated with dicarboxy-ethers or -thioethers or their anhydrides, *e.g.*, diglycollic acid, carboxymethyl ether of salicylic acid, methyl diglycollic acid, "thiodiglycollic acid" (sulphidodiacetic acid), etc.; the products may be hardened by further heating. The condensation may be modified by the addition of other polybasic acids, fatty acids or esters (including drying oils and their acids), fillers, colouring matters, resins, softening agents, diluents, or solvents. A glycerol partly esterified with fatty acids (*e.g.*, linoleic) may be used as the polyhydric alcohol. The products serve all the purposes of "Glyptal" resins, and also form substitutes for glass, horn, or shellac (for binding together

mica plates); mixed with hydraulic cement, lime, etc. they harden rapidly at 40—100°. C. HOLLINS.

Preparation of resinous condensation products.

A. H. V. DURR, ASSR. TO COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ETABL. KUHLMANN (U.S.P. 1,739,446—8, 10.12.29. Appl., 2.1.29. Fr., 31.12.27).—Mixed phthalic and resinic esters of glycerol are made (A) by heating together glycerin, phthalic anhydride, and colophony, (B) with subsequent incorporation of drying oils, or (C) by heating together a glyceryl phthalate and a glyceryl resinate.

C. HOLLINS.

* **Manufacture of [resinous] condensation products of carbazole compounds and olefines.** A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 327,746, 11.12.28).—Mono- or poly-alkylated carbazoles or higher condensation products are obtained by heating carbazoles with olefines at 100—200°, in presence of aluminium chloride etc., fuller's earth, tonsil, or frankonite, preferably in presence of an inert solvent (light petroleum, paraffin oil, cyclohexane, decahydronaphthalene, etc.). Carbazole with propylene gives a soft resin, b.p. 210—240°/1 mm., probably diisopropylcarbazole, with cyclohexene a hard resin, b.p. 270—340°/1 mm.; *N*-ethylcarbazole and propylene yield a soft resin, b.p. 200—230°/mm.

C. HOLLINS.

Manufacture of products having the properties of resins and waxes. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 327,417, 29.9.28).—Bleached montan wax, beeswax, carnauba wax, or their conversion products, containing carboxylic acid groups, are esterified with hydroxylated high-molecular resoles or resins (free from aromatic or hydroaromatic sulphur compounds), e.g., the product from formaldehyde and *o*-cresol, or aldol resin. Part of the acidity may be neutralised by addition, e.g., of lime before reaction. The resins may be hardened, and are then thermoplastic, although they do not melt. If formaldehyde or trioxymethylene be added during esterification, hard, non-thermoplastic masses result.

C. HOLLINS.

Material containing carbon, iron, and iron oxide (B.P. 308,351). **Products from tars and pitches** (B.P. 327,797).—See II.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spiral [molecular] model for rubber. F. KIRCHHOFF (Kautschuk, 1930, 6, 31—33).—By means of quotations from earlier publications (cf. B., 1922, 335 A) the author demonstrates that he had anticipated the essential features of the spiral formula recently formulated for rubber by Fikentscher and Mark (B., 1930, 249).

D. F. TWISS.

Solvation of substances of high mol. wt., particularly rubber. P. STAMBERGER and C. M. BLOW (Kautschuk, 1930, 6, 22—27; cf. B., 1929, 692).—From measurements of the swelling pressure of rubber in toluene and of the lowering of the vapour pressure of this solvent by rubber it is probable that rubber does not consist of a mixture of molecules in various stages of polymerisation and that the mastication of rubber does not induce depolymerisation as is commonly assumed. The vapour-pressure lowering is not influenced by the

viscosity of the solution, so that the latter is not an index to the molecular magnitude of the dissolved rubber; this conclusion also follows from measurements of the viscosity of solutions of rubber from mixtures with carbon black. The condition of substances of high mol. wt. in solution is discussed briefly.

D. F. TWISS.

Adsorption-chemical studies on rubber fillers and rubber mixings. I. G. FROMANDI (Kautschuk, 1930, 6, 27—30).—Various types of amorphous carbon used as ingredients for rubber mixings exhibit different degrees of adsorptive power for acetic acid from aqueous solution. The adsorptive power is conveniently estimated by measuring the final acidity of the filtered solution. The relation of this adsorptive power to the reinforcing qualities of the powder is under investigation.

D. F. TWISS.

Comparison of white fillers for rubber, with special reference to pigmentation. T. R. DAWSON (India-rubber J., 1930, 79, 315—317).—Vulcanised mixtures of rubber with various white pigments and fillers, e.g., lithopone, titanium white, zinc oxide, zinc sulphide, whiting, barium sulphate, magnesium carbonate, were examined as to their mechanical properties and colour. In reinforcing effect zinc oxide and magnesium carbonate were exceptionally good. Zinc sulphide had the most intense pigmenting effect, but imparted a distinctly yellowish tint; titanium white was second, but amorphous titanium dioxide was deficient in pigmentation power. The lithopone samples discoloured considerably on exposure to light; the zinc oxide samples appeared to be the most resistant to "yellowing" in storage. A bibliography of papers dealing with "whiteness" in pigments, paints, and rubber is appended.

D. F. TWISS.

Vulcanisation with sulphur chloride. E. WURM (Kautschuk, 1930, 6, 33—36).—Some advantages of the cold-vulcanisation process for manufacturing purposes are indicated, and consideration is given to various factors, e.g., choice of compounding ingredients and pigments, dusting powders, calendering, conditions of vulcanisation, etc., which are favourable to satisfactory results.

D. F. TWISS.

Ageing of rubber and its retardation by the surface application of antioxidants. Diffusion process. C. MOUREU, C. DUFRAISSE, and P. LOTTE (Ind. Eng. Chem., 1930, 22, 549—551).—The changes caused in organic substances by oxygen are of two distinct types: one of ordinary chemical nature and the other catalytic. In order to accelerate the natural ageing of rubber, it is not sufficient to expedite the absorption of oxygen. An excess of oxygen may even alter the course of the reaction, as it does with acrolein, and the oxygen-ageing test for rubber may lead to false conclusions. The Geer heat-ageing test does not give a faithful representation of natural ageing, but is useful for comparative purposes, especially for articles to be used at elevated temperatures. For this purpose comparison of the behaviour at 90° until the control samples show considerable deterioration is useful; care is necessary, however, to prevent contamination of the control samples by vapours or dust from antioxidants in other

samples, but circulation of fresh air is unessential. Exposure to ultra-violet light is only of use where objects are to be used under similar conditions; ultra-violet light leads to formation of ozone, and deterioration by ozone is a different phenomenon from depreciation by oxygen, being influenced by different factors. The presence of ozone should be avoided in comparative experiments designed to determine the efficiency of an antioxygen. It is often sufficient to apply an antioxygen to the surface of the rubber, in solid, liquid, or vapour form. If a solution be used, the nature of the solvent is of importance, probably by influencing the degree of penetration of the antioxygen; ether is a suitable solvent for antioxygens of the quinol type. The "diffusion process," as this method of surface treatment is termed, has several advantages, including that of renewability. On scientific grounds the term "antioxygen" is preferred to "antioxidant," the latter designation being likely to lead to misconception as to the mode of action and limitations of such preservatives against ageing.

D. F. TWISS.

Determination of total sulphur in rubber. J. G. MACKAY (J.S.C.I., 1930, 49, 233–241 r).—An account is given of an attempt to devise a method of determining total sulphur, of greater rapidity than but still comparable in accuracy with the Carius method. A series of vulcanised rubber samples were prepared containing varying proportions of sulphur and involving the commoner compounding ingredients; these were standardised by the Carius method. The analytical figures obtained in this way were lower than the "theoretical" values by more than the allowable amount in mixings containing carbon black, china clay, silica, French chalk, antimony sulphide, and compounds of iron and calcium. Suggested explanations of these discrepancies are given. The method of oxidation recently described by Kahane (B., 1927, 532), involving the use of fuming nitric and perchloric acids, was found to be inaccurate, but when several modifications were introduced satisfactory results were obtained. It is shown that the sulphuric acid produced by this method of oxidation can be accurately determined volumetrically as benzidine sulphate. A procedure for the determination of total sulphur was thus developed, and when applied to the standardised mixings yielded results in good agreement with those obtained by the Carius method. The new procedure gave better figures for the mixings containing calcium, antimony, and iron compounds than did the older method, and a determination in duplicate requires less than 4 hrs.

PATENTS.

Purification of aqueous dispersions [e.g., rubber latex]. ANODE RUBBER CO., LTD., Assees. of E. B. NEWTON (B.P. 319,344, 15.3.29. U.S., 22.9.28).—The greater part of the dissolved gases or volatile substances can be removed from latex or similar dispersions by injecting a vigorous current of inert gas into the heated liquid. In order to prevent coagulation on account of the loss of ammonia or other volatile base a non-volatile alkali or alkaline substance, e.g., potassium hydroxide or sodium phosphate, is added. A preservative, e.g.,

an alkali phenoxide, may also be introduced before, during, or after the heating or blowing operation.

D. F. TWISS.

Manufacture of rubber composition [and carbon black therefor]. THERMATOMIC CARBON CO., Assees. of E. B. SPEAR and R. L. MOORE (B.P. 307,743, 20.2.29. U.S., 12.3.28).—An improved thermatomic black of low stiffening qualities for rubber, but possessing the black colour and rubber-strengthening qualities associated with carbon black made by the channel process, and imparting a greater ultimate elongation than that given either by commercial carbon black or ordinary thermatomic black, is produced by decomposing hydrocarbon gases, diluted with inert gases, in a heated retort. Normally natural petroleum gas is used, and the inert gases consist of the gaseous products of decomposition, mainly hydrogen, resulting from the process after removal of the entrained carbon.

D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 327,451, 5.1.29).—Compounding ingredients such as gas black, clay, silica, or whiting, which in sufficient amounts normally cause coagulation of latex, are added to aqueous dispersions of rubber or similar material in the presence of a stabilising agent, e.g., an ammonium soap, which arrests their coagulative effect. Further addition of similar ingredients or others, e.g., zinc oxide, with stirring then leads to the formation of an incoherent mass, which dries to a crumb-like condition. The products can be consolidated by pressure or by milling for a short time. It is possible in this way to obtain uniform workable mixtures of rubber with high proportions of carbon black.

D. F. TWISS.

Manufacture of articles of rubber or similar material. DUNLOP RUBBER CO., LTD., R. F. MCKAY, and W. G. THORPE (B.P. 328,015, 16.1.29).—A former is dipped into latex, which preferably has been concentrated and/or compounded, and the uncoagulated deposit thereon is submitted to a spray or gaseous or vaporous current containing a coagulant, such as acetic acid, and/or to a spray containing a setting agent. Deposits of considerable thickness can thus be set; thicker articles can be obtained by superposing several such deposits.

D. F. TWISS.

Manufacture of artificial rubber. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 304,207, 15.1.29).—Artificial rubber produced by the polymerisation of aqueous dispersions of α -butadiene or its analogues is separated from the resulting latex-like emulsion by cooling below 0° and removing the coagulum. The emulsifying agent is thereby recovered in the aqueous layer. Emulsifying agent retained in the coagulum may also be recovered, e.g., by washing, but, being strongly adsorbed by unchanged diolefine, it is advisable to remove this previously, e.g., by evaporation.

D. F. TWISS.

Manufacture of age-resisting rubber compounds. IMPERIAL CHEM. INDUSTRIES, LTD., S. COFFEY, and W. J. S. NAUNTON (B.P. 328,115, 18.4.29).—Nitrogenous organic compounds capable of dissociating into free radicals containing bivalent nitrogen (excluding aryl-

naphthylnitrosoamines) are incorporated in rubber mixes before vulcanisation. Examples are tetraphenylhydrazine and diphenyltriphenylmethylamine.

C. HOLLINS.

Coating of metal articles with rubber or similar materials. C. MACINTOSH & Co., LTD., H. C. YOUNG, and C. HEMM (B.P. 327,452, 5.1.29).—Objects with closely-grooved, corrugated, or ribbed surfaces, or with deep re-entrant angles are given coatings of substantially uniform thickness by first subjecting them to electrophoretic deposition, simultaneously or subsequently conditioning the deposits to impart a high specific resistance throughout, and then effecting further electrophoretic deposition in any known manner. The initial coating of high resistance may be produced, for example, by electrophoretic treatment for a few seconds with an aqueous dispersion of high ammonia content or by partial drying of an electrophoretic deposit produced in the ordinary manner.

D. F. TWISS.

Adhesive material [from rubber]. W. C. GEER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,744,880, 28.1.30. Appl., 9.2.25).—The balata-like isomeride obtained from rubber by the action of sulphuric or sulphonic acids is mixed with an anti-ager (acetaldehyde- α -naphthylamine, benzidine, 1:8-naphthylenediamine, 4:4'-tetramethyldiaminothiocarbanilide) and tung oil, and used as an adhesive, especially for rubber-metal joins.

C. HOLLINS.

Heterocyclic bases (B.P. 310,534). **Vulcanisation accelerators** (U.S.P. 1,737,384 and 1,737,391).—See III. **Artificial threads etc.** (B.P. 327,164).—See V.

XV.—LEATHER; GLUE.

Biochemistry of soaking and liming [of animal skins]. V. Effect of various acids on the diffusion of coagulable proteins from animal skin. E. R. THEIS (J. Amer. Leather Chem. Assoc., 1930, 25, 48–56; cf. B., 1930, 252).—Pieces of skin were treated with solutions of sodium chloride of different concentrations respectively, solutions of hydrochloric acid, and solutions of different organic acids of different concentrations. After 48 hrs. treatment the solutions were analysed for coagulable and non-coagulable proteins. As the concentration of the sodium chloride was increased, the total dissolved protein decreased. Maximum coagulable protein was dissolved by the 10–15% solutions of sodium chloride. At high concentrations of hydrochloric acid (0.1–1.0N), practically no coagulable protein was dissolved, but as the p_H value increased both the amount of total dissolved and of coagulable nitrogen increased, thus showing that the coagulable protein in skin was coagulated by acid. Pieces of skin were treated for 48 hrs. with 0.1N-solutions of hydrochloric, nitric, sulphuric, phosphoric, formic, acetic, lactic, citric, oxalic, and mono-, di-, and tri-chloroacetic acids, respectively, then for 48 hrs. with a solution of sodium hydroxide equivalent to the acid present in the pieces, and finally for 48 hrs. with a 15% solution of sodium chloride. The amounts of total dissolved and coagulable nitrogen were determined in the acid soak-liquors, the

neutralisation liquors, and in the sodium chloride solutions after use. The coagulable protein in the skin was rendered insoluble by all these acids, but this coagulation effect was reversed by the sodium hydroxide except in the case of hydrochloric, phosphoric, formic, mono- and di-chloroacetic acids. The time of bating will be increased for skins which have been soaked in solutions of these coagulating acids.

D. WOODROFFE.

Natural fats of goatskins and their relation to the formation of fatty spue in chrome[-tanned] leather. R. F. INNES (J. Soc. Leather Trades' Chem., 1929, 13, 375–382).—A purple compound of chromium and fatty acid associated with fatty spues on finished chrome-tanned leathers was observed on chrome-tanned skins which had not been fat-liquored. The fatty spue associated with this on finished leathers was shown to be chiefly free fatty acids (m.p. 50–51°). The purplish compound could be produced artificially only on skins containing free oleic acid or calcium oleate. No purple stain could be produced even on greasy skins containing no free fatty acid. In imported goatskins 20% of the total fat was shown to be free fatty acids, from which lime soaps are formed during manufacture, and these are decomposed only in the two-bath chrome-tanning process. Degreased, bated skins were impregnated with oleic acid, olein, and stearic acid, respectively, and then chrome-tanned, fat-liquored, and finished; pink stains were observed on the "blue" skins prepared from the pelt which had been treated with oleic acid and spue was observed on the finished leather, which contained the stearic acid. Lipase, produced by micro-organisms in the presence of moisture and protein, has been found in cured goatskins. It is suggested that if putrefactive organisms develop before or during the curing of the skins, or if the cured skins become damp, lipase is formed, the natural fats are hydrolysed, and white spues result from the stearic acid thus liberated. The remedy is more efficient curing and preservation of the cured skins in a dry store.

D. WOODROFFE.

Conservation of hides and skins. M. C. LAMB (J. Soc. Leather Trades' Chem., 1930, 14, 207–210).—Sheepskins are often damaged by burrs, i.e., the fruits of certain grasses, e.g., *Tragus racemosus*, All., *Medicago*, App., *Triumfetta annua*, L., and *Xanthium spinosum*, L., which collect in the wool. Short hairs of vegetable origin have been found on unhaird skins. The grain of South African sheepskins is often perforated by "spear grass" (*Hepteropogon contortus*, L.) and "steek grass" (*Aristida barbicollis* and *A. congesta*). Uruguayan sheepskins are often damaged by the awns of *Stipa charruana*, Arech.

D. WOODROFFE.

Analysis of artificial pancreatic bates. A. ULČEK (J. Soc. Leather Trades' Chem., 1930, 14, 189–202; cf. B., 1927, 662, 758).—Kubelka and Wagner's criticisms (B., 1929, 755, 1025) are answered. The author's method evaluates the increase in acidity in the presence of formaldehyde, which has been shown to be proportional to the dissolved nitrogenous matter. The presence of erepsin does not affect the result. A certain minimum concentration of casein solution is necessary

to differentiate bates of different strengths. A lower temperature than 40° can be used. An apparent increased activity is obtained in presence of certain alkaline reagents due to the incomplete precipitation of the casein. The Kubelka-Wagner method of determining the enzymic strength of commercial bates is unsatisfactory and is not in accord with modern conceptions of the chemistry of proteins and enzymes. Certain neutral salts, *e.g.*, ammonium, oppose the absorption of the enzyme by the wood-meal or sawdust. Distinction must be made between "enzymic capacity" (the activity of the total amount of enzyme in the sample) and "enzymic intensity" (the activity due to the dissolved enzyme when the sample is mixed with water). The former can be determined by digesting the casein solution with water and the enzyme without previously making an extract, filtering the product, and using an aliquot portion for a nitrogen determination; another portion is mixed with formaldehyde and the acidity titrated.

D. WOODROFFE.

Determination of p_H in tan liquors. L. GOLDMAN (J. Soc. Leather Trades' Chem., 1930, 14, 211—213).—Inconsistent results with the hydrogen electrode in the determination of p_H in tan liquors are probably due to the reduction of certain constituents of the tan liquor by hydrogen in the presence of the platinum-black catalyst. To minimise this, the electrode is placed above the liquor and hydrogen is allowed to pass for $\frac{1}{2}$ hr., after which the electrode is immersed in tan liquor and an immediate reading is taken; the process is repeated until a constant *P.D.* is reached. The electrode was prepared by coating a platinum wire with palladium-black from a 1% solution of palladium chloride; it is then washed, placed as the negative pole in a 5% solution of sulphuric acid, and a current of 20 milliamp. passed until hydrogen bubbled freely from it. Palladium is used as it has greater absorptive power for hydrogen than platinum. The method is not applicable to sulphited extracts. A freshly palladised electrode should be used for each tan liquor.

D. WOODROFFE.

Functions of oils and fats in currying, fat-liquoring, oiling-off, and chamoising [of leather]. III. Report of a Committee of the Society of Leather Trades' Chemists. D. BURTON and G. F. ROBERTSHAW (J. Soc. Leather Trades' Chem., 1929, 13, 383—397).—Light leathers are fat-liquored to give them the desired softness, pliability, and increased tensile strength. The kind and amount of oil which gives the right pliability, minimum stretch, and maximum area should be determined, also the effect of the stability of the fat-liquor emulsion and the drying of the leather. Oiling-off prevents oxidation and reduces evaporation from the grain side to a minimum. The use of cod oil for oiling-off is discussed. Pieces of leather at different stages of tanning were divided into two portions, one of which was oiled-off with cod oil and dried and the other dried out without treatment. It was shown that a good colour could be obtained without oiling, and that it depended on the rate of drying and on the tannin content of the water-soluble matter. The chamoising properties of oils depend on the relative tanning properties of the fatty acids con-

tained in them. Greasiness in finished leathers may be caused by natural or added grease, mouldiness by moulds in the oils used, cracky grain by lack of grease or low moisture content, and springiness by excessive currying. Brittle fibres in belting leather have been produced by fatty acids. Some white spues have been traced to natural skin fat, fatty acids, stearins, or hydroxy-acids, respectively, of high m.p. The literature and previous work in the various processes are reviewed.

D. WOODROFFE.

Wearing quality and other properties of vegetable-tanned and of chrome-retanned sole leather. R. W. FREY and I. D. CLARKE (Tech. U.S. Dept. Agric. Tech. Bull. No. 169, 1930, 17 pp.).—Six hides were cut in two, one half of each was vegetable-tanned for sole leather, the others were tanned with a one-bath chrome-tanning liquor, neutralised, washed, and retanned with a mixture of chestnut and sulphited quebracho extracts; one pair was then retanned lightly, another to a slightly greater degree, and the third pair fully retanned. From 100 lb. of green-salted cured hide were obtained 66 lb. of vegetable-tanned leather (showing a 4% increase in area and having d 1.044) and 32—51 lb. of chrome-retanned leather (6% decrease in area, d 0.70—0.84), respectively. A lower tensile strength was shown by the chrome-retanned than by the vegetable-tanned leather, the difference increasing as the degree of retannage increases. The stretch of the vegetable-tanned leather was 73% of that of the chrome-retanned leather at the breaking load. A larger volume of "voids" was observed in the chrome-retanned than in the vegetable-tanned leather, and 60—80% greater wear was obtained with the former, although it was thinner than the vegetable-tanned leather. The wear-resistance of the chrome-retanned leather decreased as the degree of retannage increased. No relationship was shown between the density or apparent density and the wear-resistance of the leathers. The chrome-retanned leather was quickly penetrated by water, was slippery in wet weather, and was not sufficiently solid to protect the foot against uneven surfaces in wear.

D. WOODROFFE.

Relationship between physical properties and chemical constitution. VI. **Waterproofness of different sole leathers.** D. WOODROFFE (J. Soc. Leather Trades' Chem., 1929, 13, 631—636; cf. B., 1926, 989).—The time taken to force water through pieces of sole leather was noted for different leathers before and after removal of the grain, but no appreciable difference was noted, thus contradicting Van der Waerden's results (B., 1928, 828). The water-soluble matter was determined in these leathers, and it is shown that there is no connexion between the water penetrability and the content of water-soluble matter. Four French leathers were impenetrable to water from the grain side, but they absorbed water readily by immersion in it. It is shown that there is no relationship between the water absorption and the water permeability of a leather, thus disagreeing with Bradley, McKay, and Worswick's suggestion (B., 1929, 446) to compare water permeabilities by determining the "diffusion constants." The results obtained afford further evidence of the difficulty of comparing simple

physical properties of leathers owing to other differences, e.g., the compacting of the fibres in French leathers.

D. WOODROFFE.

Analysis of vegetable-tanned leather. Method proposed by a Committee of the Society of Leather Trades' Chemists. P. CHAMBARD (J. Soc. Leather Trades' Chem., 1929, 13, 355—365).—The sample is cut into pieces of maximum length (2 cm.) and thickness (0.5 mm.). Moisture is determined at 110°, then total ash, and soluble ash by calcining the dried residues from the water-soluble matter determinations. The insoluble ash is the difference between this and the total ash. The grease is extracted with light petroleum (b.p. 40—65°) and the residue heated at 95—100° to constant weight. Water-soluble matter is determined either by the French method (cold) or by the Procter extractor at 45°. Glucose and hide substance are determined by the usual methods. D. WOODROFFE.

Analysis of [vegetable-tanned] leather. A. T. HOUGH (J. Soc. Leather Trades' Chem., 1929, 13, 637—639).—Results of analyses of various leathers show that a larger figure is sometimes obtained for the soluble ash, as determined by the proposed official method (cf. preceding abstract), than for the total ash. The insoluble ash should be determined by ashing the residual leather after removal of the grease and water-soluble matter. The soluble ash should be sulphated and the figure obtained used in calculating the combined tannin. The grease extract should be ashed and weighed, since it sometimes contains metallic soaps. D. WOODROFFE.

Effect of acidity on the dyeing of leather. D. WOODROFFE and S. J. HILL (J. Soc. Leather Trades' Chem., 1930, 14, 203—206).—Pieces of sumac-tanned skiver were dyed with solutions of a number of acid dyes at different p_H values. Maximum depth of shade was obtained in most cases at p_H 2.0. Sulphuric acid is more effective than acetic acid in producing this p_H , and hence for obtaining fuller shades in dyeing leather with acid dyes. A full shade was obtained with a few dyes at higher p_H values. Variations in the shade of leather dyed with mixed dyes may be due to variations in the acidity of different portions of the skins arising from variations in texture or from incomplete neutralisation after chrome-tanning. D. WOODROFFE.

PATENTS.

Preparation of leather. A. EHRENREICH (U.S.P. 1,758,564, 13.5.30. Appl., 31.1.28. Belg., 23.9.27).—See B.P. 299,599; B., 1929, 30.

Production of structural forms of small dimensions of colloidal material, especially of glue or gelatin. A. MAREK (B.P. 328,623, 31.12.28).

Coating composition (B.P. 297,681).—See XIII. **Adhesive from rubber** (U.S.P. 1,744,880).—See XIV.

XVI.—AGRICULTURE.

Physical properties of soil. V. Hysteresis effect in capillary properties, and modes of moisture distribution associated therewith. W. B. HAINES (J. Agric. Sci., 1930, 20, 97—116; cf. B., 1927, 588).—A discussion of the physics of the moisture

distribution in an ideal soil followed by a description of experiments with bronze balls and paraffin oil to determine suction values. Certain differences of opinion with R. A. Fisher are resolved (*ibid.*, 1928, 18, 406).

E. HOLMES.

Laterite and lateritic soil in Sierra Leone. II. F. J. MARTIN and H. C. DOYNE (J. Agric. Sci., 1930, 20, 135—143; cf. B., 1928, 28).—Laterites, lateritic soils, and non-lateritic soils are defined as soils having a molecular silica/alumina ratio of less than 1.33, between 1.33 and 2, and above 2, respectively. The detrital soils of Sierra Leone contain a higher ratio of alumina to silica than the residual soils. They are all acid in reaction, have a definite lime requirement, and mineral constituents, particularly potash and exchangeable calcium, are low. Such soils readily remove soluble phosphorus from solution. E. HOLMES.

Purdue technique for taking and mounting monolithic soil profile samples. T. M. BUSHNELL (Soil Sci., 1930, 29, 395—399).—Samples are obtained from the profile face by glueing to it strips of cloth etc. and subsequently breaking away the strips with adhering soil. A. G. POLLARD.

Approximate size of soil particles at which the heat of wetting is manifested. L. C. KAPP (Soil Sci., 1930, 29, 401—412).—Heat of wetting is exhibited by soil particles of mean diam. up to 0.005—0.01 mm. The size limit of powdered mineral particles which show heat of wetting is lower than that of soils. Soil organic matter may affect the heat of wetting, but to no great extent. Soil particles of mean diam. <0.00005 mm., the heat of wetting property of which was lost after ignition at 830°, were subsequently found to contain approx. 90% of particles of mean diam. 0.02 mm. Regrinding after ignition partially restored the heat of wetting phenomenon. A. G. POLLARD.

Equipment and procedure for obtaining the displaced soil solution. J. P. CONRAD, E. L. PROBSTING, and L. R. MCKINNON (Soil Sci., 1930, 29, 323—329).—The soil sample is packed in a specially-designed metal cylinder, and covered with water. Air pressure is applied at the water surface. A. G. POLLARD.

Translocation of calcium in soils as measured by electrodialysis and plant growth. B. D. WILSON (Soil Sci., 1930, 29, 331—337).—Soils, limed at the surface and subsequently leached with water for a lengthy period, were examined. The fixation of calcium by the lower soil layers, from the drainage water passing through them, was very small but sufficient to influence the growth of clover for a brief period. A. G. POLLARD.

Rôle of humus in the absorption complex [in soils]. L. KOTZMANN (Landw. Forsch. Ungarn., 1929, 12, 537; Bied. Zentr., 1930, 59, 200—202).—Recorded equivalent weights of humic acid vary with the methods of examination adopted. Humus "fixes" bases both by chemical combination and by physical adsorption. The latter process is governed by the concentration of bases in the solution with which the humus is in contact. By repeated evaporation of ammoniacal humus extracts free ammonia is eliminated and residual ammonia is

determined as being chemically combined. Hissink's baryta method for examining humus does not eliminate adsorption. The determination of the ($T-S$) value by di Gléria's method indicates combined ammonia only. By repetition of this method with a soil treated with hydrochloric acid (sufficiently dilute to avoid decomposition of the humus) the chemical value T is obtained. Only a portion of the total humus content of soils exhibits adsorptive properties, and this portion approximates closely to that determined by the Grandeau-Hilgard method. A. G. POLLARD.

Inaccuracy of the quinhydrone electrode in [determinations of p_H of] many Kentucky soils. P. E. KARRAKER (J. Amer. Soc. Agron., 1930, 22, 171—180).—Higher values (max. p_H 1.5) than those given by the hydrogen electrode or colorimetric methods were obtained. Soils giving incorrect values in suspensions afforded filtrates giving correct values; the inaccuracy is attributed to the presence of manganese. The colorimetric method is preferred for routine determinations. CHEMICAL ABSTRACTS.

Significance of the volume-weight for soil science and plant nutrition. L. SMOLÍK (Bull. Czechoslov. Acad. Agric., 1929, [v], 460; Proc. Internat. Soc. Soil Sci., 1930, 5, 32—33).—The expression of soil analyses on a basis of "volume-weight" (Kopecky) instead of the customary dry-matter basis is discussed and recommended. A. G. POLLARD.

Relationship between the nutrient content and reaction of soil. H. WIESSMANN and E. SCHRAMM (Pflanzenbau, 1929, 6, 97; Bied. Zentr., 1930, 59, 195—197).—Acid soils are more frequently deficient in phosphates than neutral ones, since their phosphate content consists largely of iron and aluminium phosphates of low assimilability. Applications of phosphatic fertilisers to such soils are often ineffective, but the phosphate may be mobilised by liming. Chalk-bearing soils with an alkaline reaction are frequently poorer in assimilable phosphate than neutral soils, their phosphate being largely tricalcium phosphate, which is not freely available to plants. On such soils phosphate manuring is made successful by the conjoint use of physiologically acid fertilisers, especially ammonium sulphate. In both acid and alkaline soils the adjustment of the "lime condition" must be considered in determinations of their phosphate requirements. The available potash contents of neutral soils is higher than that of acid ones. Liming the latter mobilises potash as a result of base exchange, thereby increasing the rate of availability, and, in soils poor in potash, leading subsequently to more acute deficiency. Among chalk-bearing soils little difference is apparent in the assimilable potash contents of neutral and alkaline samples.

A. G. POLLARD.

Determination of the nutrient requirement of soils by means of field and pot experiments. E. A. MITSCHERLICH (Landw. Jahrb., 1930, 71, 445—467).—Numerous comparative determinations are recorded of the nutrient contents and fertiliser requirements of soils by means of field trials and by the author's method. The errors to which the two methods are subject are discussed. The first approximation to the

"effect law" offers a satisfactory means of examination of soils for the above values. A. G. POLLARD.

Practical significance of soil research with special reference to phosphates and the methods of Neubauer and Lemmermann. H. ELLEDER (Z. Pflanz. Düng., 1930, 9B, 145—161).—Results of determinations of the phosphate requirements of soils by the two methods are discussed in comparison with field trials, and the relative values of these methods to the practical agriculturist examined.

A. G. POLLARD.

New hypothesis of the mode of action of potash [in plants]. A. BRUNO (Sci. Agric., 1930, 10, 422—425).—In the process of carbon assimilation by plants, energy supplied by direct solar radiation is supplemented by, or transformed into, radioactive emissions from potash (associated particularly with the isotope 41) present in the cell-sap. A. G. POLLARD.

Effects of raw phosphate on the soil. A. N. LEBEDIANTZEV (Fertilisers and Yields, Russia, 1929, 26—30).—Raw phosphates are effective on chernozem and chestnut as well as podsol and degraded chernozem soils. Raw phosphate is beneficial also on ameliorated alkali soils. CHEMICAL ABSTRACTS.

Forms of phosphorus in the soil and response of soils to phosphate fertilisers. A. I. DUSHECHKIN (Fertilisers and Yields, Russia, 1929, No. 4, 195—201).—Response to phosphate fertilisation diminishes in the order: chernozem, degraded chernozem, chernozem-like loam, grey forest loam. In chernozem soil (which is richest in phosphorus) the amount of organic phosphorus is greater than in other soil types. The soils which did not respond to phosphorus fertilisation responded to treatment with nitrogen.

CHEMICAL ABSTRACTS.

Mineralogical characteristics of Russian phosphate deposits and their agronomic utilisation. M. P. FIVEC and S. N. ROZANOV (Fertilisers and Yields, Russia, 1929, 201—211).—Russian phosphate deposits containing no apatite consist of kurskite and staffelite. The three petrographic types are: glauconitic clay, sandy, and glauconitic. Amorphous kurskite is as good as soluble phosphate in sand or soil cultures, whilst crystalline staffelite gives negative results.

CHEMICAL ABSTRACTS.

Influence of peat on the utilisation of phosphoric acid from phosphates. Z. V. LOGVINOVA (Fertilisers and Yields, Russia, 1929, 211—216).—On podsol soils mixtures of raw phosphate and peat gave a more prolonged increase in yield (clover) than phosphate alone. Composting did not affect the availability of phosphate; the hydrogen-ion exchange appears to take place immediately. With meadow peat, where composting is advantageous, the influence was not so marked.

CHEMICAL ABSTRACTS.

Improved quality of crops manured with phosphates and potash. O. NOLTE (Ernähr. der Pflanze, 1929, 25, 306—309; Bied. Zentr., 1930, 59, 210—211).—The importance of a consideration of the quality as well as quantity of crops produced in fertiliser trials is emphasised. A. G. POLLARD.

Determination of the lime requirement of soils.

DENSCH and STEINFATT (Landw. Jahrb., 1930, 71, 433—444).—The lime requirement of light soils cannot be based on determinations of exchange acidity alone. There is no parallelism between the exchange and hydrolytic acidities in the soils examined. Light soils with < 3 c.c. of hydrolytic acidity are but little harmful to barley. As the hydrolytic acidity increases beyond 4 c.c. injurious effects steadily increase. With oats there is no serious damage until the hydrolytic acidity reaches 8 c.c. On heavy soils these limiting values are higher. *Azotobacter* are inactive in soils having a hydrolytic acidity in excess of 1.4 c.c. Kappen's views as to the relative toxicity of hydrolytic and exchange acidities towards acid-sensitive plants are not confirmed. Numerous pot and field experiments indicate that soils should be grouped in three classes according to whether their hydrolytic acidities are within the ranges 0.4, 4.8, and > 8 c.c. Such grouping forms the basis of determinations of lime requirement. A. G. POLLARD.

Manurial action of magnesium sulphate and its dependence on the lime condition of soils.

A. GEHRING (Prakt. Blät. Pflanzenbau ü Pflanzenschutz, 1929, 6, [12]; Bied. Zentr., 1930, 59, 209—210).—Field trials indicated that the manurial effect of magnesium sulphate is primarily dependent on the lime status of soil and on the manner of application. On soils poor in lime magnesium sulphate principally affects the physical condition, but in lime-rich soils its nutrient effect is predominant. In laboratory experiments soils treated with increasing proportions of calcium hydroxide showed steadily increasing contents of easily soluble magnesia (0.05% HCl) which, however, declined when the amount of lime added exceeded the soils' absorption capacity. A. G. POLLARD.

Effect of cropping with various fertiliser, manure, and lime treatments on the exchangeable bases of plot soils.

C. J. SCHOLLENBERGER and F. R. DREIBELBIS (Soil Sci., 1930, 29, 371—394).—Continuous cropping of unlimed soils increases the proportion of exchangeable hydrogen and aluminium and decreases that of manganese, calcium, magnesium, potassium, and sodium. On limed soils the effects are reversed. In unlimed but fertilised soils the exchangeable hydrogen content is increased by the use of ammonium sulphate, increased slightly by superphosphate, dried blood, and oil-cake meal, not affected by potassium chloride, and decreased by sodium and calcium nitrates, bone meal, and basic fertilisers generally. The effect of farmyard manure is indefinite. In general, variations in the p_H values of soils follow those of the exchangeable-hydrogen content. Changes in the exchangeable-aluminium content follow those of hydrogen, but the actual amounts concerned are insignificant. The exchangeable-manganese content is not markedly affected by fertilisers, being increased slightly by ammonium sulphate and farmyard manure. Fertilisers containing calcium, and also sodium nitrate and organic manures, increase the proportion of exchangeable calcium, whilst ammonium sulphate decreases this value and potassium chloride is without effect. Organic manures cause an increase, and ammonium sulphate a heavy decrease,

in the exchangeable-magnesium content of the soil. Exchangeable potash is increased by potash fertilisers and to some extent by farmyard manure. Of the total potassium applied to soils and not removed in cropping, about one quarter appears in the exchangeable form in the surface soil. Sodium nitrate slightly increases the exchangeable-sodium content of soils. Liming increases the proportion of exchangeable calcium and magnesium and decreases that of manganese. Ground limestone is as effective as quicklime in this respect. The presence of chalk in soils does not prevent the exchange of manganese, magnesium, etc. for ammonium during leaching with ammonium acetate solution, but may delay the completion of the exchange.

A. G. POLLARD.

Utilisation of nitrogenous organic compounds and sodium salts of organic acids by certain soil algæ in darkness and in light.

C. E. SKINNER and C. G. GARDNER (J. Bact., 1930, 19, 161—179).—Citric, lactic, malic, succinic, tartaric acids, but not oxalic acid (all neutralised to p_H 6.0), glucose, gelatin, peptone, albumin, and casein increased the growth of several species of soil algæ. Glucose and the nitrogenous substances served as sole sources of energy for some algæ grown in total darkness in liquid media.

A. G. POLLARD.

Relative fertilising action of sodium nitrate, calcium nitrate, and Chile saltpetre.

HUPPERT (Fortschr. Landw., 1929, 4, 452—457; Chem. Zentr., 1929, ii, 2814—2815).—All the observed variations were within the limits of experimental error.

A. A. ELDRIDGE.

Calcium cyanamide as a nitrogen fertiliser.

E. I. RATNER (Fertilisers and Yields, Russia, 1929, 71—76).—Calcium cyanamide with superphosphate or precipitated phosphate gave higher yields than either nitrates or ammonium sulphate. Results obtained with fresh and old samples were compared. The amount of soil moisture does not affect the calcium cyanamide; sandy soils are more likely to be injured than clay soils.

CHEMICAL ABSTRACTS.

Action of nitrogen, and nitrogenous fertilisers.

KLEBERGER and RUDEL (Fortschr. Landw., 1929, 4, 441—444; Chem. Zentr., 1929, ii, 2814).—A question of relative cost.

A. A. ELDRIDGE.

Nitrogenous fertilisers. E. HOFFMANN (Fortschr. Landw., 1929, 4, 478—482; Chem. Zentr., 1929, ii, 2814).—A discussion of chemical considerations relating to the economic value of nitrogenous fertilisers.

A. A. ELDRIDGE.

Fertilisation experiments with stable manure, their evaluation and interpretation.

H. VON RATHLEF (Fortschr. Landw., 1929, 4, 411—415; Chem. Zentr., 1929, ii, 2814).—A discussion.

A. A. ELDRIDGE.

Effect of alfalfa [lucerne] and sweet clover roots and tops on the carbon dioxide evolution and nitrate accumulation of soils. T. L. MARTIN (Soil Sci., 1930, 29, 363—369). The rate of decomposition of lucerne and sweet clover tissue in soil is examined by means of the carbon dioxide and nitrate produced. Results generally may be correlated with the C:N ratio of the material, except in soils where certain *Mucors* occur in vigorous

colonies. The activity of these fungi appears to vary with the nature of the organic matter supplied.

A. G. POLLARD.

Fungicidal properties of certain spray-fluids. VI. W. GOODWIN, H. MARTIN, and E. S. SALMON (J. Agric. Sci., 1930, 20, 18—31).—Tests of ground sulphur or flowers of sulphur against hop powdery mildew, *Sphaerotheca humuli*, showed that when wetted by soft soap solutions the materials were completely effective at a concentration of 5 g. per 100 c.c. of 0.5% soap solution. Commercial colloidal sulphur (Ialine) was completely effective at 0.3 g. per 100 c.c. of 0.5% soap solution. Spreaders of an acid nature (gelatin and saponin) inhibited, and alkaline spreaders (lime-casein and soda-casein) favoured fungicidal action.

E. HOLMES.

Hydrolysis of sulphur in relation to its fungicidal activity. H. MARTIN (J. Agric. Sci., 1930, 20, 32—44).—It is suggested that the fungicidal action of sulphur on the *Erysiphaceae* is due primarily to hydrolysis, which action should be accelerated by the presence of alkalis, and should afford an explanation of the action of acid and alkaline spreaders (cf. previous abstract).

E. HOLMES.

Sulphur as a soil fungicide against the potato-wart disease organism. W. A. ROACH [with appendix by R. P. HOBSON] (J. Agric. Sci., 1930, 20, 74—96).—Thiosulphuric acid, which has been shown to exist in a free state, is sufficiently stable in dilute solution to account for the fungicidal action of acidified thiosulphate solutions in terms of the liberated thiosulphuric acid. The toxicity of the minimum quantity of sulphur found effective against wart disease of potatoes may be accounted for by assuming only 6% of the applied sulphur as being converted into thiosulphuric acid over a period of ten days. Pentathionates were found in sulphur-treated Rothamsted soils, but not in Ormskirk soils at 30°, and in neither at 0° and 15°. No evidence of accumulation of appreciable quantities of thiosulphuric acid in the soil was obtained, but this negative evidence is not final. The pentathionates found were probably derived from thiosulphuric acid previously formed.

E. HOLMES.

Effect of seed-potato treatment on yield and *Rhizoctonia* in N.E. Maine in 1925—1928. E. S. SCHULTZ, L. O. GRATZ, and R. BONDE (Phytopath., 1930, 20, 47—64).—Treatment of potatoes with mercuric chloride, formaldehyde, and various mercury organic preparations is described. In general, sprouting and vigour were improved, stem lesions and sclerotial infection with *Rhizoctonia* inhibited, and yields increased. Dipping of seed in 1 : 1000 mercuric chloride solution for 1½ hrs. proved most effective.

A. G. POLLARD.

Linseed oil as an adhesive for Bordeaux mixture. M. J. N. (J. Mysore Agric. Exp. Union, 1929, 9, No. 3; Trop. Agriculturist, 1930, 74, 115).—Linseed oil in the proportion of 10 fl. oz. per 25 gals. of 5 : 5 : 25-Bordeaux mixture proved an effective adhesive.

Relation of green manures to the carbon and nitrogen contents and reaction of soils at Peradeniya. A. W. R. JOACHIM and D. G. PANDITSEKERE

(Trop. Agriculturist, 1930, 74, 10—14).—Losses of carbon and nitrogen from green-manured soils were small in comparison with those of similar but uncultivated soils. Green manuring reduced soil acidity.

A. G. POLLARD.

Biological decomposition of some types of litter from North American forests. E. MELIN (Ecology, 1930, 11, 72—101).—The rate of decomposition of the leaves of a number of forest trees is examined by means of the carbon dioxide produced. In any one species the rate of primary decomposition is proportional to the nitrogen content of the leaves, but this relation is not applicable to leaves of all species. The rate of decomposition is affected to varying extents by the proportion of lignin and water-soluble matter in the leaves and by the nature of the leaf proteins. The effect of these factors is probably characteristic for a given species.

A. G. POLLARD.

Seasonal fluctuations in numbers of micro-organisms and nitrate-nitrogen in an Alberta soil. J. D. NEWTON (Sci. Agric., 1930, 10, 361—368).—Nitrate accumulation was generally greatest in fallow soils, least under grass, with cropped soils in an intermediate position. Although variations in nitrate content and bacterial numbers in soils were not closely related, there was a general parallelism between them. Variations in fungal numbers were smaller than, but similar to, those of bacteria. The numbers of *Actinomyces* fluctuated irregularly.

A. G. POLLARD.

Explanation of the action of the so-called accessory substances in the association of *Azotobacter* and cellulose-decomposing organisms. C. E. SKINNER (J. Bact., 1930, 19, 149—159).—*Azotobacter chroococcum* increased the rate of change toward acidity of media containing ammonium sulphate and cellulose during decomposition by *Spirochaeta cytophaga*. In similar media containing potassium nitrate the change toward alkalinity was similarly accelerated. When *Trichoderma Koningi* was used to decompose the cellulose, *A. chroococcum* had no effect on the reaction changes. The amounts of cellulose decomposed by these organisms were unaltered by the presence of *A. chroococcum*. The postulation of the secretion of growth-promoting substances by *A. chroococcum* is unnecessary, since its effect on the reaction of the media may be explained by the utilisation of the intermediate products of decomposition of the cellulose.

A. G. POLLARD.

Influence of soil reaction and manuring on the composition of a mixture of various grasses and clovers. K. NEHRING and A. KELLER (Landw. Jahrb., 1930, 71, 487—503).—In mineral soils exhibiting exchange acidity, soil reaction has no very marked effect on the yield of grasses nor on the utilisation of physiologically different fertiliser mixtures. Liming of these soils increases the calcium content of the grasses. In moorland pastures clover was present although the soils were acid. Liming increased the yields on these soils. Physiologically acid fertilisers largely affected the clover yields, but with physiologically alkaline mixtures the ratio clover : grasses was independent of the soil reaction. Potash-phosphate fertiliser mixtures pro-

duced larger proportions of clover in the herbage than did complete fertilisers. Variations in the proportion of ash, calcium, and nitrogen in the herbage, following fertiliser treatment, were mainly the outcome of changes in the proportion of clover produced.

A. G. POLLARD.

Early *versus* late ploughing of sweet clover for green manure. D. H. JONES and E. H. GARRARD (Sci. Agric., 1930, 10, 419—422).—Changes in the nitrogen content of sweet clover during the growth period are recorded and used as a basis for discussion of the best period of ploughing-in.

A. G. POLLARD.

Effect of soil type and fertiliser treatment on the composition of the soya-bean plant. R. H. AUSTIN (J. Amer. Soc. Agron., 1930, 22, 136—156).—The calcium, magnesium, and nitrogen contents decreased with the age of the plants, the sulphur content underwent little change, the phosphorus content tended to increase, whilst the potassium content was irregular. The effects of moderate fertiliser treatments were small, the soil type exerting a greater influence.

CHEMICAL ABSTRACTS.

"Lime antagonism" of lupins. DENSCH and STEINFATT (Z. Pflanz. Düng., 1930, 9B, 161—174).—Characteristic lupin sickness (as distinct from injury from acid soil conditions, or lack of nutrients) is not directly attributable to calcium carbonate, but is due to the presence in soil of alkali carbonates or conditions favourable to their production, *e.g.*, the presence of excessive proportions of chalk.

A. G. POLLARD.

Utilisation of certain nitrogen compounds by the sugar cane. J. H. PARDO (Internat. Sugar J., 1930, 32, 11—18).—Results of experiments are given showing that sugar cane is able to obtain its nitrogen requirements from compounds other than nitrates, *e.g.*, by the direct utilisation of ammonium compounds. In the case of absorption of ammonium compounds the total nitrogen content of the cane plants is well above that of plants fed on nitrates and nitrites. There is a greater assimilation of nitrogen by sugar cane when this element is present in the form of ammonium compounds than when given as nitrates. The author's experiments have shown that the nutritive value to the young cane plant of one unit of nitrogen as ammonia is superior to that of the same unit given in the form of nitrate.

J. P. OGILVIE.

Sugar-beet trials in 1929. T. REMY [with E. OHLY and F. WEISKE] (Landw. Jahrb., 1930, 71, 345—432).—Numerous field trials are recorded and discussed from the point of view of the effects of cultural methods, fertilisers, and general treatment on the yield, sugar content, and growth characteristics of sugar beet.

A. G. POLLARD.

System of proximate chemical analysis of plant materials. S. A. WAKSMAN and K. R. STEVENS (Ind. Eng. Chem. [Anal.], 1930, 2, 167—173).—Whilst it is impossible for any method of analysis of plant materials to give results having a total of 100%, unnecessary errors are caused by ignoring water-soluble constituents, by confusion in the methods used for

pentosans and lignins, and by high results in the determination of cellulose by Cross and Bevan's method. Known methods are criticised in detail. The present authors' system divides plant constituents into (1) ether- and alcohol-soluble fractions, (2) cold- and hot-water-soluble fractions, (3) hemicelluloses, (4) celluloses, (5) lignins, (6) proteins, (7) ash. Part of the organic nitrogen is always water-soluble and a correction must be made. Extractions are made successively with ether, cold water, hot water, and alcohol. The water-soluble portions are tested for reducing sugars, total organic matter, ash, and nitrogen. The residue from the alcohol extraction is boiled with 2% hydrochloric acid for 5 hrs. and the hemicellulose determined as reducing sugar. The dried residue from this treatment is subjected to the action of cold 80% sulphuric acid for 2½ hrs., which converts cellulose into hydrocellulose. On diluting and boiling, the last-named is converted into reducing sugar. The residue is washed, dried, and weighed as (lignin and ash and organic nitrogen compounds). The nitrogen content of the residue is determined. Examples of analyses of plants and peat by this system give results totalling 88—95%.

C. IRWIN.

Chemistry and certain problems of applied mycology. H. MARTIN (J.S.C.I., 1930, 49, 11—14 T).—A discussion of the chemistry of Bordeaux and Burgundy mixtures and of the fungicidal properties of sulphur and of the products of the alkaline hydrolysis of sulphur.

Wetting, spreading, and emulsifying agents for use with spray fluids. I. Wetters and spreaders. II. Emulsifiers. R. M. WOODMAN (J.S.C.I., 1930, 49, 93—98 T, 193—197 T).—I. The terms "wettters" and "spreaders" are critically defined, and the importance of wetting and spreading with all classes of spray fluids is demonstrated. A laboratory examination is made of several well-known and possible wettters and spreaders, the tension fluid/air being measured; drawbacks to this method of estimating the wetting and spreading powers of a fluid are pointed out. Barium caseinate will probably serve the dual purposes of an internal poison insecticide and a wetter (or spreader). Traces of various liquids may act as wettters and spreaders; solutions of some liquids may also be insecticidal at the same time. The use of emulsions as wettters and spreaders is discussed. Sodium resinate is slightly more effective as a wetter and spreader in moderately concentrated salt solutions than in water alone. The differences existing between the practical and theoretical conceptions of adequate wetting and spreading are discussed, and methods of conferring these properties other than by lowering the tension spray fluid/air are advocated to some extent. The problem of the retention of the maximum amount of spray fluid by the plant is also mentioned.

II. The rules to be obeyed by an emulsifier used in the preparation of spraying emulsions for insecticidal purposes are discussed. The influence of hard and pseudo-hard waters on certain emulsifiers is demonstrated; results are given for the emulsification of oils, embracing all the known types used as spraying oils,

by various emulsifiers. Oil solutions of naphthalene and mixtures of oils of possible horticultural value have been emulsified.

Tests of various aliphatic compounds as fumigants. R. C. ROARK and R. T. COTTON (U.S. Dept. Agric. Tech. Bull. 162, 1929, 52 pp.).—Of 309 aliphatic compounds tested against the rice weevil in wheat, iodides, bromides, mercaptans, thiocyanates, isothiocyanates, disulphides, oxides, epichlorohydrin, halogenated ethers and esters, and formates showed the greatest toxicity. Examination of the most toxic and least toxic compounds indicated that there was no apparent relation between their b.p. and toxicity, except that most compounds with b.p. above 150° have too low a vapour pressure at ordinary temperatures. Branched-chain are more toxic than straight-chain compounds, and whilst compounds relatively inert chemically have little toxicity, others of high reactivity may not kill weevils, probably owing to their absorption by the wheat. In general, chlorides, formates, disulphides, thiocyanates, isothiocyanates, and mercaptans, in applications lethal to the insect, do not injure seed germination, but the other materials mentioned may injure germination. Ethylene dichloride with carbon tetrachloride in the ratio of 3:1 by vol., at a rate of 6 lb. per 1000 cub. ft. of stored grain, appeared to be the most promising fumigant.

E. HOLMES.

Rôle of vitamins and minerals in stock feeding.
II. Minerals. J. B. ORR (J. Min. Agric., 1930, 37, 115—129).—A review of the mineral requirements of farm animals and poultry, the mineral contents of common feeding stuffs, and simple methods of adjusting deficient rations.

E. HOLMES.

Effect of green manures and cover crops on soil moisture. A. W. R. JOACHIM and S. KANDIAH (Trop. Agriculturalist, 1930, 74, 3—9).

Method of studying the activity and rate of diffusion of protozoa and bacteria in the soil. L. LOSINA-LOSINSKY and P. F. MARTINOV (Soil Sci., 1930, 29, 349—362).

Soils of Logan, Whiteside, Henry, Morgan, Douglas, and Coles Counties. R. S. SMITH, E. E. DE TURK, F. C. BAUER, and L. H. SMITH (Illinois Agric. Exp. Sta. Soil Rep., 1929, Nos. 39—44).

Analyses of commercial fertilisers, fertiliser supplies, and home mixtures for 1928. C. S. CATHCART (New Jersey Agric. Exp. Sta. Bull., 1928, No. 479, 1—39).

Analyses of commercial fertilisers and ground bone; analyses of agricultural lime, 1928. C. S. CATHCART (New Jersey Agric. Exp. Sta. Bull., 1928, No. 481, 1—29).

Spray residues on fruit. STREETER and HARMAN; SHEER.—See XIX.

PATENT.

Appliances for spraying liquids [for agricultural purposes]. A. H. BURGESS (B.P. 328,707, 14.2.29).

XVII.—SUGARS; STARCHES; GUMS.

Electrodialysis of exhausted molasses. II. N. KAMEYAMA and Y. MAYEDA (J. Soc. Chem. Ind., Japan, 1929, 32, 206 B).—In the process previously described (B., 1930, 478) the anode compartment becomes acidic owing to the preferential transfer of solution cations through the anode membrane. It is advisable, therefore, to use for the anodic membrane a silk membrane which has been soaked in dichromated gelatin and exposed to sunlight. A certain amount of sugar is, however, lost with this membrane.

S. K. TWEEDY.

Heat capacity of pure sugar solutions. V. V. YUANOVSKI and P. A. ARKHANGELSKI (J. Appl. Chem., Russia, 1929, 2, 614—618).—The heat capacity at constant temperature decreases linearly with increasing concentration, and at definite concentration is a linear function of the temperature. $C^{20} = 1 - 0.00567p$; $C^{50} = 1 - 0.00510p$; $C^{80} = 1 - 0.00456p$, where p is the percentage of sugar in solution (Brix) and C is the sp. heat.; $M^{20} = 148.2$, $M^{50} = 167.7$, $M^{80} = 185.9$, $C_y^{20} = 0.433$, $C_y^{50} = 0.490$, $C_y^{80} = 0.544$, where M is the mol. heat.

CHEMICAL ABSTRACTS.

Errors of weight and polarisation arising from the pulping of [sugar] beet. O. KOPECKÝ (Z. Ver. deut. Zucker-Ind., 1930, 80, 139—154).—A study of the losses by evaporation occurring when 1-g. portions of beet tissue are ground in a small mortar for 3 or 5 min. and afterwards exposed to the air for 40 min. The losses of weight during actual grinding were 0.4—1.24%, and the final losses after 40 min. were 1.14—2.5%.

J. H. LANE.

Temperature coefficient in the polarisation of raw cane sugars. K. D. DEKKER (Archief, 1929, 37, 909—914; Internat. Sugar J., 1930, 32, 202—203).—Using the so-called Java method (dissolving 26.048 g. in a flask calibrated at 17.5°, making up to 100 c.c. at 30°, and polarising the solution at 30°), 16 raw cane sugars were found to polarise from 0.05° to 0.50° lower than when the procedure of the International Commission was followed. The differences between the two sets of readings were very irregular, due to the varying amounts of reducing sugars present in the samples and to the relative proportions of dextrose and lævulose they contained.

J. P. OGILVIE.

Molar coefficient of impurities and coagulating method for purifying [sugar] diffusion juices. P. V. GOLOVIN (Zhur. Sakh. Prom., 1929, 3, 409—410).—The purity of sugar is conveniently expressed as mols. of non-sugar per mol. of sugar. The diffusion juice at 40° is treated with milk of lime (0.5% CaO) and freshly precipitated calcium carbonate (1—2% CaO); the filtered juice is treated at 95—100° for 20 min. with lime (1%) and then saturated with carbon dioxide.

CHEMICAL ABSTRACTS.

[Sugar] juice of first and second carbonations and optimal conditions in saturation. N. V. ALEINIKOV (Nauch. Zapiski, 1929, 8, 401—417).—A temperature of 90°, fractional defecation, addition of milk of lime and filter cakes from the second

carbonatation are recommended. The juice of the first defecation is carbonatated to the lowest optimal point, heated at 100°, and filtered; after addition of milk of lime the heating and filtration are repeated. At the second defecation the juice is submitted at a high temperature to the prolonged action of lime (about 0.25%), carbonatated to the lowest optimal point, and filtered without preheating.

CHEMICAL ABSTRACTS.

Sweet sorghums for syrup and forage. C. K. McCLELLAND (Ark. Agric. Exp. Sta. Bull., No. 241, 1929, 1—24).—"Honey sorghum" yields 15—238 (average 75) gals. of syrup per acre; 30—45% of the weight of the cane is obtained as juice. The sugar content varies from 5.7 to 14.7%.

CHEMICAL ABSTRACTS.

Methods of preparing quickly-soluble lactose. R. W. BELL (Ind. Eng. Chem., 1930, 22, 51—54).—The fact that β -lactose anhydride, though unstable at temperatures below 93°, is more soluble than the commercial α -hydrate was applied to the preparation of an apparently "sweeter" lactose for commercial purposes. Under suitable conditions a high yield of β -anhydride was obtained by vacuum-drum or atmospheric double-drum dryers. By the Gray-Jensen spray process a powder was obtained having $[\alpha]_D^{20} + 55^\circ$, indicating an equilibrium mixture. E. B. HUGHES.

Influence of cold on the tenacity of potato starch. SCHULZ and PARLOW (Z. Spiritusind., 1930, 53, 135—136).—Potatoes which were frozen at 18—20° below zero showed the characteristic soft and sponge-like texture after thawing, but gave none of the reactions showing the formation of sugar. The tenacity, which was measured according to the Wolff standards, was determined on the starch of the original potatoes and on that of the potatoes immediately after freezing and after the frozen potatoes had been thawed for eight days. Similar measurements were made on free potato starch containing various amounts of moisture. Both the free starch and that from the potatoes showed an increase of tenacity after freezing which, in the case of the potatoes, diminished again after thawing, but did not completely return to its original value.

C. RANKEN.

Analysis of starch formate. TOCCO and NYSENS.—See V. **Sugar-beet trials.** REMY and others.—See XVI. **Determination of sucrose in condensed milk.** ANON.—See XIX. **Waste-waters from sugar-beet.** GARNER and WISHART.—See XXIII.

PATENTS.

Continuous washing of tricalcium saccharate. C. STEFFEN, JUN. (U.S.P. 1,757,979, 13.5.30. Appl., 21.2.24. Austr., 28.2.23).—See G.P. 406,060; B., 1925, 466.

Fermentation of starch etc. (B.P. 328,723).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Poisonous action of grain meal on yeast. J. RUCHS (Woch. Brau., 1930, 47, 171—174, 183—186).—From the results of staining and fermentation experi-

ments it is claimed that no substance poisonous to yeast is present in normal meals. Wheat and barley meals exhibited apparent harmful effects, but only after the yeast had been exposed to the action of distilled water or of sugar dissolved in distilled water (cf. Fink and Weinfurtner, B., 1930, 526). When the distilled water is replaced by supply water or the sugar solution by wort the yeast is unaffected by the addition of meal; further, the yeast cannot be accustomed to the supposed poison. F. E. DAY.

Influence of carbon dioxide on yeast fermentation. HOFMANN (Woch. Brau., 1930, 47, 228—230).—When 20% sucrose solution is fermented in an autoclave with 10% of yeast, fermentation ceases when the pressure reaches about 40 atm., the liquid then containing about 4.2 g. of alcohol per 100 c.c. When the vessel was connected to a cylinder of carbon dioxide for 1 hr., at the beginning of the experiment the initial pressure was 56 atm., which fell to 32 atm. and then increased to 38 atm., 2.7 g. of alcohol per 100 c.c. being produced. After 2 hrs.' connexion and occasional shaking the alcohol produced (per 100 c.c.) was 1.7 g., and after 6 hrs. it was 0.74 g. Under the latter conditions the same result was obtained when the yeast was forced into the autoclave by hydrogen after treatment of the culture medium with the carbon dioxide. It is suggested that the small amount of fermentation which occurs under high pressure of carbon dioxide is due to the enzyme already present in the yeast, and that the favourable influence of low pressure of that gas, noted by Stockhausen and Windisch (B., 1928, 685), are due to the well-known activating effect of small amounts of inhibiting substances. The suggestion of Windisch (B., 1930, 479) that the inhibiting effect of high pressure of carbon dioxide is due to lowering of the p_H , is considered unlikely as the degree of dissociation decreases with increasing concentration. The addition of alcohol increases the inhibiting effect of carbon dioxide; after an initial addition of 3.6%, the final pressure was 28 atm. and 1.8% of alcohol was formed (cf. A., 1929, 1199). F. E. DAY.

Physical aspects of fermentation and their relation to brewery practice. A. C. DOULL and S. E. BARNETT (J. Inst. Brew., 1930, 36, 215—220).—Although the fermentation loss varies according to the original gravity of the wort and the range of attenuation, a figure can be obtained which may be considered as normal for the different types of beer brewed in a particular brewery. The change in volume of the wort which occurs from the commencement of fermentation till the beer is ready for racking is not appreciably affected either by the change of temperature or by the production of alcohol during fermentation. The evolution and loss of carbon dioxide causes a slight but definite loss of volume which is compensated by a prior increase due to the loss or fall in density of the wort during the first few hours of fermentation. The amount of yeast reproduction may be rapidly ascertained by calculating the weight of that volume of beer, which is the difference between the dip at collection and the dip after skimming. C. RANKEN.

Iodine value and degree of luminescence of fermentation vinegar and its content of the products of bacterial metabolism. A. JANKE and H. LACROIX (Biochem. Z., 1929, 215, 460—467).—Fermentation vinegar and "essence" vinegar may be differentiated in terms of the degree of fluorescence in ultra-violet light, but for differentiation of mixtures of these the iodine value is the more useful. The degree of fluorescence does not run parallel with the iodine binding power. The fluid obtained by the growth of pure cultures of *B. ascendens* on alcoholic solutions of mineral salts shows luminescence, has an iodine-binding power, and stimulates the growth of yeast cells. It would appear therefore that substances giving rise to these properties in fermentation vinegar are products of bacterial metabolism. P. W. CLUTTERBUCK.

Time and temperature of kilning [malt]. J. BOLDT (Woch. Brau., 1930, 47, 174—177).—A discussion of kilning conditions, with special reference to the production of pale (Pilsener) malt on a two-stage kiln. Periods of 12 hrs. on each floor are recommended, also thin loading and a good draught. On the upper floor the temperature should begin at about 35°, and rise, slowly at first, to about 45°. The moisture should be 10—12% after 8—10 hrs. A temperature of about 62° on the lower floor is favourable to head retention. Rapid and sufficient drying in the earlier stages allows pale malt to be finished at about 80° during the last 3 hrs., and such malt gives better flavoured and more stable beers. F. E. DAY.

Fluorescence of malt and beer. V. BERMANN (Woch. Brau., 1930, 47, 215—217).—Malt extracts and beers exhibit a faint green to blue-green fluorescence which is caused neither by the mineral constituents nor by the products of hydrolysis of starch and protein. It is considered to be due to melanoidins, and to indicate the presence of a ring-structure in their constitution. F. E. DAY.

Volatility of acid and ester in brandy manufacture. E. WALTER (Deut. Dest.-Ztg., 1929, 50, 401—402; Chem. Zentr., 1930, i, 141).—The esters are much more readily volatile than the acids. A. A. ELDRIDGE.

Determination of p_H values in provincial distilleries. J. DEHNICKE (Z. Spiritusind., 1930, 53, 133—135).—The determination of p_H values in provincial distilleries is of little assistance as a control of the fermentation processes. In the mashing of the sweet wort and during the primary fermentation and preparation of the lactic acid yeast the usual technical methods yield worts with a high buffer content which prevents any great variation in the p_H values, and retains them well within the range of optimum action of the yeast enzymes and after-saccharification. For these processes the determination of the acidity by titration with normal sodium hydroxide is more important, since it permits the detection of undue formation of the undesirable butyric and acetic acids resulting from infection of the wort or yeast. Only in the preparation of the sulphuric acid yeast is the p_H determination a valuable supplement. It allows the control of the added sul-

phuric acid, so that the optimum p_H of 3.6—3.8 is not exceeded. Although the titrated acidity may be normal during addition of this acid, the buffer content is so low that the p_H value may easily be diminished to such an extent that the action of the yeast enzymes is inhibited. C. RANKEN.

Wheat glutenin. BLISH and SANDSTEDT.—See XIX.

PATENTS.

Fermentation of starch, sugar, or the like containing materials. DEUTS. HYDRIERWERKE A.-G. (B.P. 328,723, 28.2.29. Ger., 22.10.28).—Butyl alcohol and acetone are produced by incubating starch, sugar, etc. for 1—2 hrs. under pressure, and then inoculating with *Butylobacter*. The latter is a new type of bacteria characterised by the Gram-positive, young cells becoming later Gram-negative, and finally changing to Gram-positive after 48 hrs. The cells, which are 2—3 μ in length and 0.25—0.3 μ in diam., form neither chains nor threads under normal conditions. C. RANKEN.

Apparatus for production of carbon monoxide [from fermenter gas]. W. C. ARSEM, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,740,139, 17.12.29. Appl., 6.5.25).—Fermenter gas, evolved during butyl alcohol-acetone fermentation of carbohydrates and modified so that the carbon dioxide and hydrogen are present in molecular proportions, is led into a converter shell in which is suspended a heating element containing a catalyst. A series of baffles is placed to cause the gas passing through the reaction chamber to make contact alternately with the heating element and the internal walls of the shell, which provide the condensing surfaces for the resulting water vapour. C. RANKEN.

Leuco-indigo preparations (B.P. 327,864).—See III.
Articles resistant to wear etc. (B.P. 307,011).—See X.

XIX.—FOODS.

Milk products. II. Report of the Milk Products Sub-Committee to the Standing Committee on Uniformity of Analytical Methods. **Determination of sucrose in sweetened condensed milk** (Analyst, 1930, 55, 111—124; cf. B., 1927, 613).—The methods examined for the reduction of sucrose in sweetened condensed milk comprised (a) copper reduction, (b) iodometric and chloramine-T, and (c) polarimetric methods. Of these, method (a) was rejected, and method (b) of Hinton and Macara (B., 1928, 66) was found promising for lactose, but not sufficiently accurate for sucrose. Polarimetric methods (c), all based on the principle of the Clerget inversion, were investigated in regard to the following details: **Clarification.**—The Dutch Codex method, using zinc acetate and potassium ferrocyanide, was adopted with phosphotungstic acid as a reagent suitable in certain circumstances. **Mutarotation.**—Although use of water at 80—90° is sufficient to destroy the effects of mutarotation, ammonia followed by neutralisation is recommended. **Effect of non-sugars.**—The direct reading should be taken on the clarified filtrate without further

treatment, and the invert reading without neutralisation. *The inversion process.*—The principle of the Jackson and Gillis process (B., 1920, 634 A), slightly modified, has been adopted. *Inversion divisor factor.*—Factors for both clarifying reagents with sodium light, mercury-green line, and International sugar scale (*j*) light are given. *Correction for volume of precipitate.*—The actual determination of the volume correction should be made, and details are given. Various results are given both in the report and in an appendix thereto; in each set of tabulated results, at least one obtained by the use of each of the three types of polarimeter, viz., saccharimeter with International sugar scale, polarimeter with sodium light, and polarimeter with mercury-vapour lamp, is included. The Committee recommend that for the determination of sucrose in sweetened condensed milk, the polarimeter method with zinc acetate-potassium ferrocyanide clarification should be employed, and that for the purpose of the Public Health regulations the percentage of total milk solids should be determined by subtracting the percentage of sucrose found by this method from the percentage of total solids as determined by the process described in Report I (*loc. cit.*). D. G. HEWER.

Evaporated and condensed milk from the chemical and nutritional point of view. F. E. RICE (Ind. Eng. Chem., 1930, 22, 45—48).—A description is given of the manufacture of evaporated and sweetened condensed milk from the chemical viewpoint, with comments on the causes leading to the production of faulty material and a discussion of the effect of condensation on the nutritional value of the milk.

E. B. HUGHES.

Irradiated milk. P. BAMBERGER (Deut. med. Woch., 1929, 55, 1547—1550; Chem. Zentr., 1929, ii, 2473).—Milk irradiated by the Hanauer or Scheidt method was not injurious to infants; it possessed marked antirachitic properties. 100 c.c. of Hanauer milk correspond with 5—6 g. of commercial cod-liver oil; Scheidt milk is less active.

A. A. ELDRIDGE.

Lipase in raw, heated, and desiccated milk. J. H. NAIR (Ind. Eng. Chem., 1930, 22, 42—45).—The work of Rice and Markley (J. Dairy Sci., 1922, 5, 64) that raw milk contains lipase has been repeated and the results are confirmed. Pasteurisation was found to cause destruction of the enzyme to an extent depending on the temperature used and the time of holding. No lipase was detected in dried whole or skim milk made from milk pasteurised at 63—64.5° for 30 min.

E. B. HUGHES.

Attempt to apply the cryoscopic method to the analysis of altered milk. C. MANICARDI (Giorn. Chim. Ind. Appl., 1930, 12, 115—118).—The cryoscopic test of milk is generally regarded as valueless when the milk has "turned." The results now given show, however, that the difference between the f.p. of genuine and watered milks when fresh is approximately maintained if the two samples are allowed to undergo spontaneous acidification to the same extent, provided that such acidification does not proceed too far.

T. H. POPE.

Some recent advances in the chemistry of milk. L. S. PALMER (Ind. Eng. Chem., 1930, 22, 39—42).—The results of work carried out by various experimenters during the last 2 years on milk, its analysis, and by-products are reviewed.

E. B. HUGHES.

Rising of fat in milk; percentage of fat in cream. G. D. ELSDON and J. R. STUBBS (Analyst, 1930, 55, 124—125).—The milk from samples of milk allowed to remain in a separator for varying times was drawn off and the cream layer examined; it was found that a very considerable separation of fat may take place in a comparatively short time in milks that have once been separated. A standard of 30% of fat for cream and 45—50% of fat for thick cream is regarded as desirable.

D. G. HEWER.

Keeping quality of butter made from cream of various acidities. W. WHITE, C. S. TRIMBLE, and H. L. WILSON (U.S. Dept. Agric. Tech. Bull. No. 159, 1929, 1—7).—There was less deterioration when the cream contained 0.15—0.25% than when it contained 0.28—0.31% of acid.

CHEMICAL ABSTRACTS.

Application of physical chemistry to ice-cream. A. LEIGHTON (Ind. Eng. Chem., 1930, 22, 48—51).—The more recent information on the physico-chemical problems of ice-cream manufacture is reviewed, consideration being given to the following stages of manufacture: temperature of pasteurisation; homogenisation and its effect on texture; the ageing (ripening) process, with a study of the rôle of gelatin as a stabiliser; temperature of freezing, whipping, and hardening.

E. B. HUGHES.

Nutritive value of pasture. V. Pasture grass conservation: influence of artificial drying on the digestibility of pasture herbage. H. E. WOODMAN, J. W. BEE, and G. GRIFFITH (J. Agric. Sci., 1930, 20, 53—62; cf. B., 1929, 487).—Young grass does not suffer depression of digestibility either by drying at 100° or by direct heat in a kiln at temperatures rising from 50° to over 90° towards the end of the 3 hrs.' drying period. Digestion coefficients are given for the organic matter (78.1%), crude protein (78.2%), ether extract (70.6%), nitrogen-free extractives (79.3%), and crude fibre (78.3%) of dried (100°) grass cake, showing them to be very similar to the figures for weekly-cut herbage in the fresh condition. Only the figure for ether extract differed materially—in favour of the dried grass cakes. Kiln-dried grass showed similar values.

E. HOLMES.

Nature and identity of wheat glutenin. M. J. BLISH and R. M. SANDSTEDT (J. Biol. Chem., 1929, 85, 195—206).—The discrepancies between the properties of different preparations of wheat glutenin as described by earlier workers have been due to the use of alkali in the extraction of the protein, since the properties of the latter are distinctly modified even by brief contact with dilute alkali. The protein is best prepared by kneading flour under tap-water and macerating the insoluble residue with a large volume of very dilute acetic acid; after keeping overnight, the solution is made 65—70% with respect to methyl alcohol and is

cleared from starch and other insoluble impurities on the centrifuge; the cautious addition of sodium hydroxide to p_H 7.0 (approx.) causes precipitation of the glutenin, gliadin remaining in solution. After two or three reprecipitations in a similar manner the glutenin is pure. The protein has a higher amide-nitrogen content (22%) and a greater molecular magnitude than glutenin prepared with the aid of alkali.

C. R. HARRINGTON.

Vitamin contents of Philippine foods. I. Vitamins-A and -B in *Basella rubra*, *Capsicum frutescens*, and *Vigna sinensis*. A. J. HERMANO (Philippine J. Sci., 1930, 41, 387—401).—Biological analyses of various Philippine foodstuffs show that the ether extract of crude rice bran contains the anti-sterility vitamin-E, that libato (*Basella rubra*) is rich in vitamins-A and -B, that the green pods of sitao (*Vigna sinensis*) contain vitamins-A and -B, but to a smaller extent than does alugbati, and that powdered chile leaves (*Capsicum frutescens*) are rich in vitamins-A and -B as shown by the curative effect on rats and pigeons. E. HOLMES.

Action of heat on preserved tomatoes. O. CARASCO and E. SARTORI (Giorn. Chim. Ind. Appl., 1930, 12, 119—121).—When heated at 100°, preserved tomatoes give carbon dioxide, furaldehyde, hydroxymethylfuraldehyde, etc., the proportions of reducing sugars and acid present undergoing marked diminution. Hence determination of the dry matter of the preserves by drying at 100° gives results considerably lower than those obtained by exposure in a vacuum in presence of fused potassium hydroxide and phosphoric anhydride, the differences being most marked with concentrated products. If the sugars are previously eliminated by fermentation, or the material is rendered alkaline, the two methods of drying give far more concordant results.

T. H. POPE.

Handling of sweet cherries with special reference to chemical and physiological activities during ripening. H. HARTMAN and D. E. BULLIS (Oregon Agric. Exp. Sta. Bull., 1929, No. 247, 1—38).—During ripening, the sugar, solids, and acidity of sweet cherries increase, and astringent constituents decrease. The fruit of later pickings yields a juice of higher sugar content. The juice of Napoleon cherries contained 18.8% of total sugars and 7.1% of solids-not-sugar; unripe fruit contained 0.166 and ripe fruit 0.08% of tannin.

CHEMICAL ABSTRACTS.

Washing fruit to remove spray residue in the Hudson Valley. E. V. SHEAR (N.Y. Agric. Exp. Sta. Bull. No. 575, 1929, 34 pp.).—The fruit is immersed for 1 min. in 0.2—1% hydrochloric acid and immediately rinsed in water. Alkalis, oils, and hand-wiping increase decay in storage. Rain is an important factor in reducing spray residue.

CHEMICAL ABSTRACTS.

Spray residues [on fruit]. L. R. STREETER and W. HARMAN (N.Y. Agric. Exp. Sta. Bull. No. 579, 1929, 1—12).—Arsenic is removed from apples in 1 min. at 13—21° by 1% hydrochloric acid. Only 3 of 94 samples of apples contained as much as 0.01 grain per lb.

CHEMICAL ABSTRACTS.

Use of sulphur dioxide in shipping grapes. H. E. JACOB (Calif. Agric. Exp. Sta. Bull. No. 471, 1929, 1—24).—The amount of sulphur dioxide absorbed by grapes exposed to an atmosphere containing 1.8—3.0% SO_2 is determined by the concentration and time. The preservative decreases the rate of activity of the normal life processes.

CHEMICAL ABSTRACTS.

First Report of the Sub-Committee on the determination of arsenic, lead, and other poisonous metals in food-colouring materials to the Standing Committee on the Uniformity of Analytical Methods. I. Determination of arsenic (Analyst, 1930, 55, 102—109).—The report deals with the determination of arsenic in synthetic dyestuffs, and after discussions on the destructive wet oxidation, distillation of the arsenic, and determination of arsenic in the prepared solution, the methods as adopted by the Sub-Committee are given in detail. The direct-distillation method is found to give a rapid and approximate idea of the amount of arsenic present, and as a rule the results are near to the truth, but in a few cases (e.g., nigrosine) low figures are obtained. In method A the sample is treated with nitric acid, followed by sulphuric acid and then by a further addition of nitric acid, the solution is boiled down in a special distillation apparatus without the condensing arm, diluted, again boiled down, and a chloride-hydrazine bromide mixture introduced, followed by hydrochloric acid. The resulting solution is then distilled into cooled dilute nitric acid, the distillate is evaporated to dryness, and the residue after being twice taken up with water, dissolved in sulphuric acid, and diluted is ready for transference to the arsenic apparatus. Method B is used for certain colours, particularly basic dyes, that are liable to violent deflagration in the oxidation process, and entails certain modifications in the first part of the process. The apparatus used for determination of the arsenic is that defined in the British Pharmacopœia, 1914, with the exception that the gas-exit tube of internal diam. 4 mm. shall not be widened out at the gas-exit end, but is to be ground smooth at right angles to the tube. Mercuric chloride test papers are used and the solution tested should contain 0.004—0.01 mg. As.

D. G. HEWER.

Arachidonic acid in lard. BROWN and DECK. **Babassu fat.** HEIDUSCHKA and AGSTEN. **Detection of fats in ultra-violet light.** LENFELD. **Grape-fruit seed.** JAMIESON and others.—See XII. **Minerals in feeding-stuffs.** ORR.—See XVI. **Sorghums for forage.** MCCLELLAND.—See XVII.

PATENTS.

Method and means for pasteurising milk. T. MELBALLE (B.P. 329,193, 7.6.29).

[Coating machines for] confectionery. M. M. GUGGENHEIM, G. R. BAKER, BAKER PERKINS, LTD., and ANC. ETABL. A. SAVY, JEANJEAN & CIE. SOC. ANON. (B.P. 328,607, 30.1.29).

Artificial foils etc. (B.P. 327,164).—See V. **Sodium glutamate** (B.P. 327,810).—See VII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of hydrogen cyanide in pharmaceutical preparations. G. VOGT (Pharm. J., 1930, 124, 475).—A current of air is drawn through the solution and the volatilised hydrogen cyanide is absorbed in silver nitrate solution. The results agree with those obtained by the B.P. method. It is shown that practically the whole of the hydrogen cyanide originally present in Tinct. Chlorof. et Morph., B.P. 1885, disappears after a short storage in closed vessels.

H. E. F. NOTTON.

Tests for peroxides [in ethyl ether]. C. C. PINES (Amer. J. Pharm., 1930, 102, 221—222).—The chromic acid test is preferred. Liberation of iodine from potassium cadmi-iodide or mercuri-iodide in presence of starch is a delicate test.

H. E. F. NOTTON.

Purification and testing of the paraldehyde of the German pharmacopœia. E. SCHULEK (Pharm. Zentr., 1930, 71, 177—179).—The test for per-compounds given in the German pharmacopœia is not sufficiently stringent, since samples which fulfil it liberate iodine from iodides. A more stringent test based on the iodine liberation is suggested. Purification may be effected by shaking with sodium hydroxide and silver nitrate solutions.

S. I. LEVY.

Determination of total iodine content of Tinct. Iodi with potassium bichromate. A. NEUMANN (Pharm. Ztg., 1930, 75, 532—533).—About 2 g. of the tincture are mixed with a few c.c. of chloroform, about 100 g. of water, 5 c.c. of 25% hydrochloric acid solution, and 20 c.c. of 0.1% potassium bichromate solution. After keeping for 2 min., 15 c.c. of alcohol are added and the mixture is kept for 15 min. The iodine liberated by the bichromate is titrated with 0.1N-sodium thiosulphate.

E. H. SHARPLES.

Determination of iron in Basham's mixture. J. C. KRANTZ (Amer. J. Pharm., 1930, 102, 220—221).—The iron is determined gravimetrically, after dilution and precipitation with ammonia.

H. E. F. NOTTON.

Determination of nicotine in tobacco and [tobacco] smoke. A. HEIDUSCHKA (Pharm. Zentr., 1930, 71, 305—306).—A discussion of Pfyl and Schmitt's method (B., 1927, 955) for the determination of nicotine, and of the necessity for determining both the dipicrate and iodo eosin values. The difficulty in obtaining nicotine-free tobacco is emphasised, and it is urged that alkaloid limits for the different varieties of tobacco (cf. B., 1930, 217) should be established.

E. H. SHARPLES.

Pharmacology of ergot: biological assay and standardisation. VI. Preparation of purified fluid extract of ergot: pharmacological studies. M. R. THOMPSON (J. Amer. Pharm. Assoc., 1930, 19, 221—228).—Intravenous injection of official fluid extract of ergot causes in dogs an initial decrease, due to non-specific amines, followed by an increase in blood pressure. When the de-fatted ergot is percolated at a low temperature with 5% sodium hydrogen carbonate and subsequently with water until all the amines are removed,

only about 20% of the alkaloids is dissolved. The extract prepared in the usual way from the residue produces a persistent rise in blood pressure and is therapeutically equivalent to the normal extract.

H. E. F. NOTTON.

Extract of witch hazel. S. L. HILTON (J. Amer. Pharm. Assoc., 1930, 19, 232—235).—The resorcinol-sulphuric acid test for formaldehyde is not applicable to the extract, since a positive reaction is given by pinene, a normal constituent.

H. E. F. NOTTON.

Jalap tuber and scammony root. W. PEYER and K. ROSENTHAL (Apoth.-Ztg., 1929, 44, 1329—1332; Chem. Zentr., 1930, i, 102).—The characteristics of the drugs, and of their resins and glucosides, are described.

A. A. ELDRIDGE.

Sixth Report of the Essential Oil Sub-Committee to the Standing Committee on Uniformity of Analytical Methods. Determination of citral in lemon oil (Analyst, 1930, 55, 109—110).—The method recommended, and given in detail, requires 10 g. of oil to which 0.5N-hydroxylamine hydrochloride with methyl-orange indicator (0.2% in 60% alcohol) is added and titrated with 0.5N-alcoholic potash: the completed titration liquid with a slight excess of potash is used as a colour standard for the end-point of a subsequent titration. A correction factor is necessary as the end-point of the titration occurs at a p_H different from that of neutral hydroxylamine hydrochloride. (Cf. B., 1929, 622.)

D. G. HEWER.

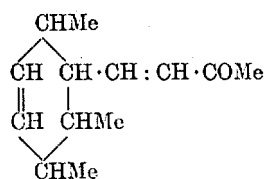
Citrus by-products research: orange oil. F. J. DE VILLIERS (Farming in S. Africa, Jan., 1930; S. Afr. Dept. Agric., 1930, Reprint 9, Series 105, 5 pp.).—Orange oils from 7 typical samples of S. African Valencia and Navel oranges have characteristics falling within the following limits: d_{20}^{20} 0.8465—0.8493, n_D^{20} 1.4723—1.4732, α_D^{20} +107° 48'—114° 54', b.p. 174.6—174.8°/760 mm., evaporation residue 4.35—5.47%, citral 1.7—2.31%, iodine value 302—314, Reichert-Meissl value 0.3—0.7, Polenske value 0.5—1.0, free alcohols 1.4—1.8%, total alcohols 2.6—3.3%, acid value 1.1—1.7, ester value 4.2—6.5, saponif. value 5.3—8.1, aldehydes 1.7—2.3%, and the yields of oil are (on the peel) 0.93—1.72%. These constants compare favourably with four other commercial oils examined, including one prepared from Italian Navels by the same method. The S. African oils seemed to have a more penetrating and pleasing aroma. Oranges should not be stored for longer than 2 months before extracting the oil, otherwise the resultant oil has higher density, refractive index, iodine and saponification values, and a lower aldehyde content. Unless the oil is stored in a cool, dark place it deteriorates and at the same time the refractive index, saponif. value, and total alcohol content increase and the rotary power and iodine value decrease.

E. H. SHARPLES.

Drugs containing phloroglucotannides or similar tanning substances; red coloration caused in the lignified elements by concentrated acids. E. LORTZ (Apoth.-Ztg., 1929, 44, 1342—1345; Chem. Zentr., 1930, i, 102).

PATENTS.

Manufacture of ketones [perfumes]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 325,669, 28.1.29).—Ketones possessing floral odours are obtained by condensing a methyl ketone with a Δ^7 -hydro-aromatic aldehyde or an *endo*-derivative thereof (cf. B.P. 300,130; B., 1930, 549), other than the aldehyde obtained from $\alpha\alpha$ -dimethylbutadiene and crotonaldehyde. Acetone is condensed, *e.g.*, with the trimethyltetrahydrobenzaldehyde prepared from $\alpha\gamma$ -dimethylbutadiene and crotonaldehyde to give an oil, b.p. 256° or 108°/4 mm., with an intense odour of violets. Acetone and the product from $\alpha\gamma$ -dimethylbutadiene and acraldehyde give a rose-scented oil, b.p. 102—103°/4 mm. Other scented oils are obtained by condensing acetone with: butadiene-crotonaldehyde (b.p. 122—123°/15 mm.); $\alpha\delta$ -dimethylbutadiene-crotonaldehyde (b.p. 133—133.5°/12.5 mm.; annexed formula); Δ^1 -*cyclo*-



hexadiene-crotonaldehyde (b.p. 131—134°/11—12 mm.); Δ^1 -*cyclo*hexadiene - acraldehyde (b.p. 134—136°/15—16 mm.); $\beta\gamma$ -dimethylbutadiene-acraldehyde (b.p. 135—137°/11—12 mm.); $\beta\gamma$ -dimethylbutadiene-crotonaldehyde [stereoisomerides (a) m.p. 52°, (b) b.p. 120°/4 mm.]; piperylene-crotonaldehyde (b.p. 124—125°/15 mm.); $\alpha\delta$ -dimethylbutadiene-acraldehyde (b.p. 128—129°/13 mm.); Δ^1 -*cyclo*hexadiene-acraldehyde (b.p. 122—126°/3—5 mm.); $\beta\gamma$ -dimethylbutadiene-crotonaldehyde (b.p. 176°/2 mm.). C. HOLLINS.

Preparation of ketones of the pyridine and quinoline series. A. BOEHRINGER (B.P. 311,387, 10.5.29. Ger., 10.5.28).—2-Methyl-pyridines or -quinolines are condensed with aromatic aldehydes, and the dibromides of the resulting styryl derivatives are converted into acetylenes, which by treatment with hot 65% sulphuric acid yield 2-phenacyl derivatives of the pyridine or quinoline. 2-Styrylpyridine, m.p. 175°, is thus converted by way of the dibromide and α -2-pyridyl- β -phenylacetylene, b.p. 152°/1 mm., into 2-phenacylpyridine, m.p. 59°, b.p. 159°/1 mm. (*ozime*, m.p. 120°). C. HOLLINS.

Colloidal monosodium tetraiodophenolphthalein. NAT. ANILINE & CHEM. CO., INC., Assees. of E. F. ELLZEY (B.P. 304,589, 31.12.28. U.S., 21.1.28).—For rendering the gall bladder opaque to X-rays the monosodium salt of tetraiodophenolphthalein is prepared in colloidal form by adding citric acid to the aqueous disodium salt in presence of gelatin or other suitable protective colloid. C. HOLLINS.

Manufacture of organic stibinic acids and salts thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,996, 15.1.29).—The nuclear amino-group in aminoarylcarbamides is diazotised and converted by the usual methods into a stibinic acid group. The stibinic acids from *p*-aminophenylcarbamide and *o*-chloro-*p*-aminophenylcarbamide are described. C. HOLLINS.

Manufacture of solutions of cinchona alkaloids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P.

327,428, 3.1.29).—Non-irritant solutions up to 40% concentration are obtained by mixing together water, sarcosine anhydride, and a cinchona alkaloid (quinine, dihydroquinine) in the form of a neutral salt. A sarcosine homologue, *e.g.*, 1 : 4-diethyl-2 : 5-diketopiperazine, may be used. C. HOLLINS.

Preparations for immunising against diphtheria. E. LOEWENSTEIN (B.P. 314,854, 18.4.29. Austr., 3.7.28).—Formaldehyde sufficient to give a 0.6% concentration is added to a 10 days' whole-culture broth of diphtheria bacilli, and after 40 days' incubation at 38° the whole is concentrated at 40° in a vacuum to 5—7% of the original volume and made neutral or slightly alkaline with ammonia, whereby the excess aldehyde is converted into hexamethylenetetramine. The resulting salve is applied by simple inunction. C. HOLLINS.

Manufacture of therapeutic substances from the pituitary gland. WELLCOME FOUNDATION, LTD., S. SMITH, and V. J. WARD (B.P. 328,347, 5.2.29).—The posterior lobe of the pituitary gland, or the crude principle obtained by precipitation with ammonium sulphate of the aqueous extract of the gland, is treated with liquefied phenol or cresol or alcoholic solutions thereof, which extract the active constituents. From these solutions the active material can be precipitated by addition of ether and light petroleum mixture. The pressor and oxytocic principles can be separated by redissolving the precipitate in the phenolic solvent followed by addition of ether. The precipitate so obtained contains mainly the pressor constituent; the oxytocic principle can be obtained from the filtrate by precipitation with light petroleum. If an aqueous pituitary extract is concentrated and treated with picric, picrolonic, or flavianic acids, a precipitate is formed from which the protein matter can be separated by known methods. It can be dissolved in phenol and treated as above. E. H. SHARPLES.

[Dental] pestles and mortars. DENTAL MANUFG. CO., LTD., and L. H. NEWBALD (B.P. 328,801, 6.5.29).

Esters of dihydric alcohols as perfumes (B.P. 327,705). **Hydroxycarboxylic acids of carbazole** (B.P. 303,901). **Substituted quinazolines** (B.P. 327,450).—See III. **Articles resistant to wear etc.** (B.P. 307,011).—See X.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Determination of silver in photographic emulsions. J. P. LAWRIE (J.S.C.I., 1930, 49, 28 r).—Four methods of determining silver in coated photographic emulsion are critically discussed, and a table is given showing three of these methods (excluding the assay) applied to a solution containing a known amount of silver. The cyanometric method of Eggert is shown to give consistent results; the author advises, contrary to the experience of other investigators, using the maximum amount of ammonia consistent with accuracy, *i.e.*, not more than 8% of the total bulk of reacting liquids.

Detection of traces of gold chloride in photographic salts. L. SETTIMI (Giorn. Chim. Ind. Appl., 1930, 12, 118—119).—This method is based on the solubility of gold salts, and the insolubility of all other salts used in photography, in alcohol. After being heated for 2 hrs. in an oven at 100—125°, 50 or 100 g. of the material are mixed with 100 c.c. of cold alcohol, then set aside for a short time, and the supernatant alcohol is filtered off. After treatment with a second similar quantity of alcohol, the whole is transferred to the filter. The total alcoholic solution is distilled to small volume, and this is evaporated to dryness in a porcelain dish and calcined. The residue is carefully heated over a small flame with 4 or 5 c.c. of aqua regia and afterwards evaporated to dryness, the acid residue being then dissolved in 1 or 2 c.c. of water and treated with a few drops of stannous chloride solution. In presence of gold the characteristic violet coloration appears.

T. H. POPE.

PATENTS.

Production of light-sensitive layers. KALLE & Co. A.-G. (B.P. 306,408, 18.2.29. Ger., 16.2.28).—Diazo-types are stabilised by adding to the light-sensitive layer containing the diazo compound either colloidal sulphur or a derivative of bivalent sulphur. Thiocarbamide is used in the examples with diazotised 1:2:4-aminonaphtholsulphonic acid or 5-ethylamino- or 4-dimethylamino-*o*-toluidine and phloroglucinol and the usual acidic ingredients.

C. HOLLINS.

Printing photographic gelatin layers. I. G. FARBENIND. A.-G. (B.P. 308,693, 26.3.29. Ger., 26.3.28).—Films are marked with serial numbers, maker's name, etc. by printing with a concentrated solution of dye in an organic solvent (glycol monomethyl or monopropyl ether) to which an equal quantity of water has been added. The water may contain a second dye in solution. A suitable dye is Direct deep black EW extra in glycol monomethyl ether, with or without addition of Magenta.

C. HOLLINS.

Development of diazo types. KALLE & Co. A.-G. (B.P. 308,653, 25.3.29. Ger., 24.3.28).—Borax, disodium phosphate, or an alkali bicarbonate may be used in place of caustic soda or ammonia in the preparations of the alkaline solutions of the coupling agents for the development of diazo-types, and afford much more stable solutions; good results, however, are obtained only in conjunction with those diazo compounds which unite rapidly with the coupling agent, *e.g.*, diazotised aminodiphenylamine, aminocarbazole, dianisidine, and their derivatives.

J. W. GLASSETT.

Manufacture of light-sensitive layers. M. P. SCHMIDT and W. KRIEGER, Assrs. to KALLE & Co. A.-G. (U.S.P. 1,758,676, 13.5.30. Appl., 12.7.28. Ger., 21.7.27).—See B.P. 294,247; B., 1929, 1033.

Production of pictures consisting of dyes in photographic manner. F. LIERG (U.S.P. 1,758,572, 13.5.30. Appl., 15.8.28. Austr., 18.10.27).—See B.P. 298,979; B., 1930, 487.

Development of photographic materials and

developer therefor. G. REDDELEIN and W. MÜLLER, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,758,762 and 1,758,892, 13.5.30. Appl., [A] 19.7.28, [B] 2.11.27. Ger., [A] 18.8.27, [B] 19.11.26).—See B.P. 295,939 and 280,873; B., 1930, 395; 1929, 38.

Photographic half-tone printing processes. V. C. ERNST (B.P. 326,519, 11.10.28).

[Tri-packs for] colour photography. A. B. KLEIN, and COLOUR SNAPSHOTS (1928), LTD. (B.P. 326,559, 20.11.28).

[Embossing roller for lenticular] films for colour photography. KODAK, LTD., Assecs. of O. WITTEL (B.P. 329,214, 27.6.29. U.S., 29.10.28).

[Two-ply] photographic films or plates. KODAK, LTD., and J. H. LAWRENCE (B.P. 328,762, 8.4.29).

Artificial foils etc. (B.P. 327,164).—See V.

XXII.—EXPLOSIVES; MATCHES.

Thermodynamic treatment of explosive reactions. III. A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 97—101, 144—148; cf. B., 1929, 379).—The explosion temperature, heat of explosion, composition and volume of the explosion gases, and the value of *F* in Abel's formula have been calculated for nitroglycerin explosives containing insufficient oxygen to ensure complete oxidation to carbon dioxide and water, but sufficient to convert the carbon wholly into gases. Results are given for rifle cordite M.D., cordite Mark I containing collodion cotton, Maxim ordnance powder, English ballistite, prismatic powder, English blank powder, nitropentaerythritol, Italian filit powder, and for a powder differing from the last-named only in the omission of the diphenylamine. Increase of the efficiency of a powder is obtained by increasing the oxygen content, lowering the heat of formation, and securing as high a value as possible for the ratio *b*:*a* in the formula $C_aH_bO_cN_d$. The oxidation of iron by explosion gases is discussed; it may be ascertained from a consideration of the thermodynamic reactions.

W. J. WRIGHT.

Apparatus for the 75° stability test for smokeless powders and nitrocellulose. (FRH.) VON MEERSCHIEDT-HÜLLESSEM (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 141—143).—In an improved form of the Lenze and Pleus apparatus (B., 1920, 281 A) a double-walled oven is employed, the space between the walls being filled with kieselguhr. The burner is almost entirely outside the oven, which prevents the blowing back of the flame due to the burner becoming hot. Instead of carbon tetrachloride, which is liable to attack the bath, a mixture of methyl alcohol and water (2:1) is used, and in order to ensure uniform heating the bath containing it is surrounded by a water-bath.

W. J. WRIGHT.

Explosion limits of gases. LÖFFLER.—See II.

PATENTS.

Blasting cartridges. D. HODGE (B.P. 328,716—7, [A, B] 20.2.29. Addn. [A] to B.P. 320,226).

XXIII.—SANITATION; WATER PURIFICATION.

Application of respiratory devices, especially carbon monoxide filters, in mines. G. RYBA, SEN. (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 128—130).—Modern types of carbon monoxide filters are efficient even in atmospheres containing 6% CO, provided that the oxygen content is 21%. After mine fires and explosions, however, this amount of oxygen is seldom present; it is usually below 15%. The lack of oxygen in the inspired air therefore constitutes an actual risk to the worker, and the use of these carbon monoxide filters in mines is condemned by various authorities.

W. J. WRIGHT.

Absorption of mercury and mercuric chloride vapours by wood charcoal. M. A. RAKUSIN (Münch. Med. Woch., 1928, 75, 1881—1882; Chem. Zentr., 1930, i, 115).—The volatility of mercury salts at the ordinary temperature is small; mercury vapour in the factory or laboratory atmosphere is more toxic. The toxic substances may be removed with wood charcoal.

A. A. ELDRIDGE.

Initial operation of Imhoff tank and contact beds at Blacksburg, Va. F. H. FISH (Ind. Eng. Chem., 1930, 22, 511—513).—This sewage-disposal plant, which consists of an Imhoff tank, four contact beds, three final settling tanks, and three glass-covered sludge beds, was designed to deal with the sewage from a population of 5000. It was placed in commission in August, 1928, and during 1929 dealt with 316,000 gals. of sewage per day. The gas from the Imhoff tank is collected in cast-iron bells sealed with road tar. It has a calorific value of 610 B.Th.U. and contains 16% CO₂. Difficulties due to foaming and the presence of a 10-ft. layer of scum have been overcome and an odourless final sludge is being produced which dries readily and contains 2.5% N. Analysis of crude sewage, effluents, and water supply are given.

C. JEPSON.

Relation of direct *B. coli* and *B. aerogenes* counts to sources of [faecal] pollution. F. O. TONNEY and R. E. NOBLE (J. Amer. Water Works Assoc., 1930, 22, 488—500).—The separate *B. coli* and *B. aerogenes* indices obtained by direct plating of human and animal faeces, soil, decaying wood, etc., on a cyanide-citrate medium (cf. B., 1928, 318) confirm the general opinion that *B. coli* is obtained exclusively from faeces, and that *B. aerogenes*, though present to only a small extent in faeces, is widely distributed in nature. The ratios in which they are present in water is a valuable guide to the possibility of dangerous pollution. The presence of *B. aerogenes* apart from *B. coli* suggests extraneous contamination of non-faecal origin; a considerable excess of *B. aerogenes* over *B. coli* points to surface contamination rather than to direct faecal pollution, though when *B. coli* is present the possibility of such pollution must not be overlooked. The use of a preliminary enrichment broth which may vary the proportions in which the organisms were originally present is deprecated and the advantage of direct plating is emphasised.

C. JEPSON.

Determination of p_H of sewage sludge. G. M. FAIR

and E. W. MOORE (Sewage Works J., 1930, 2, 1—7).—For dilutions not greater than 1 in 20 the colorimetric method is satisfactory unless the p_H has been appreciably changed by addition of an acid or a base.

CHEMICAL ABSTRACTS.

[Disinfecting action of] *N*-chlorosuccinimide. ANON. (U.S. Naval Med. Bull., 1929, 27, 235).—For the disinfection of water in small quantities, *N*-chlorosuccinimide is more efficient than bleaching powder, and is less affected than the latter by organic matter.

CHEMICAL ABSTRACTS.

Purification of waste waters from sugar-beet factories. J. H. GARNER and J. M. WISHART (West Riding Yorks. Riv. Bd. 1st Suppl. Rep.).—The methods used are described. The raw and treated wastes contained, respectively, 950—4080 and 80—280 pts. of suspended matter in 10⁶. The corresponding values for oxygen absorbed were: 53.7—175.4 and 9.7—17.1.

CHEMICAL ABSTRACTS.

Sterilisation of sea-water by means of chlorine. D. R. WOOD and E. T. ILLING (Analyst, 1930, 55, 125—126).—Bromine was found to be at least as efficient as its equivalent of chlorine for sterilisation of sea-water, and was not perceptibly more objectionable in taste and in its effect on the eyes.

D. G. HEWER.

Manganese dioxide in the aqueduct of the Catskill water supply. F. E. HALE (J. Amer. Water Works' Assoc., 1930, 22, 462—469).—Manganese dioxide was found to be present in large amounts in a slimy deposit, up to 1" thick, on the walls of an aqueduct forming part of the water supply system of New York City. Following the addition of chlorine to the water before it passes through the aqueduct, it was observed that the formation of new deposits ceased along the first 37 miles of its length and that old deposits became detached. There was no evidence of microscopical plant life, or organic structure, or of specific bacteria and moulds capable of causing the deposition of manganese. The action of the chlorine is considered to be a chemical one, and not due to a sterilising effect, as an alkaline reaction produced by dissolution of cement from the walls (which were observed to be roughened) would be reduced thereby, with a corresponding reduction in the amount of manganese precipitated.

C. JEPSON.

Lactose-broth fermenters [from filtered and chlorinated water]. H. E. JORDAN (J. Amer. Water Works' Assoc., 1930, 22, 544—546).—The paper by Spray and Laux (cf. B., 1930, 488) on a peculiar lactose-fermenting anaerobe is discussed. It is suggested that the results obtained may be due to two or more organisms acting symbiotically, and that pre- as well as post-chlorination, adjusted on a definite "excess" basis, would probably prove effective in removing the source of disturbing laboratory results.

C. JEPSON.

PATENTS.

Substituted quinazolines (B.P. 327,450).—See III. **Diazonium fluorosulphonates (B.P. 303,527).**—See IV.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 18 and 25, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat transmission. III. Flow of fluids at low velocities. A. P. COLBURN and O. A. HOUGEN (Ind. Eng. Chem., 1930, 22, 534—539).—When heat enters a slowly moving stream of liquid, convection currents are established and the fluid is defined as moving in thermal flow. As the effect of convection increases, "transitional" and then "turbulent" flow results. For flow of fluid vertically at low velocities heat-transmission coefficients are not affected by the velocity of the stream, but are influenced considerably by the temperature fall, viscosity, density, and coefficient of expansion of the fluid. This type of flow has been designated as "thermal turbulent." Empirical equations for the upward and downward vertical flow of water and general equations are deduced.

C. A. KING.

Fuel economisers: their construction, materials, and recent developments. O. KUBALEK (J. Inst. Fuel, 1930, 3, 264—278).—The development of fuel economisers is outlined and the factors influencing economy are discussed. It is shown that high gas velocities, with suitable other conditions, tend to higher heat transference. It is found that gilled tubes with gills 1 in. high and of root thickness $\frac{1}{4}$ — $\frac{1}{2}$ in. give heat transference per sq. ft. of external surface equal to that of plain tubes, but tend to accumulate external deposit more readily than plain tubes. The adoption of higher gas velocities lowers the tendency to deposition. A method is outlined for the solution of problems relating to heating surface and temperature changes in economisers. The specifications of the constructional material are discussed.

H. E. BLAYDEN.

Chemical engineering memoranda. XI. Coils for steam-heated stills. C. H. BUTCHER (Ind. Chemist, 1930, 6, 199—200).—An alcohol-rectification still of capacity 10 gals. of product per hr. may require the evaporation of 50 gals. of alcohol-water mixture per hr. The total heat requirements, allowing for 5% radiation losses, will be 240,000 B.Th.U./hr. This can be supplied by 248 lb. of steam per hr. at a pressure of 5 lb. A table of steam coils capable of giving the necessary heat transmission is given.

C. IRWIN.

Maintenance and working of simple types of autoclaves. A. W. C. HARRISON (Ind. Chemist, 1930, 6, 185—188).—Autoclaves may be classified as of the jacketed, plain, or revolving-drum type. The last-mentioned are used for the treatment of solids with gases under pressure and require special design for even heat distribution. Heating by steam or oil jackets

is essential where the inside of the autoclave is enamelled or homogeneously lead-lined, but in many cases, with care in design, good results can be obtained from the simpler unjacketed type. Lengthy processes require larger units. If either the raw material or the product is difficult to keep in suspension the autoclave should be shallow. In any case the diameter should not exceed 3 ft. 6 in., owing to the stress on the lid joints. For higher temperatures and pressures cast steel rather than cast iron is desirable. A number of practical details in design are given, and a method of installing is described. A leather belt or chain drive is preferred. Types of autoclave suitable for use in the production of dye intermediates are indicated.

C. IRWIN.

Conduction of heat. G. GREEN (Phil. Mag., 1930, [vii], 9, 241—260).—The methods of treating problems in the conduction of heat, and the results obtained therefrom, previously described (*ibid.*, 1927, [vii], 7, 784; 1928, [vii], 8, 701) are applied to the solution of definite problems.

A. E. MITCHELL.

High-sensitivity absolute-humidity recorder. C. Z. ROSECRANS (Ind. Eng. Chem. [Anal.], 1930, 2, 129—134).—An apparatus is described in which the humidity of the air can be measured. Its mode of operation is based on the fact that the thermal conductivities of dry and moist air are different. By heating two wires, forming part of a Wheatstone bridge, one of which is surrounded by the sample of humid air, while the other is enclosed in a standard sample of air, an unbalanced condition is brought about. The arrangement which has been developed is suitable for recording humidities up to saturation within the temperature range —100° to 200°. At 25° between 0% and 10% the accuracy is $\pm 0.05\%$ R.H. A commercial modification of the apparatus which gives continuous readings is in operation, but it has not yet been applied for general plant use.

H. INGLESON.

Preparation of air of known humidity and its application to the calibration of an absolute-humidity recorder. A. C. WALKER and E. J. ERNST, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 134—138).—An apparatus is described in which air of constant humidity can be passed at a constant rate through an absolute-humidity recorder (cf. preceding abstract) over very long periods with variations of not more than 0.001% by vol. in the moisture content. The method adopted is to mix dry air in fixed proportions with nearly saturated air. In the drying of the air prior to mixing the use of magnesium perchlorate trihydrate is recommended in preference to phosphorus pentoxide, since it does not become gelatinous on absorbing relatively small amounts of water.

H. INGLESON.

* The remainder of this set of Abstracts will appear in next week's issue.

Stability of technical emulsions. R. AUERBACH (Kolloid-Z., 1930, 51, 176—179).—Observations were made on 126 emulsions of different composition, and their separation on being set aside for a period of 5 months was observed. A method for preparing the emulsions is described. Fish train-oil emulsions in water stabilised with soda were found to have a maximal stability at medium concentrations of the alkali. The optimal concentration of alkali increases with the concentration of the emulsion.
E. S. HEDGES.

Prevention of nuisance from fumes and dust from power stations. M. W. TRAVERS (J. Inst. Fuel, 1930, 3, 292—299).—The problems relating to the prevention of atmospheric pollution by fumes and dust are discussed. It is suggested that the solution of the problems depends on (a) pretreatment of fuel for ash removal, (b) the design of coal-burning appliances for burning coal in the minimum and controllable amount of air.
H. E. BLAYDEN.

Coal burning and steam production. WYLDE. **Submerged combustion.** HAMMOND.—See II. **Organic fluorides as refrigerants.** MIDGLEY and HENNE.—See III. **Continuous methods of causticising.** STEWART.—See VII. **Consistometer.** BULKLEY and BITNER.—See XII. **Equation of plastic flow.** WILLIAMSON.—See XIII.

See also A., June, 688, **Collodion films and filtration** (ELFORD). 690, **Sedimentry** (BLOM). 717, **Photothermometry** (HENCKY and NEUBERT). 729, **Thermoregulators** (WING.).

PATENTS.

Heat-treatment furnaces [for enamel ware etc.]. CARBORUNDUM CO., LTD. (B.P. 308,613, 22.3.29. U.S., 23.3.28).—A furnace suitable for coating enamel ware comprises preheating and firing zones within one furnace structure and a drying zone preferably in another structure, but so situated that the goods may be conveyed through it by the return run of the conveyor from the other chambers. The goods are fired by heat from radiating combustion chambers in the sides and bottom of the furnace, separated from the goods by walls of silicon carbide or other good conductor; the flow of gases therefrom is preferably under the preheating chamber and then to the dryer.
B. M. VENABLES.

Drying ovens. S. L. GROOM (B.P. 328,624, 24.1.29).—A conveyor for plate-like objects which can only be handled on one side is described; provision is made for a 180° horizontal turn to be made so that both runs of the conveyor may be used for the same objects and the dryer thus made more compact.
B. M. VENABLES.

Supplying heat required for effecting chemical reactions. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,635, 24.10.28).—Substances are liquefied and/or kept liquid by secondary electric currents in the mass of the material which are induced from a primary circuit through a non-conducting envelope. Metallurgical processes and glass manufacture are excluded, but good conducting masses (metals, carbon, etc.) may be embedded in the mass and lifted out, if desired, when the melting is complete. [Stat. ref.] B. M. VENABLES.

Dehydration [of solids]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,032, 15.2.29).—Solid substances containing water as water of hydration or crystallisation, or hydrogels or similar substances containing absorbed or adsorbed water, or solid substances with adhering water, are dehydrated by treatment with gaseous ammonia substantially at ordinary temperature, or while cooling. If necessary, the dehydrated product is heated in order to expel any ammonia that may have combined with it.
S. K. TWEEDY.

Heating of liquids. L. L. RANSOM (B.P. 328,548, 24.12.28).—A tank system for the supply of liquid (e.g., hot water to laundries) intermittently and at constant temperature while taking a steady supply of heat is described. [Stat. ref.]
B. M. VENABLES.

Evaporation of salt, sugar, and other solutions and the generation of steam. W. RUSIECKI (B.P. 304,670, 23.1.29).—Several forms of evaporator are claimed in which ebullition takes place in the upper part of a column of liquid which is tall enough to prevent ebullition in the heating zone in the lower part of the column. Forced circulation is not used.
B. M. VENABLES.

Apparatus for the transfer of heat. F. HEPWORTH (B.P. 328,718, 21.2.29).—A heat exchanger of the bundle of tubes type is constructed so that the bundle may be removed transversely without disturbing the connexions for either fluid. The packing round the edges of the tube plates permits expansion, and is double with a vent to atmosphere, so that any leakage of one liquid is prevented from entering the other.
B. M. VENABLES.

Heat exchangers. W. H. OWEN (B.P. 328,230, 22.1.29).—A tubular heat exchanger is constructed in such a form that a number of them may be attached to the wall of an existing flue in tiers or ranks.
B. M. VENABLES.

Heat-exchange tubes. O. Y. INRAY. From INTERNATIONAL COMBUSTION ENG. CORP. (B.P. 328,557, 24.1.29).—The casting of tubes with heat-receiving extensions on one side, suitable for the walls of boiler combustion spaces, is described.
B. M. VENABLES.

Increasing the heat exchange between gases and solids or molten materials. C. STILL (B.P. 328,640, 27.12.28).—In view of the fact that at temperatures above 500° the effect of radiation becomes important compared with convection, and that above 1000° it is of dominating importance, also of the fact that the relative radiating and absorbing powers of carbon dioxide and water vapour are high and of nitrogen and oxygen low, therefore to the air entering a heat exchanger, e.g., a regenerator for coke ovens, is added a small quantity of highly concentrated carbon dioxide. If the proportion of carbon dioxide is raised from the normal 0.03 to 1% and of water from 1 to 3%, the exchange of heat between the chequerwork and the gas at the hot end where the temperature may be 1200—1300° is raised threefold. The addition must not be overdone or the bad effects of dilution will cancel the benefits. Rich carbon dioxide may often be obtained from an adjacent by-product plant.
B. M. VENABLES.

Regulating and maintaining heat-transfer [e.g., during vulcanisation]. GEN. CARBONIC CO. (B.P. 305,577, 24.1.29. U.S., 7.2.28).—The heat for vulcanisation (or other similar process) is supplied by a mixture of carbon dioxide (or other inert gas) and steam (or other heating fluid); the temperature attained corresponds to the partial pressure of the latter and will remain automatically constant if a definite mass of the former be first admitted, as determined by the pressure when cold. Examples are given of the use of the process for the vulcanisation of rubber tubes at 127° where internal pressures of 200 and of 100 lb./in.² are employed, and steam is assumed to be available at those pressures. B. M. VENABLES.

Cooling or freezing cream or other liquids. C. W. VOGT (B.P. 328,434, 23.4.29).—The liquid is sprayed upon the surface of a conical lump of refrigerant that evaporates without melting, e.g., solid carbon dioxide, the freezing being effected under a minor pressure in a closed chamber so that the atmosphere also becomes chilled and will be entrained in the solid particles produced. The major pressure to operate the spraying nozzle is generated by the evaporation of another mass of carbon dioxide above the fluid contained in a feed chamber, and the valve which permits fluid to pass through the spray is operated intermittently by a device similar to a windscreen wiper, which in turn is operated by gas from the refrigerating chamber. The intermittent spraying is stated to prevent the formation of a film of ice upon the refrigerant. B. M. VENABLES.

Refrigerants for use in vacuum refrigerating machines. DEUTS. GASLÜHLICHT-AUER-GES.M.B.H. (B.P. 310,546, 27.4.29. Ger., 27.4.28. Addn. to B.P. 310,474).—The refrigerants claimed are such as will dissolve in the motive liquid (e.g., water) to a sufficient extent to lower the f.p. to about -10°. Examples are: ethyl ether or chloride, pentane. [Staf. ref.] B. M. VENABLES.

Cooling of grinding mills. M. TRESCHOW (B.P. 328,634, 1.2.29).—The material is caused to come in contact, inside or outside the mill, with coils or jackets cooled by a fluid which is under suction to prevent harm due to any leakage. The cooling device rotates with the mill. B. M. VENABLES.

Mixing apparatus. A. B. and C. R. SMITH (B.P. 328,401, 22.3.29).—A pan which has a stirring shaft going through the bottom is provided with an upturned lip round the central hole, and any material which gets over the lip falls on to an umbrella-like guard forming the top of the chamber containing the gearing. The top of the non-rotating umbrella is spigoted into a skirt formed as a downward extension of the rotating boss carrying the stirrer. B. M. VENABLES.

Pneumatic separators. ALPINE A.-G. EISENGIESSEREI U. MASCHINENFABR., Assees. of ALPINE MASCHINEN-A.-G., and A. KUHR (B.P. 309,378, 8.4.29. Ger., 7.4.28).—In a separator of the type in which the fine dust is drawn upwards through a fan, the coarse being left behind, a supplementary separation of grits is effected after passing the fan in an annular space between two walls surrounding the fan. B. M. VENABLES.

Separation of materials [by air]. I. L. BRAMWELL, C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 328,283, 23.1.29).—The material (coal, ores, etc.) is allowed to slide down an adjustable staircase having the treads sloping sufficiently to prevent undue accumulation of material on them. A slot is left under the edge of each thread between it and the riser, and a current of air carries the finer material through these slots. B. M. VENABLES.

Centrifugal dust separators or extractors. H. SIMON, LTD., and G. WATTS (B.P. 328,652—3, 2.2.29).—The devices described comprise a number of small, conical cyclones situated in the middle one of three parallel compartments, the middle or inlet chamber serving also as a preliminary dust settler. The outer compartments receive cleaned air and concentrated dust, respectively, and it is necessary for satisfactory operation for the air pressure to be kept equal in these two; to effect this the ratio of the diameters of the axial air and dust outlets of the cyclones is 10 : 7 when the cyclones are 6—12 in. in diam. at the large end. In (A) the cyclones have vertical axes and the dust is removed by reciprocating grids in the chambers; in (B) the axes are horizontal and the dust falls out by gravity. B. M. VENABLES.

Separation of liquids by distillation. DISTILLATION DYNAMIQUE (B.P. 308,736, 27.3.29. Fr., 27.3.28).—The vapour from the top of a rectifying column is compressed, then condensed in a coil at the bottom of the column, thus supplying most of the heat for operation. Supplementary heat and regulation of the pressure are provided for by a minor supply of heat at the bottom or of cold at the top from external sources. Permanent gases are separated from the condensate in a settling chamber with regulating float and are withdrawn through a condenser, any condensed vapour from which and part also of the main condensate being returned to the tower as reflux liquid. When several constituents are to be separated a number of towers operating at different pressures are connected in series. B. M. VENABLES.

[Tunnel] kilns. H. M. ROBERTSON (B.P. 306,813, 23.1.29. U.S., 25.2.28).—See U.S.P. 1,737,540; B., 1930, 222.

Mercury boiler. A. J. NERAD, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,759,133, 20.5.30. Appl., 11.7.27).—See B.P. 293,756; B., 1929, 495.

Pulverising apparatus. R. S. RILEY and O. CRAIG, Assrs. to RILEY STOKER CORP. (U.S.P. 1,759,134, 20.5.30. Appl., 7.11.25).—See B.P. 260,952; B., 1928, 72.

Bearing-protecting devices for centrifugal separators. RAMESOHL & SCHMIDT A.-G., and C. SCHMITZ (B.P. 329,548, 20.6.29).

Mixing valves for liquids. F. P. BARBER (B.P. 329,839, 18.5.29).

Steam and gas mixture of high pressure (B.P. 328,339).—See II. Tunnel kilns (B.P. 311,316).—See VIII. Tunnel kilns (B.P. 328,265).—See IX. Porous bodies, filters, etc. (B.P. 328,273—4).—See XIV. Solidified globules from gelatinous substances (U.S.P. 1,746,543).—See XV.

II.—FUEL; GAS; TAR; MINERAL OILS.

Fuels of Italy. M. G. LEVI (Chim. et Ind., 1930, 23, 811—824).—An outline is given of the work that is being carried out at the Institute of Industrial Chemistry at Milan on the utilisation of Italy's fuel resources. This includes a survey of the coal fields, and a study of the products obtained by the low-temperature carbonisation and by the hydrogenation of the different varieties of coal available. Some account is given of the occurrence and utilisation of petroleum, natural gas, and asphaltic and bituminous rocks in Italy. A. B. MANNING.

X-Ray stereoscopic examination of coal. II. A. N. WILSON (J. Inst. Fuel, 1930, 3, 342—346; cf. B., 1930, 540).—X-Ray stereoscopic methods applied to the localisation and detection of mineral layers and particles in coal are described. A double exposure is made with the X-ray bulb in two positions separated by a distance, D , symmetrical about the path of the central ray (which is made to pass through the centre of the fixed specimen and film) and on a line parallel to the film. If d is the distance between the two resultant images of any particle and H is the height of the bulb above the film, then the height of the particle above the film is $h = Hd/(D-d)$. The use of stereoradiographs to indicate the probable manner of fracture of the coal due to mechanical treatment such as screening or dry-cleaning is discussed. H. E. BLAYDEN.

Developments and problems in coal cleaning. F. S. SINNATT and D. T. DAVIES (J. Inst. Fuel, 1930, 3, 326—336).—Statistics are given showing the development in coal cleaning by washing, dry-cleaning, and froth-flotation methods during 1927—1928 in Great Britain. There has been a rapid growth of dry-cleaning during the last seven years. Lessing's process (B., 1927, 866; 1928, 356) can now be applied for the separation of middlings of greater sp. gr. than the separating liquid, and can be modified for the efficient treatment of moist coals. Experiments have been made on the automatic control of the dirt bed in a British Baum washer capable of treating $2\frac{1}{2}$ tons of coal per hr. The optimum conditions of the dirt bed could be maintained for long periods despite alterations in feed and coal of varying dirt content. Slack (21.3% of ash; 29.5% of sink in liquid of d 1.45) gave 3.7% of sink material in the clean coal and a loss of 0.6% of float material in the dirt. It is suggested that attention be paid to the flotation aspect of jig-washing and the size distribution of coal in the feed. The treatment and utilisation of dust are discussed with reference to blending and pulverised fuel combustion. It has been found that addition of reagents (lime, aluminium sulphate, sodium aluminate, sodium silicate, casein, glue, gelatin, or albumin) to washery waters facilitates settlement. Gelatin was very efficient and 0.2 lb. per ton of recovered solid was required. It was found possible to control flocculation and fractionally to precipitate the coal and dirt. Problems arising from the variation of coal properties in a given seam are discussed with reference to the effects of screening and cleaning on the properties of the cleaned coal and its ash. Samples for float-and-sink tests should not be crushed before examination. If the bulk sample is a screened grade

it may be reduced after mixing, but not crushed. The weights recommended for samples are 50, 30, 10, 1 lb. for maximum particle size of $1\frac{1}{2}$, $\frac{3}{4}$, $\frac{3}{8}$, $\frac{1}{8}$ in., respectively. Run-of-mine coal or screened slack is usually separated into one or more screen grades and the appropriate quantities quoted are used for float-and-sink tests. It is considered inadvisable to carry out ash analysis on the coal after float-and-sink tests. The elimination of sulphur from coal and the recovery of pyrites, for commercial use, from the washery rejects are discussed.

H. E. BLAYDEN.

Blending [of coals for coke manufacture]. C. B. MARSON (Gas World, 1930, 92, Coking Sect., 47—50).—Experiments are described in which typical Durham coking, Northumberland high-volatile "non-coking," Scottish coking, Northumberland low-volatile coking, and Cumberland coking coals have been blended, by pairs, in varying proportions, and the mixtures coked. Examination of these coals showed the following results. (i) The Scottish and Cumberland coking and Northumberland "non-coking" coals, compared with the Durham coking coals, were characterised by greater amounts of moisture, volatile matter, oxygen, and hydrogen, by smaller amounts of carbon, and by lower agglutinating values; attention is drawn to the low volatile and carbon contents of a Northumberland coking coal. (ii) When tested in the Gray-King assay at 600°, the Durham and Northumberland coking coals, compared with the other coals examined, gave greater coke and lower tar yields. Again, when the "straight" coals, together with the various blends, were coked in an experimental oven, taking a charge of 3 cwt., and with an average coking temperature of 920°, it was found that (a) the mechanical strength of a Durham coke as judged by the shatter index was considerably diminished by the addition of 40% of a Northumberland "non-coking" coal, but greater proportions of the latter did not give a proportionate lowering of the shatter index; (b) small additions of a Scottish to a Durham coking coal decreased considerably the shatter index of the Durham coke, but increasing additions effected an improvement, and the blend containing 80% of the Scottish and 20% of the Durham coal possessed a shatter index of the same order as the 100% Durham coke; (c) addition of a poor quality Cumberland coking to a Northumberland coking coal gave results similar to those of the Scottish-Durham series, an optimum blend being obtained with the 80% Cumberland-20% Northumberland blend; and (d) the blending of a second Northumberland "non-coking" coal with a Durham coking coal showed that 20% of the former may be added to the latter without causing a marked decrease in the shatter index. C. B. MARSON.

Design, construction, and operation of a modern coke-oven and by-product recovery plant, equipped for the manufacture of town gas. D. V. HOLLINGWORTH (Gas J., 1930, 190, 432—435).—The coke ovens and recovery plant at the Altham works of the Lancashire Foundry Coke Co. are described.

C. B. MARSON.

Metallurgical coke. G. T. PURVES (Fuel, 1930, 9, 229—233).—Some factors of importance in the produc-

tion of metallurgical coke are discussed. By suitable control of the quenching process the coke can be delivered at the blast furnace with an average moisture content of less than 1.5%. In general, the shatter index of the coke increases with an increase in the width of the oven up to a width of about 18 in., but decreases again if wider ovens are used. Compressing the charge lowers the shatter index. Rapid carbonising in very narrow ovens does not yield a good metallurgical coke. Fine grinding of the coal produces a marked improvement in the shatter index of the coke. In order to withstand crushing and abrading in the blast furnace a certain minimum strength is necessary. Little is known, however, of the factors which control the behaviour of the coke when it reaches the hearth, and opinion is divided as to whether a reactive or a hard-burnt coke is to be desired.

A. B. MANNING.

Determination of the reducing power [of coke etc.] by Agde and Schmitt's method. W. J. MÜLLER and W. COURARD (*Brennstoff-Chem.*, 1930, 11, 125—128; cf. Agde and Schmitt, B., 1927, 833, 834).—Some minor improvements have been made in the method, particularly in the production and measurement of the constant current of carbon dioxide and in the arrangement of the combustion tube, whereby greater accuracy has been attained. Successive determinations of the reducing power of active charcoal showed only slight variations. Larger fluctuations were observed with cokes, and the values tended to fall with continued passage of the carbon dioxide.

A. B. MANNING.

Reactivity of carbon materials. Y. OSHIMA and Y. FUKUDA (*Fuel*, 1930, 9, 200—212).—The rate of reduction of carbon dioxide by coke, charcoal, or graphite has been determined by passing a current of the gas over a sample of the material maintained at 900°, and the effect of varying the temperature, the area of the external surface of the sample, and the rate of passage of the gas has been studied. In most of the determinations the exit gas was analysed by an interferometer method. The initial temperature of reaction with coke and charcoal was dependent only on the maximum temperature of carbonisation. From a comparison of the reactivity of a cylinder of high-temperature coke with that of the same material finely powdered it is concluded that the surface layer of such a coke is very inactive compared with the underlying carbon; similar experiments with a semi-coke, however, showed this to be nearly homogeneous. The higher reactivity of charcoal as compared with coke is ascribed mainly to its larger surface area per unit volume. The reactivity of a graphite rod increased as the surface became etched by the reaction. A decrease in reactivity during the measurement may be due to loss of total carbon surface, accumulation of ash, or, in the case of a semi-coke, shrinkage of the test piece. A relationship has been deduced between the effective surface areas of two cokes (A , A') and the rates of flow of carbon dioxide (V , V') necessary to give a definite amount of carbon monoxide in the resulting gaseous product, expressed by $A' = AV'/V$. If A and V are determined for a graphite rod of known surface area, then A' for a coke can be calculated from V' .

A. B. MANNING.

Coal burning [applied to steam production]. W. D. WYLDE (*J. Inst. Fuel*, 1930, 3, 241—255).—The principles of efficient combustion of solid and pulverised fuels for steam production are discussed. In the firing of slack, preheated air and the introduction of secondary air for the combustion of volatile matter tend to give increased efficiency. A suitable method for the addition of secondary air is to pass this air through channels in the brickwork of the arch of the furnace to the position of liberation of the volatiles. Ample combustion space is necessary for efficient combustion of high-volatile coals. The interpretation of the carbon dioxide content of exit gases with respect to losses due to excess air is discussed and represented graphically. H. E. BLAYDEN.

Influence of metallic and other substances on coal during carbonisation. J. G. KING and L. T. EDGCOMBE (*Fuel*, 1930, 9, 213—218).—The influence of the addition of about 5% by vol. of various metals or compounds on the carbonisation of a medium-caking bituminous coal has been studied. The Gray-King assay apparatus (B., 1929, 308) was used. Coke, carbon, or silica had no measurable effect on the yields or quality of the products. Aluminium, chromium, or stainless steel had only a very slight effect, and would therefore be suitable metals for the construction of apparatus for the study of carbonisation reactions up to 600°. Copper, nickel, cast iron, and steel became sulphided by the hydrogen sulphide evolved. Iron and copper promoted the formation of hydrogen at the expense of the saturated hydrocarbons. Pyrites and marcasite reduced both the strength of the coke and the yields of tar and gas. Sulphided cast iron, copper sulphide, and nickel sulphide had effects similar to pyrites and marcasite. Sulphided iron reduced the tar yield by 17%.

A. B. MANNING.

Peat. III. Low-temperature carbonisation of peat. J. T. DONNELLY and J. REILLY. **IV. Low-temperature carbonisation under various conditions.** C. O'SULLIVAN and J. REILLY (*Sci. Proc. Roy. Dublin Soc.*, 1930, 19, 365—376; 441—446; cf. B., 1927, 769).—III. Samples of air-dried Irish peat were carbonised at 550° and the resulting tars examined by the method of selective solvent extraction. The carbonisations were carried out in a current of coal gas in a retort holding a charge of 500 g. and similar in design to that described in B.P. 241,659 (B., 1926, 4), but heated electrically. The tar was collected in two receivers connected by a water-cooled condenser; the first receiver, which included a glass-wool trap for retaining tar fog, was maintained at 120°, and the second at —20°. A yield of 16.64% of tar (calc. on dry material) was obtained from a black peat. The tar contained 5.4% of phenols. Higher acids of the nature of waxes were also present. The "resinols" amounted to 4.41% of the tar, and the "resinamines" to 0.88% (cf. Morgan and others, B., 1929, 156). The yields of acetic acid and ammonia (calc. as sulphate) were 22.6 lb. and 17.4 lb., respectively, per ton of air-dried peat. The yield of gas amounted to 8.61% of the dry peat, but was of poor quality. The black peat contained 10.47% of wax extractable by acetone. Carbonisation of a sample of brown peat gave similar results, but the yield of tar was somewhat smaller (14.41% on the dry peat).

IV. The addition of 11% of calcium carbonate to a sample of black peat had no effect on the yield of tar obtained by low-temperature carbonisation. Separate carbonisation of the wax, which amounted to about 10% of the peat, and of the wax-free peat, gave a total yield of 15.08% of heavy tar and 1.06% of light oil, compared with the values 13.82% and 1.27%, respectively, obtained by direct carbonisation of the original air-dried peat. More than 40% of the total tar obtained by the separate carbonisation of the constituents was derived from the wax, whilst the whole of the light oil was derived from the wax-free peat. A. B. MANNING.

Hydrogen cyanide in the gaseous products from the propagation of a zone of combustion through powdered coal. H. E. NEWALL, W. J. SKILLING, and F. S. SINNATT (*Fuel*, 1930, 9, 234—236).—A current of air was aspirated over a conical heap of powdered coal which had been lighted at the apex (cf. B., 1927, 802). Hydrogen cyanide was detected in the gaseous products of combustion of the coal, and was determined quantitatively by passing the aspirated gases through dilute aqueous potassium hydroxide and converting the potassium cyanide into Prussian-blue, the solution of which was compared with a standard. The amount of hydrogen cyanide formed varied from nil to 160 pts. per million pts. of air, the highest concentration corresponding with a total amount equal to 0.11% by wt. of the coal. Attention is directed to the possible danger from this source when fires occur in the goaf, in coal stores, etc. A. B. MANNING.

Comparison of fused silica, gold, and platinum linings for calorimeter bombs. O. K. BATES (*Ind. Eng. Chem. [Anal.]*, 1930, 2, 162—164).—The heating values of a number of different coals as determined by the Emerson bomb lined with vitreosil agreed closely with those determined when using a platinum lining. The values obtained with the bomb lined with gold backed with silver agreed satisfactorily with those found when a vitreosil lining was used, for samples of low sulphur content, but were higher than these latter when the coals had a high content of sulphur. This additional heat is evolved by reactions in which the lining plays an important part. The sulphur content of coals determined from the bomb washing was lower in all three series than the value obtained by the peroxide method, but that found when vitreosil was employed was nearest to the correct value. The deficiency in all cases may be due to the production of sulphonic acids which are not determined as barium salts in the subsequent analysis. H. INGLESON.

Modern methods of tar distillation. E. WEISE (*Petroleum*, 1930, 26, 499—504, 577—582).—A survey of processes for intermittent, semi-continuous, and continuous distillation. Representative types of plant and their mode of operation are described with the aid of diagrams. Patent references are given of a selection of modern processes not described in the text; the majority of these are concerned with continuous distillation. D. G. MURDOCH.

Fractionation by means of solvents, and chemical study, of a commercial primary tar. I. UBALDINI

(*Annali Chim. Appl.*, 1930, 20, 118—138).—Fractionation of primary tar by means of solvents is an indispensable preliminary to the chemical investigation. Ether and ordinary benzine allow of the subdivision of the tars into groups of substances with clearly-defined characteristics, and benzene and alcohol permit of further subsequent fractionation. Treatment with benzine may be used with advantage on the industrial scale to separate the complex resinous products from chemically simpler products, and may replace the initial distillation which, even when carried out in a current of steam and under reduced pressure, often gives rise to a large amount of residual pitch. T. H. POPE.

Trichloroethylene as a solvent for bituminous materials in routine analysis. J. B. HYATT (*New Zealand J. Sci. Tech.*, 1930, 11, 418—419).—Trichloroethylene is a suitable solvent, being non-inflammable and less toxic than carbon disulphide. Hydrochloric acid produced by decomposition in sunlight is easily removed by shaking with water. C. W. GIBBY.

Determination of phenol in effluent waters. H. A. J. PIETERS (*Chem. Weekblad*, 1930, 27, 334—337).—The methods of removing substances which would interfere with the phenol determination are discussed for different cases. For a phenol content of about 1 g. per litre, the bromine method, in which excess of bromine is determined iodometrically, is very accurate; for concentrations down to 0.15 g. per litre, nephelometric determination of the tribromophenol formed is most satisfactory, whilst for lower concentrations, colorimetric determination, based on the Folin and Denis reagent, is best. S. I. LEVY.

Viscosity of pitch. H. A. J. PIETERS (*Chem. Weekblad*, 1930, 27, 287—288).—A series of determinations of viscosities at various temperatures by the penetration and rate of flow methods indicates that the viscosity and flow point are closely related; it is concluded that determination of one value, preferably the flow point, sufficiently characterises the other. S. I. LEVY.

Behaviour of [wood] creosote at low temperatures. W. GRUNDMANN (*Chem.-Ztg.*, 1930, 54, 353—354).—Six's maximum and minimum thermometers, in which beech-wood creosote is used, were found to be unreliable at temperatures below -30° owing to the frequent separation of gas bubbles. A series of samples of creosote were examined and tested for viscosity at low temperatures. In most cases this rapidly increased below -20° and only mixtures of creosote and alcohol retained fluidity well. The separation of gas at low temperatures was confirmed by cooling sealed ampoules of creosote to -40° . This also occurred with alcohol mixtures. C. IRWIN.

Influence of cylinder design on pinking. G. B. MAXWELL and R. V. WHEELER (*Fuel*, 1930, 9, 225—229).—It has been suggested that "pinking" in an internal-combustion engine is due to the initiation of a stationary pressure-wave in the burnt and burning gases, followed by a shock-wave when the accelerated flame impinges on the wall of the combustion space (cf. B., 1929, 873). This hypothesis has been confirmed by experiments on

the influence of the shape and dimensions of the combustion space, and the position of the source of ignition, on the explosion of pentane-air mixtures. The formation of a stationary wave appears to depend mainly on the character of the reactions proceeding in the wake of the flame, which, with a given fuel, depends in turn on the mean flame temperature. These reactions, however, are modified by turbulence, or by the addition of traces of a "pro-knock," *e.g.*, amyl nitrite. An "anti-knock," *e.g.*, lead tetraethyl, seems to affect the establishment, not of the stationary wave, but of the subsequent shock-wave. The stationary wave is modified by the shape of the reflecting surfaces at the end of the cylinder, a concave plate being the most and a convex plate the least effective in preventing the formation of the wave. The flame vibrations and acceleration which mark the setting up of the stationary wave begin when the flame has travelled about 75% of its full distance. A restriction, *e.g.*, a wire gauze diaphragm or an annulus, introduced at this point in the cylinder either stopped or considerably diminished the pinking. A. B. MANNING.

Connexion between the I.P.T. gravity temperature-correction constants and the coefficient of expansion of petroleum oils. I. A. J. DUFF (J. Inst. Petroleum Tech., 1930, 16, 501—502).—The I.P.T. figure for gravity temperature-correction is not the correct one for use as a volumetric expansion coefficient. The real figure is obtained by dividing the sp. gr. temperature-correction figure by the sp. gr. and changing the sign. The mathematical derivation is given, and also the values for a number of sp. gr. at 60° F.

H. S. GARLICK.

Variation with temperature of the sp. heat of typical crude oils and their residues when topped to 100°. H. R. LANG and R. JESSEL (J. Inst. Petroleum Tech., 1930, 16, 476—500).—The sp. heats of a number of topped and crude oils were determined by the continuous-flow electrical method over a wide range of temperatures. With Pennsylvanian crude and topped oils there is a minimum at about 16°, the results agreeing closely below 20°. Above 25° the values for the topped oil lie on a straight line, the slope of which is 0.0095 per 1°. With Persian oil the deviation between the two curves is more marked above the minimum point (18°), owing to the greater volatility of the lighter fractions in the untopped oil. Above 25° the values lie on a straight line with a slope of 0.00089 per 1°. Below 15° no consistent results could be obtained, owing apparently to the unstable state of the wax in the oil. With Californian crude oil all values lie on a straight line the slope of which is 0.00095 per 1°. With Mid-Continent oil the sp. heat falls linearly with temperature down to 11°, with a sudden increase until 9° is reached, after which it continues to rise slowly. The curve for the topped oil is the same as with the crude oil above 24°, the slope of the straight part of the line being 0.00085 per 1°. Tables of the total heat content of the oils are given, together with a formula for calculating the sp. heats, which gives values agreeing more closely with experimental results than those of previous observers.

H. S. GARLICK.

Ageing of mineral oils. J. MARCUSSEN and W. BAUERSCHÄFER (Chem.-Ztg., 1930, 54, 401).—An

examination of the non-acidic products formed in turbine and transformer oils during long use is described. An old transformer oil freed from acidic substances (2.4%) contained 84.3% C, 12.4% H, 0.37% S, and 2.6% O. Hydroxyl and carbonyl compounds were detected and 6% of petroleum resin (mean mol. wt. 293, acetyl value 40.2) was isolated. An oil heated for a long time at 120° was transformed into a hard asphalt, m.p. 56°, containing 0.9% of acid, 4.9% of petroleum resin, and 31.2% of asphaltene. The acetyl value, 1.4, was increased to 8.0 after reduction of the carbonyl compounds; a process for this is described which gives a measure of the acidic and neutral decomposition products of an oil. E. H. SHARPLES.

Determination of the coumarone resin content of the solvent naphtha fraction. I. I. BURDA (Ukraine Chem. J., 1929, 4, [Tech.], 257—258).—To 100 c.c. of solvent naphtha in a long separating funnel 2 c.c. of concentrated sulphuric acid are added drop by drop with shaking. The acid layer is then run off and the remainder is steam-distilled, when coumarone resins remain chiefly in the residue; the residue obtained by redistilling the distillate is added to the first residue, and, after dehydration, the yield of coumarone resins is weighed. R. TRUSZKOWSKI.

Higher hydrocarbons from water-gas. BERL and JÜNGLING. **Synthetic alcohols from water-gas.** NATTA and STRADA. **Solvents from natural gas.** CLARK.—See III. **Pressure-synthesis operations.** ANON. **Synthetic ammonia.** CLAUDE.—See VII. **Determination of montan wax in beeswax.** BUCHNER.—See XII. **Coumarone resins.** BURDA.—See XIII. **Egg-killing washes.** STANILAND and others.—See XVI. **Coal mine explosives.** NAOUM and BERTHMANN.—See XXII.

See also A., June, 715, **Catalytic decomposition of methane** (FISCHER and BAHR). **Equilibrium** $6\text{CO}_2 + \text{C}_6\text{H}_6 = 12\text{CO} + 3\text{H}_2$ (BAHR). 735, **Catalytic oxidation of methane by sulphuric acid** (FISCHER and BAHR). 786, **Piperidine derivatives** (BAILEY and McELVAIN). 788, **Bases in the kerosene distillate of California petroleum** (POTH and others).

PATENTS.

Briquetting of fuel. C. A. BURNE (B.P. 328,082, 7.3.29).—Coal dust is mixed with a relatively small quantity (2—2½%) of gluten or gluten-bearing material, *e.g.*, ground rice or meal offals, and ¾—1% of sodium silicate, in the form of a dilute aqueous solution, and the mixture is briquetted. The briquettes are dried by being heated at 65° for about 6 hrs. A. B. MANNING.

Gas generators. HUMPHREYS & GLASGOW, LTD., Assees. of J. S. HAUG (B.P. 312,162, 14.5.29. U.S., 21.5.28).—A cylindrical producer is provided with feeding means which deposit the finer fuel at the periphery and the coarser fuel at the centre, the air and/or steam being admitted only to an annular zone at the bottom of the fuel bed, whence sufficient finds its way to the centre through the coarser particles to secure the desired rate of combustion there. The rotating grate has a flattened, imperforate top provided with curved blades which displace the ash outwardly towards the

peripheral part of the bottom of the fuel bed, where it is discharged.

A. B. MANNING.

Producing a steam and gas mixture of high pressure. N. A. KALABIN (B.P. 328,339, 1.2.29).—Powdered or liquid fuel is supplied through a minor chamber into a larger chamber containing water. Only enough air (under pressure) is supplied to the minor chamber to effect gasification and to cause the walls to glow, the secondary air being added to the water chamber. The incoming water may be preheated or the outgoing mixture superheated in passages surrounding the minor chamber. Means are provided for the regulation and agitation of the water.

B. M. VENABLES.

Washing cyanide compounds, ammonia, and sulphuretted hydrogen from gases. A. C. BECKER and W. BERTELSMANN (B.P. 328,738, 18.3.29).—In such washing processes in which solutions containing ferrous oxide are employed, additional ammonia, obtained by melting ammonium sulphate, is used for washing out the hydrogen sulphide. The ammonium bisulphate produced is used for treating the used washing lye.

W. J. WRIGHT.

Extraction of hydrocarbons from gases. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,008, 15.1.29).—Coal gases etc. are treated with sulphuric acid of at least 70% concentration, and preferably in two or more stages with acid of successively higher concentrations (up to 95%), and the benzene hydrocarbons are absorbed from the purified and dry gas in known manner, *e.g.*, by means of activated charcoal.

A. B. MANNING.

Production of aqueous [bitumen pitch] dispersions. L. KIRSCHBRAUN (U.S.P. 1,734,437, 5.11.29. Appl., 22.7.27).—Solid or semi-solid bitumen pitch or asphalt is dispersed by agitation in a suspension of bentonite or ball clay stabilised by the addition of a protective colloid, *e.g.*, alkaline casein solution. The resulting dispersion is unbroken by electrolytes and, on drying, yields a product having an irreversible film.

A. R. POWELL.

Manufacture of lubricating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,721, 10.12.28).—The flash point and viscosity of lubricating oils, especially used lubricating oils, are raised by treating the oils at high temperatures, under vacuum, with inert gases or with the vapours of liquids of low b.p. Wet steam or carbon dioxide containing benzene vapour is suitable.

T. A. SMITH.

Treating and purifying hydrocarbon lubricating oils. W. M. STRATFORD, Assr. to TEXAS CO. (U.S.P. 1,738,330, 3.12.29. Appl., 14.6.26).—Lubricating oil fractions are agitated at normal temperature with sulphuric acid and after separation of the sludge and neutralisation are distilled under vacuum in the presence of alkaline material. Caustic soda may be added as a solution or solid, or anhydrous soda ash may be used.

T. A. SMITH.

Treatment of hydrocarbon gases. ANGLO-PERSIAN OIL CO., LTD., A. E. DUNSTAN, and R. V. WHEELER (B.P. 327,715, 5.10.28).—Paraffin-containing gases, such as natural gas, wild gases from stills, and gases from cracking plants and low-temperature retorts,

are gradually heated to a temperature below that at which decomposition of the olefines and paraffins takes place. The temperature is then raised to 550–1250°, according to the nature of the gases used, and the gases are expanded and cooled. The free carbon produced is deposited and the aromatic substances formed are condensed. The reaction may be carried out in the presence of catalysts, such as nickel powder. The residual gas may be further decomposed to produce carbon-black, or mixed with oxygen-containing gases, such as carbon monoxide or dioxide, and used for the production of oxygenated hydrocarbons.

T. A. SMITH.

Apparatus for production of active carbon. J. N. A. SAUER, Assr. to N.V. ALGEM. NORIT MAATSCH. (U.S.P. 1,759,138, 20.5.30. Appl., 6.4.26. U.K., 9.8.23).—See B.P. 206,862; B., 1924, 548.

Process and apparatus for destructive distillation. R. H. CROZIER (U.S.P. 1,759,821, 20.5.30. Appl., 15.7.29. U.K., 18.5.28).—See B.P. 319,224; B., 1930, 179.

Purification of hydrocarbon compounds. Process and apparatus for treating hydrocarbons. T. T. GRAY, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,759,812—3, 20.5.30. Appl., [A] 26.9.23, [B] 24.3.25. Can., [B] 18.9.24).—See B.P. 222,481 and 249,871; B., 1926, 230; 1927, 290.

Jig washers [for washing coal etc.]. W., R. H., and W. REID (B.P. 329,488, 1.5.29).

Burners for pulverised or gaseous fuel. Soc. ANON. DES APPAREILS DE MANUTENTION ET FOURS STEIN (B.P. 316,667, 19.7.29. Fr., 2.8.28).

Diffuser for obtaining gaseous combustible mixtures. J. W. PENICUD (B.P. 328,493, 18.6.29).

Acetylene generator [for use with internal-combustion engines]. C. DE LA ROCHETTE (B.P. 307,401, 6.3.29. Fr., 6.3.28).

Heat exchange between gases and solids. (B.P. 328,640). **Separation of materials** (B.P. 328,283).—See I. **Modifying isocolloid materials** (B.P. 321,689, 321,691—4, 321,699, 321,722—7).—See III. **Ammonium sulphate** (B.P. 327,488). **Heating hydrogen for hydrogenation** (B.P. 329,045).—See VII. **Cellular aggregate** (U.S.P. 1,746,860). **Bituminous masses** (B.P. 310,923). **Stones, road surfaces, etc.** (B.P. 328,969). **Road-making** (B.P. 307,751).—See IX.

III.—ORGANIC INTERMEDIATES.

Synthesis of the higher hydrocarbons from water-gas at atmospheric pressure. E. BERL and K. JÜNGLING (Z. angew. Chem., 1930, 43, 435—440).—The authors have repeated Fischer's experiments on the synthesis of hydrocarbons from water-gas, using various catalysts. These were prepared by reduction of various mixtures of oxides of iron, copper, zinc, manganese, cobalt, and copper, with, in some cases, addition of potassium carbonate. The temperature was usually 290°. Details of yields are given with elementary analyses of the more and the less volatile liquid products. Iron catalysts were found to oxidise carbon monoxide preferentially, cobalt catalysts to oxidise hydrogen

first; cobalt catalysts are the more active, and the presence of copper is advantageous. The activity of the catalyst varies greatly with the method of preparation. It is best to prepare the catalyst by reduction of nitrates with diluted hydrogen or water-gas at as low a temperature as possible. Cobalt can be regenerated by heating in hydrogen, but iron catalysts should be heated in air to 400° and then treated with water-gas at the working temperature. The degree of saturation of the hydrocarbons produced increases with the mol. wt. The greater the hydrogenating activity of the catalyst the less is the yield of unsaturated compounds, aldehydes, and acids. C. IRWIN.

Synthesis from water-gas of alcohols higher than methyl alcohol. G. NATTA and M. STRADA (Giorn. Chim. Ind. Appl., 1930, 12, 169—174).—Previous processes for the high-pressure synthesis of oxygenated organic products from the components of water-gas by means of catalysts containing strong alkalis yield highly complex mixtures containing, besides methyl and other alcohols, considerable quantities of hydrocarbons, acids, aldehydes, ketones, esters, etc. The experiments now described show that, in presence of catalysts containing zinc oxide (obtained in a very active form by calcining certain smithsonite minerals at low temperatures) and certain alkali-metal (potassium, rubidium, caesium) compounds, it is possible, at about 400° and under high pressures, to convert mixtures of carbon monoxide and hydrogen completely into mixtures of liquid, oxygenated organic compounds consisting principally of saturated aliphatic alcohols. Contrary to what has been stated previously, the formation of alcohols higher than methyl alcohol does not require the presence of free metals and oxides of the groups IV—VIII of the periodic system. The complete absence of such metals and oxides is, indeed, advisable in order that formation of methane and other undesirable secondary reactions may be avoided. The formation of the higher alcohols takes place through the successive formation of methyl alcohol and alkali methoxide, and the addition of carbon monoxide with formation of alkali salts of fatty acids, with subsequent decomposition of these and hydrogenation of the resulting products. The intermediate alkali salts of the fatty acids have been identified on the catalysts and may with advantage be added to the latter, as they confer high catalytic activity, which remains constant over long periods. T. H. POPE.

High-boiling solvents from natural-gas pentanes. L. H. CLARK (Ind. Eng. Chem., 1930, 22, 439—443).—Amyl acetate for use as lacquer solvent is manufactured from pentane derived from natural gas. A fraction, 95% of which distills between 28° and 39°, consists chiefly of *n*- and *iso*-pentane. It is dried, and the vapour mixed with chlorine and passed to a pipe-still followed by four fractionating columns. These serve to separate hydrochloric acid, unchlorinated pentanes, and the mono- and di-chloro-derivatives. A mixture of six isomeric monochloropentanes is hydrolysed by digestion with 30% caustic soda and a catalyst. The vapour from the digesters consisting of amyl alcohols, amyl chlorides, and amylenes is fractionated and the

wet alcohols are dehydrated. The low-boiling tertiary alcohol is removed and the mixture of the other five isomerides acetylated in a kettle at 125°. The crude ester is neutralised with soda ash and rectified. The main difficulty in the development of this process has been the design of the coolers for chlorinated pentanes. These are now of iron, which is not corroded so long as the gas is perfectly dry and cooled by open water-sprays. Amongst the by-products of the process are amylenes, diamylene, and amyl ether. C. IRWIN.

Detection of small quantities of methyl alcohol in ethyl alcohol. O. MOHR (Mikrochem., 1930, 8, 154—158).—The alcohol is diluted with water so that the mixture contains about 5% of alcohol, and 5 c.c. of the mixture are treated with 0.3 c.c. of 85% phosphoric acid and 2 c.c. of 3% permanganate solution. After 10 min. 1 c.c. of 10% oxalic acid solution and, after a further 2 min., 10—12 drops of sulphuric acid (*d* 1.84) are added and the resulting aldehydes distilled into a test-tube cooled in ice and containing 5 c.c. of a 2.5% solution of ammonium chloride. After 30 min. the chloride solution, which contains hexamethylenetetramine corresponding with the methyl alcohol in the original alcohol mixture, is evaporated to dryness to expel acetaldehyde. The residue is dissolved in 5 c.c. of water, a few drops of sulphuric acid are added, and the liberated formaldehyde is distilled into 0.5—1 c.c. of a 0.5% solution of dimethyldihydroresorcinol (methone) made feebly alkaline with 1—2 drops of 10% sodium hydroxide solution. The distillate is then rendered acid with acetic acid and a drop placed on a microscope slide, which is set aside for some time; the formation of characteristic needles of methylenedimethone indicates the presence of methyl alcohol in the original alcohol. The test is sensitive to 0.05% of methyl alcohol. A. R. POWELL.

Industrial solvents : ethylene dichloride, dichloroethyl ether, and isopropyl ether. H. R. FIFE and E. W. REID (Ind. Eng. Chem., 1930, 22, 513—515).—The physical properties of these three solvents are tabulated and graphs are given showing their sp. gr. at various temperatures, vapour pressures, and comparative rates of evaporation in still and in circulating air. The solubilities of some fats, oils, waxes, resins, balsams, and dyes in the solvents are tabulated. H. INGLESON.

Organic fluorides as refrigerants. T. MIDGLEY, JUN., and A. L. HENNE (Ind. Eng. Chem., 1930, 22, 542—545).—Dichlorodifluoromethane, b.p. —30°, is quite stable at 175°, at which temperature when pure and dry it does not corrode steel, aluminium, copper, or tin. The liquid is non-corrosive to all constructional metals at atmospheric temperature. Experiments with animals show the gas to be, for practical purposes, non-toxic; it is also non-inflammable. It is prepared in 88% yield by bringing pure and dry antimony trifluoride into contact with carbon tetrachloride in the presence of a small amount of antimony pentachloride. It is economical to work at 60 lb. pressure. If any of the monofluoro-compound is formed, this may be re-worked and the yield of the difluoro-compound raised to 98%. The plant necessary consists of an autoclave, fractionating column, and dephlegmator. C. IRWIN.

[Methyl alcohol] pressure-synthesis. ANON.—See VII. Xylose. EMLEY.—See XVII. Determination of aldehydes in alcohol. WOJCIECHOWSKA-STRUSZYŃSKA.—See XVIII.

See also A., June, 680, Physical methods of separating constant-boiling mixtures (SUNIER and ROSENBLUM). 698, Vapour-phase esterification of acetic acid by ethyl alcohol (FROLICH and others). 711, Coupling of diazonium salts with phenols (CONANT and PETERSON). 788, Bases in the kerosene distillate of California petroleum (POTH and others).

PATENTS.

Modifying the physical properties of (A) fatty oils, (B) organic isocolloids, containing unsaturated high-molecular acids. (C) Manufacture of bases for water-fast varnishes and lacquers. (D) Production of rubber substitutes. (E) Modifying the physical properties of resins. L. AUER (B.P. [A] 321,689 and (B—E) 321,724—7, 4.5.28. [A] Addn. to B.P. 287,943; B., 1928, 398).—(A) The process of the prior patent gives particularly useful products from fatty oils containing high-molecular unsaturated acids or esters, *e.g.*, tung oil, linseed oil, castor oil, or fish oil. The best “modifying” agents are those which by Frumkin’s method (A., 1924, ii, 462) impart a negative surface charge to water in which they are dispersed (*e.g.*, sodium hydrogen sulphite). (B) The “modifying” effect is observed also with all substances comprising within the molecule an organic residue and an acidic inorganic residue (*e.g.*, trichloroacetic acid, triphenylmethyl chloride, 2:5-dichlorobenzenesulphonic acid, triphenyl phosphate, diphenylamine hydrochloride, *p*-nitroacetanilide); these may be used in conjunction with peroxides, carbonates, sulphites, sulphides, and other agents which generate gas during the process, and preferably the modifying agents are introduced in the colloidal condition. (C) For the production of water-fast varnish bases a “modifying” agent insoluble in water is used with linseed oil or other organic isocolloids containing unsaturated high-molecular acids; the agent may be produced *in situ*, and the product may be vulcanised. Resins may be used alone or with the fatty oil. (D) For the production of rubber substitutes the “modifying” agents should be such as develop during the process gases other than oxygen, *e.g.*, carbonates, sulphites, sulphides, etc., and may be produced *in situ*; the starting material is preferably a drying oil, castor oil, or a fish oil. (E) American, French, or Spanish rosins are converted into liquids suitable for lacquers by heating under reduced pressure with “modifying” agents, particularly ammonium iodide, zinc carbonate, magnesium sulphate, or chloride, sodium sulphide, barium thiocyanate, etc. with the addition of a little formic acid. C. HOLLINS.

Modifying the physical properties of organic isocolloid materials, by treatment of their emulsions. L. AUER (B.P. 321,692, 9.5.28).—The starting material (linseed oil, tung oil, rape oil) is emulsified in water and treated with “modifying” agents (*cf.* B.P. 287,943; B., 1928, 398; also preceding abstracts), with or without a gas-treatment. Preferably the emulsion is

maintained alkaline by addition of ammonia as required. The products during or after formation may be vulcanised. C. HOLLINS.

Vulcanisation or sulphurisation of organic isocolloids. L. AUER and N. STRACHOVSKY (B.P. 321,699, 13.6.28).—Organic isocolloids other than rubber are mixed with the usual vulcanising agents and are then vulcanised by treatment with steam, with or without the application of heat and pressure. The process is also applicable to rubber and other isocolloids which have been treated with “modifying” agents (*cf.* B.P. 287,943; B., 1928, 398; and preceding abstracts). C. HOLLINS.

Vulcanisation or sulphurisation of substances capable of vulcanisation or sulphurisation. Sulphurisation of organic isocolloids which have been treated with modifying agents. P. STAMBERGER and L. AUER (B.P. 321,693 and Addn. B.P. 321,694, 10.5.28).—The starting materials, which in (B) are organic unsaturated isocolloids modified, *e.g.*, by the process of B.P. 287,943 (B., 1928, 398), are vulcanised or sulphurised to the degree necessary to complete the first stage of vulcanisation, *i.e.*, distribution of the sulphur (probably as an additive compound) evenly through the mass, and the process is then completed by treatment with water, preferably by emulsification. Accelerators may be used. C. HOLLINS.

Manufacture of new saponaceous materials from organic isocolloids containing unsaturated high-molecular acids; vulcanisation of the products. L. AUER (B.P. 321,691, 321,722—3, 8.5.28).—(A) Alkali metals or their oxides or hydroxides are dispersed at 250–300° in substantially anhydrous isocolloids containing unsaturated high-molecular acids or their esters (linseed oil, fish oil, sunflower oil, rosin, castor oil, olive oil) to give soaps suitable also for use as binders in varnishes and linoleums, and as rubber substitutes, which can be vulcanised. The glycerin is apparently retained and acts as plasticiser. Barium peroxide is added with advantage. (C) The process is performed under pressure. (B) The products are vulcanised. C. HOLLINS.

Manufacture of acetaldehyde. W. O. HERRMANN and H. DEUTSCH, ASSRS. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,714,783, 28.5.29. Appl., 3.4.28. Ger., 4.4.27).—See B.P. 288,213; B., 1930, 275.

Preparation of cyclic ketonic compounds. H. GREUNE, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,111, 20.5.30. Appl., 5.7.27. Ger., 8.7.26).—See B.P. 274,095; B., 1928, 922.

Preparation of 6-chloro-2-nitro-1-methylbenzene-4-sulphonic acid. F. HENLE and B. VOSSEN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,554, 20.5.30. Appl., 24.8.25. Ger., 1.9.24).—See F.P. 434,402; B., 1927, 276.

Production of 1[3]-phenylbenzanthrone compounds. W. TRAUTNER, B. STEIN, and R. BERLINER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,519, 4.3.30. Appl., 11.7.27. Ger., 23.11.25).—See B.P. 297,129; B., 1928, 847.

IV.—DYESTUFFS.

Azotriphenylcarbinol dyes. R. N. SEN and (the late) A. K. SEN (J. Indian Chem. Soc., 1930, 7, 151—156. Cf. Green and Sen, B., 1912, 579; Sen and Sett, B., 1924, 211).—The leuco-azotriphenylmethane dyes obtained by condensing *p*-azobenzaldehyde-*oo'*-disulphonic acid or *p'*-salicylazobenzaldehyde-*o*-sulphonic acid with phenols are lighter in shade when dyed on wool than the azobenzaldehydes themselves, but the azotriphenylcarbinols produced by oxidation are slightly deeper. A methyl group *meta* to the hydroxyl group decreases the colour and dyeing properties; an additional hydroxyl group *para* to the azo group (salicylazotriphenylcarbinols) is almost without influence. The following are described [shades given are on wool (i) before and (ii) after chroming]. From azobenzaldehyde-disulphonic acid and salicylic acid, *methane*, (i) yellow, (ii) violet-black, *carbinol*, (i) red; 2-hydroxy-3-toluic acid, *methane*, (i) pink, (ii) black, *carbinol*, (i) bluish-red; 3-hydroxy-4-toluic acid, *methane*, (i) yellow, (ii) black, *carbinol*, (i) light chocolate; from salicylazobenzaldehyde-sulphonic acid: 2-hydroxy-3-toluic acid, *methane*, (i) yellow, (ii) black, *carbinol*, (i) red; 3-hydroxy-4-toluic acid, *methane*, (i) yellow, (ii) black, *carbinol*, (i) chocolate.

H. A. PIGGOTT.

Black, chromable disazo dye derived from amino-sulphosalicylic acid. V. G. GULINOV (Ukraine Chem. J., 1929, 4, [Tech.], 267—273).—A black dye for wool is prepared in the following way. 4-Nitroso-3-sulphosalicylic acid is reduced by sulphite to yield a mixture containing 62% of 4-amino-3-sulphosalicylic acid, together with, amongst other substances, 4-amino-3:5-disulphosalicylic acid. The crude product is diazotised and coupled with α -naphthylamine, and the disazo compound is again diazotised and coupled with β -naphthol. The final product is a mixture of two black dyes, the one containing disulphosalicylic acid being the more soluble in water and separable from the other by extraction with boiling water. These dyes should be treated with chromium solution after deposition on wool. Tables giving the fastness of these dyes to various factors are given.

R. TRUSZKOWSKI.

Solvents. FIFE and REID.—See III.

See also A., June, 694, **Colloid-chemical colour changes in organic dyes** (OSTWALD and RUDOLPH). 780, **Colouring matter from saffron** (KARRER and HELFENSTEIN). 793, **Formation of pyrrole-blacks** (QUILICO and FRERI).

PATENTS.

Manufacture of sulphur dyes. G. KALISCHER and H. RITTER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,261, 20.5.30. Appl., 26.6.28. Ger., 11.7.27).—See B.P. 319,860; B., 1930, 136.

Sulphur dyes. E. KRAMER, B. BOLLWEG, and L. ZEH, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,264, 20.5.30. Appl., 21.6.28. Ger., 31.8.27).—See B.P. 317,139; B., 1929, 890.

Dyes of the triphenylmethane series. K. SCHMIDT, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,113, 4.3.30. Appl., 20.10.28. Ger., 21.4.27).—See B.P. 324,966; B., 1930, 411.

Vat dyes. H. NERESHEIMER and W. SCHNEIDER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,501, 4.3.30. Appl., 9.11.27. Ger., 15.11.26).—See B.P. 280,492; B., 1928, 667.

Preparation of vat dyes of the anthracene series containing nitrogen. K. WILKE and J. STOCK, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,249, 20.5.30. Appl., 21.6.26. Ger., 2.7.25).—See B.P. 254,742; B., 1927, 550.

Acylaminodibenzanthrones and products resulting therefrom by treatment with alkylating agents. P. NAWIASKY, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,272, 20.5.30. Appl., 10.7.25. Ger., 11.8.24).—See F.P. 598,752; B., 1926, 311.

Manufacture of azo dyes. H. WAGNER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,681, 4.3.30. Appl., 23.12.25. Ger., 24.2.22).—See B.P. 201,712; B., 1923, 968 A.

Production of trisazo dyes. H. SCHWEITZER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,513, 4.3.30. Appl., 4.2.27. Ger., 26.2.26).—See B.P. 287,232; B., 1928, 399.

Aminodiarylsulphonepyrazolone-azo dyes. H. SCHWEITZER and W. NEELMEIER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,677, 4.3.30. Appl., 30.3.27. Ger., 8.1.25).—See B.P. 245,765; B., 1926, 734.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Study of the fibroin from silk in the isoelectric region. M. HARRIS and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 539—542).—Stable colloidal solutions containing up to 5% of fibroin were prepared by treating highly purified, degummed silk with 50% lithium bromide or 70% calcium thiocyanate solutions at 80° and dialysing for about two weeks, after addition of a few drops of toluene to prevent bacterial action. Using a 1 or 2% solution, determinations of the isoelectric point by means of measurements of solubility, viscosity, electrophoresis, and precipitation of the dyed fibroin gave results varying between p_H 2.0 and 2.3.

F. R. ENNOS.

Manufacture of chemical cotton. W. D. MUNSON (Ind. Eng. Chem., 1930, 22, 467—471).—The purification of cotton linters and its conversion into cotton cellulose are described. The value of linters for production of chemical cotton depends not only on the percentage of pure cellulose present, but also on the quantity and character of the foreign matter. Most of the chemical cotton is now produced from second-cut linters, corresponding to U.S. Standard grade Nos. 6 and 7, which are made by de-linting the seeds after removal of the longer fibre in the first cut.

F. R. ENNOS.

Synthetic fibre industry of America. C. E. MULLIN (Ind. Eng. Chem., 1930, 22, 461—467).—A short description of the methods of manufacture of the different types of synthetic fibre, with statistics of their production in the United States.

F. R. ENNOS.

Cellulose as it is completely revealed by X-rays. Special application to growth and classification of cotton, structure of wood, and manufacture of

rayon. G. L. CLARK (Ind. Eng. Chem., 1930, 22, 474—487).—An account is given of the fundamental structure of cellulose as deduced from X-ray diffraction spectra, employing certain improvements in technique. The method is also applied to show the various stages in the growth of cotton fibres, as a means of classifying the different grades of cotton, to obtain information as to wood structure, and to serve as a control in the manufacturing processes for viscose silk. F. R. ENNOS.

Determination of the degree of purity of cellulose fibres. M. FREIBERGER [with Z. VON VASS and L. HÖNIG] (J. Soc. Dyers and Col., 1930, 46, 111—118).—The reducing power of cellulose is utilised as a means of determining its purity or degree of degradation, and is determined by extracting the soluble reducing constituents with caustic soda (d 1.0375) and oxidising the extract with potassium ferricyanide, or, preferably, by digesting the cellulose directly with a caustic alkaline solution of potassium ferricyanide and determining the ferrocyanide formed. In the first method several successive extractions with caustic soda must be made to ensure complete extraction of the reducing constituents. The reducing power of the cellulose is termed the ferricyanide number (= g. of potassium ferricyanide reduced by 100 g. of the cellulose); the oxygen number is the equivalent quantity (in g.) of oxygen per 100,000 g. of the cellulose. In the first method 1 g. of cellulose cut up into small pieces is boiled for $\frac{1}{2}$ hr. with 150 c.c. of caustic soda (d 1.0375) in an Erlenmeyer flask provided with a ball stopper. The contents are then cooled, filtered through a Jena fritted-glass filter (No. 1), the filtrate is diluted to 250 c.c., and the residue similarly extracted until the extract is free from reducing properties. 10 C.c. of each extract are then heated together with 9 c.c. of 0.005*N*-potassium ferricyanide in a test-tube immersed in a boiling water-bath for 10 min., 2 c.c. of 30% acetic acid are added, the mixture is cooled, a few crystals of potassium iodide are dissolved therein, 10 c.c. of a zinc sulphate solution (50 g. of zinc sulphate crystals and 250 g. of sodium chloride per litre) are added, and the iodine liberated by the residual ferricyanide is titrated with 0.005*N*-thiosulphate. In the second method 0.2 g. of cellulose is heated as above with 10 c.c. of 0.005*N*-potassium ferricyanide and 10 c.c. of 10% caustic soda, and the residual ferricyanide then determined also as in the first method. The ferricyanide number obtained by the first method is usually about 14% higher than that given by the second, which is not attributable to oxidation during the extraction. Sizing impurities in the cellulose affect the ferricyanide number and must be removed before its determination. A. J. HALL.

Dispersion of cellulose and cellulose derivatives. S. E. SHEPPARD (J. Physical Chem., 1930, 34, 1041—1052).—Cellulose in the form of cotton fibres or wood pulp can be dispersed in inert organic liquids to give finely-divided suspensions by wet grinding in pebble or ball mills. A small fraction becomes colloiddally dispersed. Different rates of dispersion obtain with different organic liquids. The viscosities of the solutions in Schweitzer's reagent fall with increasing subdivision, and the higher the initial viscosity of the

cellulose the longer is the time required for disintegration to a given degree of dispersion. When grinding and drying are carried out in an atmosphere of carbon dioxide the copper number remains unchanged. The dispersions produced by mechanical disintegration in organic liquids behave almost identically with the hydrocellulose produced by the action of mineral acids on native cellulose. The modified properties include (i) diminished cuprammonium viscosity, (ii) increased solubility in 10% potassium hydroxide solution, (iii) increased reactivity on esterification, and (iv) increased adsorption of basic dyes. Small differences in reactivity, however, can be detected in the degraded celluloses of the same degree of mechanical dispersion produced by the action of acids in water or glacial acetic acid or by the action of organic liquids in the mill.

L. S. THEOBALD.

Manufacture and properties of a cellulose product (maizolith) from cornstalks and corncobs.

C. E. HARTFORD (U.S. Bur. Stand. Misc. Publ. No. 108, 1930, 10 pp.).—Maize stalks or corncobs are digested for 3 hrs. under 40 lb. steam pressure with 10% of their weight of caustic soda diluted to a 1% solution, and, after being washed free from alkali, the cooked pulp is diluted with water to a consistency of 4%, beaten for 2 hrs. to yield a gel, which is dried in moulds at 70°. The product is a dense, hard, bone-like substance, which is practically unaffected by oil, but softens on prolonged soaking in water; it is somewhat heavier and harder than the hardwoods and is a good electrical insulator. Possible cheaper sources of maizolith are the fine fibres or pith particles rejected in the insulating wall-board industry, and the soft residue from the process of extracting adhesive material from corncobs.

F. R. ENNOS.

Decomposition of [rye and] maize straw with nitric acid. H. SUIDA, H. SADLER, and F. NOSS (Papier-Fabr., 1930, 28, 345—346, 363—365; cf. B., 1929, 675).—The results are given, mainly in tabular form, of experimental digestions of straw with nitric acid. In the case of rye straw, digestion at 60—70° with acid of 5—10% concentration gives yields of 50—60%, the resulting pulps being of normal bleachability. Lower concentrations of acid give higher yields, but the pulps are difficult to bleach. Maize straw gives lower yields of pulp, optimum results being obtained with 4% acid at 80°; digestion for 90 min. gives a yield of 42%, the pulp being difficult to bleach. The effects of varying conditions of digestion on the parenchymal cells, knots, and pith have been investigated, and the results are illustrated by means of photomicrographs.

T. T. PORTS.

[Manufacture of wood-pulp board etc. by] the Masonite process. R. M. BOEHM (Ind. Eng. Chem., 1930, 22, 493—497).—Wood chips are disintegrated without the use of chemicals by heating in a "gun" for 30—40 sec. with steam at a pressure of 350 lb./in.², which is afterwards increased to 1000 lb./in.² and maintained thereat for 5 sec. On discharging from the "gun," the chips explode immediately owing to the high internal pressure, yielding a mass of long-fibre bundles, which, after suitable treatment, are formed into

board for insulating, panelling, and generally as a substitute for wood in many directions.

F. R. ENNOS.

Production of naval stores from waste wood.

R. C. PALMER (Chem. Met. Eng., 1930, 37, 289—292).—The preparation of turpentine, pine oil, and rosin is described. All the turpentine and part of the pine oil are isolated by steam-distillation of the chipped wood, which is then extracted with petroleum naphtha, b.p. 100—175°, to give an extract from which pure rosin is subsequently obtained. Refining of the above products, the preparation of abietic acid, and uses of the extracted wood are discussed.

E. H. SHARPLES.

Influence of experimental conditions on results of pulp-strength tests. E. GRUND (Papier-Fabr., 1930, 28, 329—337).—The effect of varying the successive operations in the preparation of test-sheets is discussed. Variation of the substance of the sheets gives a maximum breaking length with sheets of 80 g./m.² By allowing the fibre suspension to settle for 1 min., a reduction in breaking length of 15% is effected; longer settling times have no further influence. Dilution beyond 1:750 has no influence on strength, but less dilution gives lower strengths. Variation of couching pressure shows that maximum strength is obtained at 120—150 kg./cm.², and for times of application over 2 min. constant results are obtained. Increasing the drying temperature causes a reduction of strength. The effect of using a shorter strip in the Schopper tensile tester is to increase the breaking length. The Schopper-Riegler beating tester is used throughout to control the beating degree of the pulps.

T. T. POTTS.

Viscosimeter. KÄMPF.—See I. **Causticising plant.** STEWART.—See VII. **Xylose.** EMLEY.—See XVII.

See also A., June, 695, **Swelling of cellulose fibres** (HESS and others). 749, **Cellulose chemistry** (WELTZIEN). **Acetolysis of cellulose** (WEBBER and others). 751, **Cellulose xanthate** (GEIGER). 814, **Enzymic hydrolysis of viscose silk** (PRINGSHEIM and THILO).

PATENTS.

Manufacture of textile packages. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 328,944, 5.2.29).—In order that the yarn package may be removed from the bobbin after winding, an adhesive (wax or gelatin) is applied to the threads while travelling to the package or to the package itself during its formation.

F. R. ENNOS.

Manufacture of material capable of being marked by electrolytic decomposition. I. G. FARBENIND. A.-G. (B.P. 329,258, 16.10.29. Ger., 16.10.28).—Paper, textile fabric, etc. is impregnated with an acid solution which has been used as a photographic fixing bath, and is then dried; alternatively the silver in the impregnating solution may be displaced by a non-precious heavy metal, e.g., lead, before use.

F. R. ENNOS.

Production of high- α -cellulose fibre for the manufacture of cellulose derivatives. G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,741,540, 31.12.29. Appl., 16.12.25).—Raw pulp is treated with caustic alkali, hot or cold, to remove non- α -cellulose constituents, bleached, and again treated with a more dilute alkali, either hot

or cold, to dissolve oxycelluloses formed during bleaching.

F. R. ENNOS.

Manufacture of ammoniacal copper cellulose solutions. K. HESS and C. TROGUS (B.P. 301,752, 3.12.28. Ger., 3.12.27).—Cellulose is immersed in cuprammonium solution containing caustic alkali, in which the proportion of copper present to cellulose treated is at least 1:2.6, until the fibrous product has the composition $\text{Cu}(\text{C}_6\text{H}_{10}\text{O}_5)_2 \cdot x\text{NaOH}$; it is then dissolved in water, aqueous ammonia, or dilute cuprammonium solution.

F. R. ENNOS.

Manufacture of organic esters of cellulose. BRIT. CELANESE, LTD. (B.P. 300,140, 5.11.28. U.S., 5.11.27).—Cellulose acetate is partially hydrolysed in two stages. First a mild hydrolysis is produced by allowing the ester to remain for a time with sufficient water, which may contain acetic acid or hydrogen peroxide, and, after addition of excess of water, the ripening of the precipitated ester is completed in the presence of dilute acid.

F. R. ENNOS.

Manufacture of cellulosic esters. KODAK, LTD., ASSEES. of H. T. CLARKE and C. J. MALM (B.P. 304,279, 4.1.29. U.S., 18.1.28).—Cellulose esters of halogen-substituted fatty acids are produced by treating cellulosic material with a halogen-substituted fatty acid containing more than five carbon atoms (e.g. stearic or hexoic acid) and a halogen- or alkoxy-substituted fatty acid anhydride containing less than 10 carbon atoms, preferably chloroacetic anhydride, in the presence of catalysts, e.g., perchlorates, zinc chloride, or red phosphorus, if desired.

F. R. ENNOS.

Preparation of cellulose esters. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 328,588, 22.12.28).—For the production of crotonyl esters, cellulose is pretreated with crotonic acid and subsequently esterified with crotonic anhydride in the presence of a catalyst (sulphuric acid, sulphonic acids, zinc chloride, etc.), with or without diluents (benzene, toluene, or melted crotonic acid).

F. R. ENNOS.

Cellulose esters and ethers and their uses. G. P. DAVIES, W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,657, 25.10.28).—To avoid the high cost of the "vehicle" in the production of cellulose ester enamels, emulsions of the ester of the water-in-oil type, which give tough and transparent films, may be prepared by dispersing water, to an extent such that it is substantially absorbed in the emulsion, in a solution of the ester in small quantities of a solvent (cyclohexanone or its methyl derivatives) which is only slightly miscible with water, is sufficiently volatile to prevent undue prolongation of the drying period, and, under the conditions of drying, is less volatile than water, so that excess of solvent is present during drying of the film: besides pigments, gums and plasticisers may also be added.

F. R. ENNOS.

Manufacture of artificial threads by the dry-spinning method. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,423, 3.11.28).—Solutions of cellulose ethers or esters containing benzene and alcohol in addition to the usual solvents are heated before spinning to near the point at which they begin to boil

within the nozzle, and are extruded into a chamber where the threads produced meet a countercurrent of air containing the vaporised solvent, if desired, at a lower temperature.

F. R. ENNOS.

Manufacture of filaments, films, and the like from cellulose derivatives. H. DREYFUS (B.P. 328,636, 29.10.28).—Filaments of low denier, *e.g.*, below 1.5, are produced by spinning solutions of cellulose acetate in aqueous acetone (containing upwards of 10% of water) which have a low viscosity compared with solutions of the same cellulose acetate in commercial acetone, and drawing out the resulting filaments to the desired denier.

F. R. ENNOS.

Manufacture of artificial silk from cellulose esters or ethers. J. Y. JOHNSON. FROM I. G. FARBEN-IND. A.-G. (B.P. 327,420, 4.10.28).—On leaving the spinning nozzles, liquid threads consisting of a cellulose derivative in a volatile solvent, with or without a swelling agent, are treated for a short time with a volatile liquid or vapour capable of swelling the cellulose derivative without completely coagulating it, while being stretched, and are then solidified by evaporation of the volatile material, with or without continued stretching.

F. R. ENNOS.

Uniting [cellulosic] sheets or articles. BRIT. CELANESE, LTD. (B.P. 305,992, 12.2.29. U.S., 13.2.28).—Organic derivatives of cellulose (cellulose acetate), which have been ground with plasticisers to a finely-divided state in the absence of volatile solvents as described in B.P. 282,723 (B., 1928, 853), with or without the addition of fillers, pigments, etc., are applied between the surfaces of the articles to be united; these are then subjected to heat and pressure.

F. R. ENNOS.

[Cellulose binding agent for] joining wood, metal, glass, and the like together or to other materials. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS, Assecs. of W. PLINATUS (B.P. 303,855, 1.1.29. Ger., 11.1.28. Addn. to B.P. 302,324; B., 1930, 504).—Leather meal or sawdust is added to the cellulose ester adhesive specified in the prior patent.

A. R. POWELL.

Sizing of paper. E. LECOCQ (B.P. 313,171, 6.6.29. Belg., 8.6.28).—The pulp containing an agglutinant such as a resin soap is treated first with sodium bisulphate and then with aluminium sulphate, the acidity being afterwards neutralised by the addition of powdered chalk.

F. R. ENNOS.

Smoothing of paper. I. G. FARBEN-IND. A.-G. (B.P. 327,635, 6.6.29. Ger., 19.10.28. Addn. to B.P. 310,340; B., 1930, 505).—In modification of the prior patent, the paper is first coated with a substance, such as glue, gelatin, cellulose esters or ethers, which is impermeable to the smoothing agent.

F. R. ENNOS.

Manufacture of cellular products [from pulp]. M. GRUNBAUM (U.S.P. 1,746,648, 11.2.30. Appl., 8.5.28).—Paper, paper board, wall-paper, etc. may be rendered insect-, germ-, and fungus-proof by incorporating a cinchona derivative (0.04% of cinchonine) in the pulp, fillers, or printing ink during process of manufacture.

F. R. ENNOS.

Production of highly glazed, insulating paste-board. M. M. SEREBRIANY (B.P. 328,962, 30.1.29).—

Bark or wood material, worked up in the mill and in the form of dry fibre, is mixed with boiled linseed oil, raw turpentine, etc., with addition, if necessary, of solvents such as benzene, turpentine, petroleum, etc., and with or without resins dissolved therein; the paper mass so obtained is worked up into pasteboard on suitable machines.

F. R. ENNOS.

Treatment of residual liquors etc. [from wood-pulp manufacture]. L. BRADLEY and E. P. MCKEEFE, ASSRS. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,747,047, 11.2.30. Appl., 28.3.21).—The cyclic process described consists in cooking fibrous material in a suitable reagent, *e.g.*, alkaline sulphite, neutralising the residual liquor, *e.g.*, with sulphite, to precipitate organic matter, which is then separated, and adding acid sulphite to adapt the liquor for use as a cooking liquor. A further quantity of fibrous material is then cooked in this acid sulphite, and the residual liquor is neutralised, *e.g.*, with calcium hydroxide, separated from the precipitated organic matter, and treated so as to adapt it for use as a cooking liquor in the first process.

F. R. ENNOS.

Manufacture of vegetable wool. J. L. LAMBRECHT (U.S.P. 1,759,752, 20.5.30. Appl., 31.5.29. Fr., 9.6.28).—See B.P. 313,169; B., 1930, 236.

[Apparatus for] opening and cleaning of fibrous materials. TEXTILE & FILATURE SOC. ANON., and G. PLACQUET (B.P. 304,666, 23.1.29. Fr., 23.1.28).

Manufacture of [composite] textile yarn or thread. O. RASCH (B.P. 328,989, 9.2.29).

Method and apparatus for spinning fibrous material. BRIT. COTTON INDUSTRY RES. ASSOC., PLATT BROS. & CO., LTD., and A. E. OXLEY (B.P. 329,457, 6.4.29).

Manufacture or [stretching] treatment of [traveling] artificial threads or filaments. BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 329,620, 20.12.28).

Acid-resisting materials (U.S.P. 1,732,140).—See VII. Stratified bodies (B.P. 328,919).—See VIII. Insulating material (B.P. 329,604).—See XI. Grease from wool-scouring waters (B.P. 328,606).—See XII. Plastic masses (B.P. 304,612 and 305,238).—See XIII. Cellulose films (B.P. 305,653).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Absorption of tannic acid by cellulose acetate silk. P. E. KING and A. R. SAHASRANAM (J. Soc. Dyers and Col., 1930, 46, 118—121).—The observation of Ermen (B., 1929, 850) that cellulose acetate silk absorbs tannic acid similarly to viscose silk is confirmed, and tabulated results show the comparative affinities of these two types of artificial silks as obtained by steeping the silks in aqueous tannic acid and determining the non-absorbed portion by Loewenthal's method. Viscose silk absorbs about 40% more tannic acid than does cellulose acetate silk under comparable conditions. The tannic acid absorbed by cellulose acetate silk is most satisfactorily fixed by further steeping the silk in a solution containing an amount of tartar emetic equal to 25% of the weight of tannic acid originally applied; 50% of the weight is usual in similarly fixing cotton mordanted

with tannic acid. Cellulose acetate silk mordanted with tannin-tartar emetic has an increased affinity for the majority of the basic dyes examined, but not for Malachite Green. Rhodamine B and 6G give fluorescent shades on non-mordanted cellulose acetate silk, but on mordanted silk no fluorescence appears. A. J. HALL.

Fading of dyed textiles. I. P. W. CUNLIFFE (J. Soc. Dyers and Col., 1930, 46, 108—111).—The results obtained by exposing dyed textile materials to sunlight and artificial light under various conditions are summarised. Fading due to exposure to strong sunlight during the midday hours is fairly reproducible in different parts of the world except with certain dyes known to be sensitive to humidity or other agents such as sulphur dioxide. Dyed wools and silks behave more regularly than other fibres during fading; this is ascribed to their smaller sensitivity to humidity. Changes of hue which the unexposed parts of the samples frequently show in outdoor tests at Leeds are generally of the same type as is produced by a little sulphur dioxide acting in the dark. Tables are given showing, semi-quantitatively, the reaction of dyed wool, silk, cotton, linen, and viscose towards light and moisture as determined in the laboratory. Cotton dyes show wide differences in their sensitivity to moisture, but basic and azoic dyes are the most sensitive. Dyes are rather more sensitive to moisture on linen than on cotton; they are least sensitive on wool and silk. Lissamine Fast Red B, Erio Fast Floxine BL, and Naphthol Green B are appreciably more fugitive in the presence of sulphur dioxide than in a pure atmosphere. A. J. HALL.

Submerged combustion. HAMMOND.—See II. **Tanning processes.** SCHINDLER and KLANFER.—See XV.

See also A., June, 749, **Cellulose chemistry** (WELTZIEN). **Action of sodium hydroxide on cellulose** (RUMBOLD). 779, **Theory of dyeing** (PFEIFFER and others).

PATENTS.

Bleaching of [organic] materials. L. MELLERSH-JACKSON. From PILOT LAB., INC. (B.P. 328,544, 3.10.28).—Mixtures of mutually activating organic or organic and inorganic peroxides (e.g., a mixture of benzoyl peroxide with 5—10% of butyryl, hexoyl, or decoyl peroxide) are used for bleaching flour, soaps, oils, etc. [Stat. ref.] L. A. COLES.

Transfer of colour designs to fibrous materials. S. J. COX (B.P. 328,909, 2.11.28).—The design is first produced in printing ink on a temporary support of flexible material such as sized paper, which has been coated with an emulsion of soft resins, ordinary rosin, and water-soluble gums. The coated surface of the support, after dusting with powdered resin, is then applied to the surface of the material to be treated, and the back of the support is subjected to the action of steam and pressure, whereby the colouring matter is intimately mixed with the fused coating and carried into the fibres of the material, giving a clean, sharp outline. F. R. ENNOS.

Mercerisation of cotton for the production of a silk-like appearance and handle. E. LANG (F.P.

637,921, 23.11.26).—Cotton is kier-boiled, treated for 6—12 hrs. in a liquor containing 2—4% of an alkali, 0.5% of rosin soap, 5—20% of starch, and (optionally) a protective colloid, washed, mercerised in caustic soda of d 1.115 containing 1% of sodium hyposulphite or other reducing agent, soured, and bleached. The starch may be replaced by dextrin, flour, glue, or gelatin.

A. J. HALL.

Waterproofing of fabrics. M. and R. WALRAVE (F.P. 638,374—5, 3.12.26).—(A) Fabric is impregnated with a mixture containing 8 kg. of flour dissolved in 100 litres of dilute caustic soda to which are added 15 kg. of white soap (2% solution) and a mixture of 3 kg. of beeswax, 4 kg. of linseed oil, and 3.5 kg. of paraffin; it is then dried and treated with a solution containing 10 pts. of blood albumin, 35 pts. of aluminium acetate, and 55 pts. of water. (B) Fabric is impregnated with a solution consisting of 6 kg. of Marseilles soap, 8 kg. of blood albumin, and 100 litres of water, then dried, treated with a solution consisting of 47 litres of aluminium acetate (d 1.115), 11 litres of formaldehyde, and 50 litres of water, heated to 60°, and again passed through this solution. A. J. HALL.

Preparation of dyeings on the fibre. W. HENTRICH, M. HARDTMANN, and E. TIETZE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,258, 20.5.30. Appl., 20.12.28. Ger., 28.12.27).—See B.P. 321,737; B., 1930, 320.

Dyeing of acetate silk. R. S. HORSFALL, L. G. LAWRIE, and J. A. R. HENDERSON, Assrs. to BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,759,010, 20.5.30. Appl., 19.5.26. U.K., 29.5.25).—See B.P. 257,654; B., 1926, 976.

Treatment of fabrics, cloths, and similar material [to remove shininess]. A. ROSS (B.P. 329,003, 10.1.29).

Apparatus for liquid treatment of textile materials. M. FREIBERGER (B.P. 329,526, 5.6.29).

Drying machines for fabrics. Machines for treating fabrics by drying, dry-cleaning, dyeing, or washing. H. PIERCE (B.P. 329,347—8, 15.12.28. U.S., 7.5.28).

Heating of liquids (B.P. 328,548).—See I.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Pressure-synthesis operations of the Du Pont Ammonia Corporation. ANON. (Ind. Eng. Chem., 1930, 22, 433—437).—A description is given of the ammonia and methyl alcohol synthesis of the Du Pont Ammonia Corporation at Belle, W.Va. Coal is carbonised in beehive coke ovens and water-gas is produced from the coke thus obtained. The coke-oven gas is not used in hydrogen production. The crude water-gas, containing about 50% H₂ and 40% CO, is passed through scrubbers to low-pressure holders. The water-gas generators have a capacity of 70 million cub. ft. per day. The correct mixture is made in these holders and compressed at 25 atm., the pressure of the gas being further raised to 1000 atm. in the following stages by hypercompressors: inlet 25 atm., second stage 85 atm., third stage 320 atm. Carbon monoxide is removed in

methyl alcohol converters. After the purification step the gas is converted into ammonia, which is drawn off and stored as a liquid. Uncondensed gas is scrubbed with condensed steam. Tanks for the transport of ammonia have a shell thickness of $\frac{3}{4}$ in. H. INGLESON.

Application of continuous methods of operation to causticising by the lime-soda method. R. F. STEWART (Ind. Chemist, 1930, 6, 194—198).—The balanced type of Dorr thickener consisting of a steel tank divided into two or more compartments by sloping trays is described. Each compartment has its own feed and clear-liquor overflow. This apparatus does not deal with gritty solids; these, if present, should be previously removed in a classifier. The Dorr agitator employing a hollow shaft carrying compressed air to be distributed by the rotating arms is suitable for causticising in most cases. In kraft-pulp mills, however, air should not be used. A Dorr continuous causticising plant installed in such a mill is described. It includes a slaking mill, classifier, three agitators, and three thickeners; the last-named effect systematic washing. The units are naturally small compared with those necessary for batch-working. The advantages of continuous working are summarised. C. IRWIN.

Manufacture of hydrogen peroxide and X-ray ingestion material. J. SCHWYZER (Pharm. Ztg., 1930, 75, 594—595).—A process by which pure hydrogen peroxide solution and barium sulphate of quality suitable for administration for purposes of X-ray examination are obtained consists in treatment of barium peroxide with phosphoric acid, dissolution of the precipitated monohydrogen phosphate with further phosphoric acid, and regeneration of the latter by addition of sulphuric acid to the purified solution of the dihydrogen phosphate. S. I. LEVY.

Analysis of sodium bisulphite and of its compounds with formaldehyde. V. MORLÍK (Chem. Listy, 1930, 24, 179—181).—The indigo titration method for the determination of sodium bisulphite or of its formaldehyde compound is, if carried out in an inert atmosphere, such as coal gas, as exact and more convenient than the Bosshart-Grob copper-reduction method.

R. TRUSZKOWSKI.

Conversion of superphosphate into ammonium phosphate. K. MONIKOWSKI (Przemysl Chem., 1930, 14, 217—221).—Solutions containing about 3.3% P_2O_5 , 0.76% H_2SO_4 , and 0.15% Ca, suitable for use in the fermentation industries, can be cheaply obtained by treating 200 pts. of superphosphate with 16.6 pts. of sulphuric acid and 20.6 pts. of ammonium sulphate in 1000 pts. of water; under these conditions the greater part of the calcium present is precipitated as sulphate. The use of oxalates for the precipitation of calcium is not advantageous, owing to the high cost of recovery of oxalic acid from calcium oxalate. R. TRUSZKOWSKI.

Calcination or enrichment of phosphate rock. C. G. MEMMINGER, W. H. WAGGAMAN, and W. T. WHITNEY (Ind. Eng. Chem., 1930, 22, 443—446).—The more extensive and cheaply mined pebble deposits may be concentrated by calcination after a preliminary washing process. When calcined at 1100° in rotary

kilns pebble phosphate containing 76.17% of tricalcium phosphate may be raised to a content of 80.43%. All combined water is expelled, calcium carbonate is decomposed, organic matter completely oxidised, and most of the sulphur trioxide eliminated. Treatment at this temperature renders the phosphate rather resistant to attack by sulphuric acid, and for certain purposes a less drastic calcination at about 800° is preferred. The phosphate content is increased then only by about 2.5%. The kilns used are 140 ft. long and 8 ft. in diam., and revolve once in 70 sec. C. A. KING.

Interaction of ammonium carbonate with alcoholic potassium chloride solution. A. STERN (Z. angew. Chem., 1930, 43, 425—427).—The ammonia-soda reaction cannot be applied to the preparation of potassium carbonate in aqueous solution, the comparative solubilities of the salts causing their action to be reversed. It has been found, however, that it is practicable in alcoholic solution. Ammonium bicarbonate is at once decomposed by alcohol, but the normal salt is sufficiently stable to be used. The reaction was studied under varying conditions. The best yield, about 40%, was obtained at 5° , using 85% alcohol. The salt precipitated is a mixture of potassium carbonate and potassium chloride. The latter can be readily recovered if the final product of the process is to be caustic potash. C. IRWIN.

Conversion of sodium chromate into dichromate by the action of carbon dioxide under pressure. B. NEUMANN and C. EXSSNER (Z. angew. Chem., 1930, 43, 440—444).—This reaction was studied in a glass apparatus which permitted the precipitated sodium bicarbonate to be collected on a filter without releasing the pressure. The authors used a saturated solution of sodium chromate (16.7 g. Cr/100 c.c.) and one of half this concentration, the carbon dioxide being under pressures up to 10 atm. The conversion decreases with rise of temperature and is nil at 50° . Concordant results were difficult to obtain with the saturated solution, but with the more dilute solution conversion increased regularly with increase of pressure and reduction of temperature, rising to 70% at 0° and 10 atm. Conversions were apparently better in the saturated solution at 15 – 25° , but lower temperatures could not be used. Higher conversion figures than 65–75%, such as have been claimed, are not possible of attainment. It is, however, practicable to separate the sodium chromate and dichromate by evaporating *in vacuo* and then heating in an open pan to 145° , when the unconverted chromate is entirely precipitated. C. IRWIN.

Antiformin and antiformin substitutes. J. K. GJALDBÆK (Dansk Tidsskr. Farm., 1930, 4, 137—146).—The composition and stability of a number of technical solutions containing sodium hydroxide and sodium hypochlorite has been investigated. H. F. HARWOOD.

Analysis of calcium hypochlorite solutions. [Chloramine-T as a volumetric reagent.] E. FABRIZI (Papier-Fabr., 1930, 28, 346—347).—A method is given which is claimed to be less expensive and easier to manipulate than the usual methods. 10 C.c. of bleaching liquor, containing about 26 g. of available chlorine per litre, are diluted with 300 c.c. of water,

75 c.c. of 0.1*N*-arsenite solution are added, and the excess is titrated with 0.1*N*-Chloramine-T solution, starch-iodide solution being used as an indicator.

T. T. POTTS.

Assay of calcium carbide and Czechoslovakian standards. R. VONDRAČEK (Chem. Listy, 1930, 24, 193—197).—The provisional Czechoslovakian standards envisage the use of a modified Caro's apparatus for the assay of calcium carbide; this method is not exact, and an apparatus giving more accurate results is described. The yield of acetylene should not be given in c.c. of moist gas, as the Gay Lussac formula is only applicable to dry gases. The experimental error in the assay of carbide should not exceed 2%. R. TRUSZKOWSKI.

Manufacture of carbon dioxide and Epsom salts. W. P. HEATH (Ind. Eng. Chem., 1930, 22, 437—439).—Dolomite containing about 54% CaCO_3 and 45% MgCO_3 is ground and stirred with water and sulphuric acid. The carbon dioxide evolved is scrubbed, compressed, liquefied, and finally stored in steel cylinders. Specifications to which these cylinders are required to conform are mentioned. A solution of potassium permanganate is added to the neutral sulphates and iron and aluminium are removed. The sludge is then centrifuged and magnesium sulphate is washed out, crystallised, dried, and screened. H. INGLESON.

Titrimetric determination of magnesium. J. S. PIERCE and M. B. GEIGER (Ind. Eng. Chem. [Anal.], 1930, 2, 193—194).—Previous methods have been modified (B., 1928, 402; 1929, 284). For limestone, 0.5 g. is dissolved in 50 c.c. of 0.25*N*-hydrochloric acid, the solution being boiled and titrated with alkali until neutral to bromothymol-blue. The precipitate is collected and washed; to the filtrate are added 5 drops of a 0.1% alcoholic solution of dimethylaminoazobenzene and 0.25*N*-hydrochloric acid until faintly pink. After the addition of 10 c.c. of a saturated alcoholic solution of trinitrobenzene, the liquid is titrated with 0.25*N*-sodium hydroxide (carbonate-free) until the colour is dark red. The liquid is diluted to 100 c.c. and filtered; 50 c.c. of the filtrate are titrated with 0.25*N*-hydrochloric acid until neutral to an indicator having a colour change at p_H about 4.0 easily recognisable in a reddish solution (Congo-red). E. H. SHARPLES.

Commercial preparation of phosphorescent substances. B. RHODE (Chem.-Ztg., 1930, 54, 369—371).—The phosphorescence of the alkaline-earth sulphides is associated with the crystalline state and also involves the presence of traces of activators, usually heavy-metal sulphides. These substances are prepared by treating the oxides with sulphur, a flux, and the necessary activator in a crucible at 1000°. After the sintered mass has cooled it is crushed and sieved. A full range of colour in the phosphorescence is possible by varying the metals used, and several formulæ are given. The longest duration of phosphorescence is obtained by the use of strontia and magnesia. Phosphorescent zinc sulphide is prepared by precipitating an ammoniacal zinc solution with pure hydrogen sulphide, lead being excluded from the apparatus. The zinc sulphide is settled, filtered, and washed, and then ignited with a mixture of sulphur and the chlorides of potassium,

sodium, and ammonium, with copper chloride as activator. Careful attention to the purity of all the ingredients is necessary. A mixture of zinc and cadmium sulphides gives a greenish-yellow to red phosphorescence.

C. IRWIN.

Splitting strength of mica. J. W. OBRIMOV (Proc. Roy. Soc., 1930, A, 127, 290—297).—It is shown that the splitting strength of mica is a constant. A surface energy of mica can be determined which is independent of the shape of the mica sheets, and it is found that mica placed in optical contact is completely restored. Some electrical phenomena obtained by splitting mica in a high vacuum are described. The strength of mica appears to be greater in a high vacuum than in air. L. L. BIRCHUMSHAW.

Manufacture of bismuth salts. E. A. MAUERSBERGER (Chem. Weekblad, 1930, 27, 337—338).—An account is given of the preparation of various pharmaceutical bismuth compounds from the metal and its oxide ores. S. I. LEVY.

Dependence of the catalytic activity of vanadium pentoxide on its mode of combination with silica. I. E. ADADUROV and G. K. BORESKOV (Ukraine Chem. J., 1929, 4, [Tech.], 259—266).—Vanadium pentoxide deposited on dehydrated silicic acid has a greater catalytic action on the oxidation of sulphur dioxide than in the absence of silica; at the optimum temperature of 400°, 97–85% oxidation is effected. If vanadium is precipitated together with silicic acid and the resultant gel is dried, 99.4% yields of sulphur trioxide are obtained, at a rate of flow of gas (containing 6.2–7.0% SO_2) of 60 c.c./sec., at 400°. The second type of catalyst probably contains vanadium zeolite.

R. TRUSZKOWSKI.

Vanadium compounds as catalysts for the oxidation of sulphur dioxide. H. N. HOLMES and A. L. ELDER (Ind. Eng. Chem., 1930, 22, 471—473).—The principle in preparing a series of vanadium catalysts was to precipitate the vanadium compound, the promoter, and the supports simultaneously as far as possible in order that the catalyst should be peptised or colloiddally dispersed. The best catalyst was made by mixing potassium metavanadate with an excess of calcium chloride and then neutralising the excess calcium chloride with sodium silicate. The gel should be prepared from dilute solutions. This form of catalyst showed a 98% conversion of sulphur dioxide when working on 8% mixtures. Replacement of calcium by iron also gave good results, but copper, cobalt, nickel, chromium, and aluminium were not efficient promoters. Tungsten was inefficient and carnotite proved a poor catalyst. It is calculated that 1 pt. of vanadium is required for 48 pts. of sulphur, which indicates much larger contact space than with platinum, though with an equal life the cost of preparation of vanadium catalysts would be only 1% that of platinum. C. A. KING.

Moisture content of compressed nitrogen. A. C. WALKER and E. J. ERNST, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 139—140).—Moist nitrogen at an initial pressure of 150 atm. was allowed to discharge at a regulated rate through an instrument in which the moisture content of the free gas was continuously

measured. The humidity of the gas increased as the pressure in the cylinder decreased. Calculations of the value of the R.H. of the gas expanded to atmospheric pressure from different initial pressures were made on the assumption that the gas in the cylinder remains saturated throughout the discharge. This should give results which are higher than those observed. At pressures greater than 3 atm. the observed values are greater than those calculated. H. INGLESON.

Hydrogen cyanide in gaseous products from coal. NEWALL and others.—See II. **Uranium from its fused salt.** DRIGGS and LILLIENDAHL.—See X. **Concentration of manganese dioxide.** MARTIN.—See XI.

See also A., June, 708, **Explosion regions of gas mixtures** (POSTHUMUS). 715, **Preparation of arsine and stibine** (HLASKO and MASLOWSKI). 722, **Purity of electrolytic oxygen** (KEFFLER). 725, **Analysis of nitrating acid** (MÜLLER and KOGERT).

PATENTS.

Contact sulphuric acid process. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,741,310, 31.12.29. Appl., 3.7.28).—The catalyst comprises a complex zeolite containing vanadium in ter-, quadri-, and/or quinquivalent form with or without other metals of group VI, VII, or VIII, and with or without inert acid-resisting fillers, such as quartz, kieselguhr, or colloidal silica. Before use the substance is leached with dilute mineral or organic acids to remove the base-exchangeable alkali or alkaline earths. A. R. POWELL.

Purification of highly concentrated nitric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,156, 14.6.29).—The acid is freed from sulphuric acid by treating it with compounds of such ter- or bivalent metals as form nitrates soluble in concentrated nitric acid, *e.g.*, aluminium, magnesium, and iron. These metals give sulphates that are insoluble in concentrated nitric acid, and these can be readily separated. W. J. WRIGHT.

Production of phosphoric acid and hydrogen. BAYERISCHE STICKSTOFFE-WERKE A.-G. (B.P. 308,598, 19.3.29. Ger., 24.3.28).—Phosphorus is treated with water, both preferably being in a state of fine subdivision, at temperatures below 600° and at increased pressures (which may be produced by operating in a closed system, and stopping, if desired, when a constant pressure is developed). Phosphine which is also formed is in turn converted by the same process, in the same or a separate system, into the same products. Iron promotes the formation of phosphine, and the vessels used, therefore, should not be made of this metal; a material which catalyses the formation of phosphoric acid, *e.g.*, precious metals (silver), metal alloys (monel metal), or which becomes an active catalyst in the course of the process is, however, suitable. Other catalysts are certain metal (*e.g.*, copper) phosphides and phosphates, which may be used to line the reaction vessel; these may also be produced *in situ* by allowing the reactants, or other phosphorus compounds, to interact with the vessel wall, which is made of a suitable metal. All the said catalysts may be used in admixture. The reaction

is also favourably influenced by small quantities of bases (alkali or alkaline-earth hydroxides).

S. K. TWEEDY.

Production of chemically pure phosphoric acid. I. G. FARBENIND. A.-G. (B.P. 319,656, 25.7.29. Ger., 25.9.28).—Crude phosphoric acid is electrolysed with a cathode current density less than 1 amp./dm.² and using cathodes of copper, silver, chromium, platinum, or tantalum, at which metals or metalloids resulting from cathodic reduction of impurities are deposited without the phosphoric acid being reduced.

J. S. G. THOMAS.

Porous acid-resisting materials. A. Z. PEDERSEN, Assr. to PEPLID CORP. (U.S.P. 1,732,140, 15.10.29. Appl., 5.7.23).—A solution of an organic colloid in an organic solvent is thoroughly incorporated with an aqueous solution of a colloid, whereby mutual precipitation of the colloids takes place. After drying the product, it is rolled into sheets, and the water-soluble colloid is washed out in running water. *E.g.*, an acetone solution of cellulose nitrate is mixed with an aqueous solution of sodium silicate, and the precipitate is dried and rolled into a sheet which is soaked in water to remove the sodium silicate, leaving a porous celluloid sheet suitable for use in galvanic batteries. A. R. POWELL.

Catalytic ammonia synthesis. SELDEN Co., Assees. of A. O. JAEGER (B.P. 307,457, 8.2.29. U.S., 8.3.28).—A catalyst for the synthesis of ammonia from nitrogen and hydrogen comprises a zeolite derivative from which part of the exchangeable base may be leached out by treatment with dilute acid, and which may contain an admixture of active or inactive diluent. The catalyst may be prepared, for example, by fusing a mixture of 15 pts. of alumina, 11.5 pts. of zirconia, 18 pts. of potassium carbonate, and 100 pts. of magnetite, leaching the product with water, and treating the residue with dilute nitric acid. Other types of catalyst may contain tungsten, molybdenum, vanadium, and thorium in association with silica, iron oxide, and potash.

A. R. POWELL.

Synthetic manufacture of ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,079, 14.3.29).—The reacting gases from the catalyst chamber are passed through a water-cooler in which they are cooled under 200 atm. pressure to 15°, and about half their content of ammonia is removed in the liquid state. The gases are then passed into a low-temperature cooler in which they are cooled to -40° by allowing the already condensed ammonia to expand into the jacket of this cooler. Practically all the ammonia is thus condensed and the residual gases are returned to the catalyst circuit.

A. R. POWELL.

Production of sodium sesquicarbonate. C. SUNDSTROM and G. N. TERZIEV, Assrs. to SOLVAY PROCESS Co. (U.S.P. 1,748,739, 25.2.30. Appl., 8.4.26).—A solution containing 196 g. of sodium carbonate, 66 g. of sodium hydrogen carbonate, and 71 g. of sodium chloride per litre is mixed with sodium carbonate in the form of soda ash, and the mixture is heated at 60–80° while a regulated current of carbon dioxide is passed through the liquor in two stages, whereby crystals of sodium sesquicarbonate gradually grow in the liquor.

The finer crystals are continually returned from the second to the first saturating tank, and the coarser crystals are periodically removed from the second tank at the bottom, the mother-liquor being returned to the mixing tank at the beginning of the circuit.

A. R. POWELL.

Manufacture of sodium sulphate by the Hargreaves process. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 328,780, 22.4.29).—In order to enable the gases to penetrate readily, the sodium chloride is employed in the form of small, porous lumps (cf. B.P. 269,209; B., 1927, 831), and flue dust is incorporated with these to accelerate the reaction. A counterflow rotary furnace for causing the chloride to react with the gases below sintering temperature, with avoidance of incompletely formed sulphate, comprises a tubular shell with an axial tube for introducing and heating the gases, a spiral device attached to this tube and nearly in contact with the outer shell, and means for discharging and emptying. The furnace is electrically heated.

W. J. WRIGHT.

Production of ammonium sulphate. O. PIETTE (B.P. 327,488, 25.1.29).—Ammonium sulphate is precipitated, in a saturator fed with a cold saturated solution thereof, by means of ammonia and the mother-liquor is passed to a still, the liberated ammonia being introduced into the saturator. After cooling, the still liquor is treated with carbon dioxide, and the resulting ammonium carbonate solution mixed with finely-ground calcium sulphate. The calcium carbonate produced is removed, and the solution of ammonium sulphate returned to the saturator, the quantity of ammonia removed in the still being such that the solution of ammonium sulphate thus returned is saturated.

F. G. CLARKE.

Manufacture of commercial calcium arsenate [insecticide]. W. C. PIVER, Assr. to F. M. SIMPSON (U.S.P. 1,727,306, 3.9.29. Appl., 24.6.24).—Quicklime is slaked in a solution of calcium or sodium nitrates or in sodium hydroxide solution (d 1.02) and the granular suspension of slaked lime is agitated thoroughly while a 50% solution of arsenic acid is run into the vat. The product is claimed to be free from objectionable soluble acid arsenates.

A. R. POWELL.

Manufacture of calcium cyanamide or products containing the same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,743, 21.3.29).—Calcium cyanamide is produced by heating calcium carbamate under such conditions that the partial pressures of the gases injurious to the reaction (carbon dioxide, water vapour) are kept as low as possible. Thus the heating may be effected in presence of indifferent or favourable gases such as ammonia, carbon dioxide, nitrogen, or mixtures of these, if desired at elevated pressures and containing small amounts of other gases, e.g., hydrogen sulphide, which render innocuous any impurities such as iron which may be present in the carbamate. Alternatively, the heating may be effected very rapidly to 700–800°, preferably in an inert gas (absence of air). Further, the carbamate may also be heated under reduced pressure, e.g., 20 or 100 mm.

S. K. TWEEDY.

Treatment of lyes containing copper and zinc. N. E. LENANDER and I. RYEN (B.P. 329,225, 13.7.29).—Lyes obtained, e.g., by a chloridising roasting and subsequent leaching of copper pyrites, are treated, after the addition, if necessary, of soluble chlorides, with sufficient sulphur dioxide to impart an acidity of about 5–10% HCl per litre and an excess of finely-divided ("cement") copper is added to convert dissolved copper into cuprous chloride, the greater part of which is precipitated together with silver and gold present in the solution; the solution and the precipitate, together or separately, are treated with iron to convert the cuprous chloride into copper, the residual solution being worked up to zinc by the usual method.

L. A. COLES.

[Manufacture of] zinc sulphide [pigment]. C. W. FABER, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,742,030, 31.12.29. Appl., 16.6.27).—A solution of zinc chloride (d 1.5) is treated at 80° with a slight excess of a solution of barium sulphide (d 1.15), avoiding vigorous agitation, the mixture is diluted with an equal volume of water, the precipitate allowed to settle, and the clear liquor decanted off. The precipitate is washed by decantation until the liquor contains less than 0.2 g. of chlorine per litre, a small quantity of baryta water is added, and the mixture dried to 4–10% moisture content. The powder is then calcined at 650–725°.

Ferric alumina [for water purification]. A. R. MOBERG, Assr. to F. O. PAIGE, JUN. (U.S.P. 1,747,177, 18.2.30. Appl., 15.4.29).—A coagulant for use in purifying water over the entire range of alkalinity usually employed comprises a colloidal solution of aluminium hydroxide peptised with ferric chloride. The material is prepared by precipitation of 133 lb. of aluminium chloride in 40 gals. of water by means of ammonia and agitation of the washed precipitate with 270 lb. of ferric chloride solution (d 1.2).

A. R. POWELL.

Manufacture of aluminium fluoride. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,688, 5.2.29).—Gaseous hydrofluoric acid, or a mixture of hydrofluoric and hydrochloric acid, is passed over hydrated alumina at normal temperature, until clouds of fume are evolved, the reaction being then allowed to continue at a temperature not above 400°. A product containing 56% HF is thus obtained.

W. J. WRIGHT.

Recovery of vanadium from complex solutions. G. S. TILLEY (U.S.P. 1,747,403, 18.2.30. Appl., 3.6.27).—The mother-liquors from the crystallisation of alum obtained by digestion of alunite with dilute sulphuric acid contain iron and vanadium; these two elements are separated from the remaining aluminium by neutralising the solution (d 1.25–1.30) with milk of lime to pH 4, and, if necessary, adding bleaching powder to oxidise ferrous and vanadyl salts. The precipitate of basic ferric sulphate contains all the vanadium; it may be smelted in the usual way to obtain ferro-vanadium. The mother-liquor is returned to the alum-leaching vats.

A. R. POWELL.

Production of amphoteric hydrated oxides of metals by hydrolysis. (SIR) G. C. MARKS. From R. H. MONK and J. IRWIN (B.P. 329,041, 18.2.29).—

Amphoteric hydrated oxides of metals of group IV of the periodic table are produced by hydrolysing a solution of a salt of the metal to which a small amount of an oxide of the metal, which has been precipitated by means of an alkaline compound (ammonia, sodium carbonate), has been added, for the purpose of providing nuclei around which the hydrolysed hydrated oxide can crystallise. The product precipitates out readily, and is easily washed and separated by settling.

S. K. TWEEDY.

Detergent and its preparation. A. H. COWLES, Assr. to ELECTRIC SMELTING & ALUMINUM Co. (U.S.P. 1,745,844, 4.2.30. Appl., 6.3.26).—Mixtures of silica, aluminium silicate, and sodium (or potassium) carbonate in quantities sufficient to yield a product having the formula $\text{Al}_2\text{O}_3 \cdot (\text{SiO}_2)_x (\text{Na}_2\text{O})_{x-1}$ ($x > 7$) are heated to fusion, cooled, and pulverised; alkali hydroxides, alkali salts of weak acids, and/or emulsifying agents may be mixed with the product.

L. A. COLES.

Heating hydrogen for use in destructive hydrogenation. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,045, 21.2.29).—The hydrogen is passed through a cooling jacket on the inside wall of the hydrogenation converter, thence to a heat interchanger, utilising the sensible heat of the vaporous reaction products, and finally over an electric heater. The latter is preferably situated within the converter. The approximate temperatures attained by the hydrogen may be 100°, 300°, and 500° in the three stages, respectively.

S. K. TWEEDY.

Purification of gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,592, 18.1.29).—Gases, e.g., carbon monoxide, hydrogen, or combustible gases, containing phosphorus in combined (hydrides, chlorides) or elementary form are purified by mixing with oxygen, or a gas containing oxygen (e.g., air), and passing the mixture at a temperature above 100° (e.g., 150–400°, or even 800°) over mixed catalysts containing two constituents, one, (a), adapted to cause catalytic oxidation of the phosphorus impurities, and the other, (b), to bind chemically or to adsorb the phosphorus oxides formed by the action of (a). Constituent (a) comprises at least one earth or heavy metal of group I, III, IV, VII, or VIII of the periodic system, or a compound, e.g., oxide or hydroxide, of this metal; constituent (b) comprises at least one solid compound, e.g., oxide or silicate, of an element of group I, II, III, IV, or VIII. Examples are: kieselguhr or quartz moulded with copper nitrate solution to give a mass containing 5% Cu, and moulded brown coal briquette ashes. Pressures above or below atmospheric may be employed, and the adsorbed phosphorus oxides may be lixiviated from the catalyst.

S. K. TWEEDY.

Manufacture of oxygen preparations [for use in respirators]. W. ZISCH, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,745,465, 4.2.30. Appl., 31.1.29. Ger., 30.1.28. Cf. B.P. 319,393; B., 1929, 1034).—Pulverised alkali peroxides (100 pts.) are mixed at a low temperature with less than 23 pts. (preferably 10–15 pts.) of water as spray, vapour, or snow; oxides of iron, manganese, etc. may be added as activators.

L. A. COLES.

[Recovery of oxides of nitrogen from waste gases obtained in the] production of nitrates [from chlorides and nitric acid]. B. UEBLER, and KALIFORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 329,200, 17.6.29).—The waste gases, which contain nitrosyl chloride, chlorine, nitrogen, etc., after, if desired, removal of the free chlorine by fractional distillation at -30° , are passed at the ordinary temperature over scrap iron to yield nitric oxide and a nitrosyl chloride–ferric chloride additive product from which the nitrosyl chloride can be recovered by heating, or are passed over heated iron or lime to yield nitric oxide, or are treated with air and water or some of the original reaction liquid to yield nitric acid and chlorine. The recovered oxides of nitrogen and nitric acid are used again in the main process.

L. A. COLES.

Manufacture of liquid nitrogen dioxide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,029, 13.11.28).—Undiluted lower oxides of nitrogen are obtained by converting very dilute mixtures of nitrogen oxides into compounds in which they are chemically combined, but from which they can be recovered only in lower oxidised forms. The recovered lower oxides are then converted into nitrogen dioxide by means of pure oxygen, and the product is condensed to liquid nitrogen tetroxide by cooling to 15–18°.

W. J. WRIGHT.

Production of elemental sulphur. R. C. BENNER and A. P. THOMPSON, Assrs. to GEN. CHEM. Co. (U.S.P. 1,741,551, 31.12.29. Appl., 21.1.27).—Gases containing sulphur dioxide, e.g., roaster or smelter gases, are passed through a layer of hot carbonaceous fuel, whereby the sulphur dioxide is reduced to sulphur, carbon oxysulphide, and hydrogen sulphide. The gases are cooled to about 120° to effect deposition of the sulphur, and the residual gas mixture is passed together with more sulphur dioxide over a catalyst, e.g., bauxite or activated carbon, heated at 200–300° to effect complete conversion of all the sulphur compounds into free sulphur.

A. R. POWELL.

Recovery of sulphur from iron pyrites. R. F. BACON (U.S.P. 1,731,516, 15.10.29. Appl., 13.4.27).—Pyrites or other sulphide ore mixed with sufficient lime to combine with all the silica present is heated in steam at 400–800° in a rotary furnace, the evolved hydrogen sulphide being converted into sulphur by the Claus process or by treatment with sulphur dioxide.

A. R. POWELL.

Electrolytic production of fluorine. K. FREDENHAGEN (B.P. 315,768, 17.7.29. Ger., 17.7.28).—Solutions of alkali fluorides in hydrogen fluoride in a molecular ratio less than 1:1, but not so low that the electrodes are wetted by the melt, are electrolysed between carbon or graphite electrodes. In the case of potassium fluoride the above ratio is conveniently 5:9. The electrodes, not being wetted, are not destroyed, and a temperature appreciably lower than the usual may be employed.

S. K. TWEEDY.

[Electrolytic] production of fluorine. K. FREDENHAGEN (B.P. 315,769, 17.7.29. Ger., 17.7.28).—The comminuted alkali hydrogen fluoride is dried, prior to electrolysis, by passing dry indifferent gas, e.g., air, through

it at elevated temperature below the m.p. of the salt until the hydrogen fluoride evolved by the salt when periodically thermally decomposed has, after condensation, a specific conductivity of less than 0.02 reciprocal ohm at 0°. The drying is preferably effected in the electrolysis vessel itself, which should preferably be air-tight during the electrolysis, but it may also be effected in a separate vessel adapted to charge the cell with the dried material without the latter coming into contact with the atmosphere. The hydrogen fluoride content of the electrolyte is maintained by adding hydrofluoric acid containing only an extremely small quantity of water, *i.e.*, having the conductivity mentioned above.

S. K. TWEEDY.

Preparation of metallic cyanamides or mixtures containing them. H. H. FRANCK and H. HEIMANN (U.S.P. 1,745,753; 4.2.30. Appl., 19.10.27. Ger., 23.10.26).—See B.P. 279,419; B., 1929, 171.

Manufacture of iron carbonyl. A. MITTASCH and C. MÜLLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,759,268, 20.5.30. Appl., 29.1.25. Ger., 1.2.24).—See B.P. 244,895; B., 1926, 191.

Apparatus [freezing-press] for making carbonic ice [solid carbon dioxide]. SOLID CARBONIC CO., LTD. (B.P. 329,772, 8.4.29. U.S., 15.2.29).

Apparatus for the supply of chlorine or other gas for treatment of liquids. WALLACE & TIERNAN CO., INC., Assees. of G. D. PEET (B.P. 307,473, 8.3.29. U.S., 8.3.28).

Dehydration of solids (B.P. 329,032). Evaporation of solutions (B.P. 304,670).—See I. Cyanide compounds etc. from gases (B.P. 328,738).—See II. Aluminous materials (U.S.P. 1,719,131).—See VIII. Flux for welding (U.S.P. 1,748,785).—See X. Cyanogen compounds (B.P. 318,548).—See XI. Fertilisers (B.P. 299,896 and 328,620).—See XVI. Polluted liquids (U.S.P. 1,747,803).—See XXIII.

VIII.—GLASS; CERAMICS.

Action of dust from the batch on the checker brick of glass-furnace regenerators. D. S. BELIANKIN and M. A. BESBORODOV (J. Amer. Ceram. Soc., 1930, 13, 346—353).—The nature of slag formations in the chambers of regenerators of two types of pot-furnaces is discussed. Microscopical examination showed the constituents of this glazed slag to be alkaline aluminosilicates (nepheline, carnegieite) and some mullite. Analyses indicate that the composition of the slag is more complex than just *m*% of fireclay and (100—*m*)% of batch dust.

R. J. CARTLIDGE.

Use of borax in manufacture of tank glasses. D. D. SCHURTZ (J. Amer. Ceram. Soc., 1930, 13, 341—345).—A moderate amount of borax in making common types of tank glasses tends to improve melting, reduce factory breakage, and increase lustre, durability, and strength. A glass containing about 73.5% SiO₂, 2% Al₂O₃, 8% (Ca,Mg)O, and 1% B₂O₃ meets most demands; the beneficial effects, however, are stated to be almost independent of the composition of the glass.

R. J. CARTLIDGE.

Disintegration of glass by means of compressed steam. C. HASSLER (Chem.-Ztg., 1930, 54, 366).—The inner surfaces and edges of Petri dishes which had been heated for 30 min. in steam at 5 atm. showed, on removal from the autoclave next day, a deposit of ice-crystal-like structure. Only traces of condensed water could be detected in the dish. The deposit was distinctly alkaline. The outer surfaces remained unattacked.

S. K. TWEEDY.

Testing of "resistance-glass" ampoules. J. PRITCHARD (Pharm. J., 1930, 124, 539—541).—A 0.02% solution of phenol-red in 60% alcohol is diluted 20 times with freshly distilled water and boiled for 8 min. immediately before use. Of the resulting solution 0.3 c.c. is sealed in a 0.5-c.c. ampoule, which latter is then placed in a Koch's steriliser for 1 hr. The colour of the indicator when cold, is compared with a set of five standards ranging in *p*_H from below 6.8 to above 8.0, and the ampoule is thus assigned to one of five arbitrary classes. The heating and comparison are repeated on each of the next two days. A batch of ampoules is "good" if 90% of them after the first heating and 80% after the third have *p*_H 7.7 or less. Those of German origin fully conformed to this standard, but English-made ampoules did not, except a sample made from special hard borosilicate glass. The indicator does not change at all in ampoules of pyrex glass. The method of testing recommended in the German Pharmacopoeia (1926) gives results in agreement with the author's method, provided the glass is powdered sufficiently to pass through a No. 10 sieve but remain on a No. 20 sieve.

S. K. TWEEDY.

Volumetric determination of alkali metals in glass. N. A. TANANAEV and A. K. BABKO (Ukraine Chem. J., 1929, 4, 539—543).—0.5 Gram of glass is treated with 2—3 g. of oxalic acid and 4—5 c.c. of a mixture of hydrofluoric and sulphuric acids, and the mass is evaporated at 100° with occasional stirring. On the complete dissolution of the glass, 1 c.c. of 2*N*-barium chloride is added and evaporation is continued to dryness; then 2—3 c.c. of water are added, and the mixture is again evaporated. This operation is repeated 5—6 times, to the disappearance of acid fumes. Silica, excess hydrofluoric acid, and hydrochloric acid from the decomposed barium chloride are volatilised, and the dry residue consists of barium sulphate, a mixture of oxalates, and excess oxalic acid, the last-named being removed by sublimation at a higher temperature. The residue is extracted with hot water, and ammonia and ammonium carbonate solutions are added to precipitate aluminium and iron. The filtrate, which contains only alkali oxalates and ammonium salts, is evaporated to dryness and the residue ignited to yield carbonate. This is dissolved in water and determined by titration.

R. TRUSZKOWSKI.

Turquoise-blue glaze. G. M. PETERSEN (J. Amer. Ceram. Soc., 1930, 13, No. 5, ii, 47—49).—Trials were made to produce a turquoise-blue colour in a cone 1 alkaline glaze containing barium and calcium. Though the colour was satisfactory, all the samples crazed and had a tendency to "run."

F. SALT.

Effect of different fluxing oxides on the constitution of porcelain. A. H. FESSLER and H.

NAVRATIEL (J. Amer. Ceram. Soc., 1930, 13, 332—340).—On firing hard siliceous porcelain to a state approaching equilibrium, glasses having the formula $\text{RO} \cdot \text{Al}_2\text{O}_3 \cdot x\text{SiO}_2$ are probably obtained. Measurements of solubility and sp. gr., together with micrographs of the fused and quenched materials serve to indicate the numerical values of this formula, by determining the largest amount of silica and alumina that these fluxes (artificial feldspars) will hold in solution before crystallisation takes place. R. J. CARTLIDGE.

New form of non-plastic material [for porcelain]. A. H. FESSLER (J. Amer. Ceram. Soc., 1930, 13, 325—331).—A comparison is made of the use of semi-crystalline (unstable) and crystalline non-plastic materials in porcelain mixtures. The unstable material as produced by heat-treatment consists of a glass phase and an arrested crystalline phase, depending on the molecular ratio of alumina to silica, the fluxing oxides and impurities in the ingredients, and on their rate of cooling. During firing of porcelain the glass partially devitrifies and mullite crystals grow, forming a network of interwoven crystals and resulting in increased resistance to shock and mechanical strength of the finished product. R. J. CARTLIDGE.

Interpretation of plant and laboratory test data [in the ceramic industry]. R. F. FERGUSON (J. Amer. Ceram. Soc., 1930, 13, 354—362).—Mathematical. R. J. CARTLIDGE.

Manufactured abrasives. F. L. NOBES (Ind. Chemist, 1930, 6, 183—184).—A modern carborundum furnace is a rectangular firebrick structure with electrodes at opposite ends. A typical charge consists of 56% of sand, 35% of petroleum coke, 7% of sawdust, and 2% of salt. The initial resistor charge in the core of the furnace is granular coke, and a current of 10,000 amp. is used. After interaction is complete the furnace is pulled down, the outer layer of partly reduced silica removed, and the silicon carbide crystals are broken up. The temperature range for the reaction is 1500—2200° and the probable equation $3\text{Si} + 2\text{CO} = 2\text{SiC} + \text{SiO}_2$. Silicon carbide is slowly oxidised in air at 1000—1350° and reacts with chlorine at 900° and above. It is not acted on by acids, but is attacked by fused alkalis. It has d 3.12—3.20, very high thermal conductivity, and low coefficient of expansion. Crystalline fused alumina (alundum etc.) gives better results than carborundum for grinding materials of high tensile strength. It is prepared by treating bauxite in an electric furnace consisting of an unlined, water-cooled, cylindrical, steel shell. The impurities in the bauxite are reduced to ferrosilicon. Alundum has d 3.9, is almost as hard as carborundum, but has not so high a thermal conductivity. It is slightly soluble in hot acids. C. IRWIN.

Gypsum plaster and ceramics. WISS and others. —See IX. Cadmium-selenium red. MARLEY.—See XIII.

See also A., June, 665, Fluorescence of praseodymium and neodymium glasses (PRINGSHEIM and SCHLIVITCH). 669, Dynamic aggregation in glass (BERGER). 690, Argillaceous suspensions (DUBRISAY and TRILLAT). Dispersion of clays (ALLAM).

PATENTS.

[Continuous-type tunnel] kilns. H. M. ROBERTSON (B.P. 311,316, 23.1.29. U.S., 9.5.28).—The kiln comprises zones in succession for preheating, firing, salt-glazing, and cooling. Hot air is withdrawn from the cooling zone and passed through passages in the roof to points distributed in the preheating zone; this air returns in the same direction as the goods and in contact with them and is used for secondary combustion in the firing zone, the combined gases from this and the salt-glazing zone being withdrawn by a fan through ports situated where the cooling zone commences. B. M. VENABLES.

Manufacture of glass. K. KÜNZEL (B.P. 314,209, 20.6.28).—The charge is melted in a shaft furnace by means of a flame from the combustion of a liquid or gaseous fuel. The flame is forced through the mass by injector action or is drawn through it by suction applied at the top of the shaft. A. R. POWELL.

[Manufacture of] stratified bodies such as strengthened glass. TRIPLEX SAFETY GLASS CO., LTD., W. R. LYTTLETON, J. WILSON, and H. W. DICK (B.P. 328,919, 2.2., 18.3., and 27.3.29).—After coating the inner surfaces of two or more sheets of glass with gelatin and, preferably, with a cement containing a cellulose ester, these surfaces and/or the layers of strengthening material to be interposed (celluloid) are sprayed with a solvent or plasticiser capable of swelling both. The surfaces to be united are then brought together to effect a preliminary cohesion, and the united sheets are afterwards compressed in a closed vessel at an elevated temperature by means of a fluid under pressure, which may be inert (air) or have a softening action on the interposed layers and coatings (a phthalate or a glycol). F. R. ENNOS.

Rendering enamels cloudy. I. KREIDL (B.P. 313,411 and Addn. B.P. 315,708, [A] 10.6.29, [B] 9.7.29. Ger., [A] 9.6.28, [B] 16.7.28).—(A) Clouding agents such as ammonium oxalate etc. which act by liberating a gas are used in addition to the usual white clouding agents, e.g., tin oxide or zirconium oxide. The latter act as distributors or carriers for the gas-liberating agents, so that enamel batches of any composition may be used; a preliminary clouding of the batch, e.g., by adding fluorine compounds, is thus rendered unnecessary. (B) Any insoluble substance which is not a clouding agent is used as a distributor. It may be chosen so as to modify the viscosity or surface tension of the batch, and may be added to the mill or the melt. F. G. CLARKE.

Refractories [magnesite bricks]. B. M. O'HARRA and E. A. SLAGLE, Assrs. to AMER. SMELTING & REFINING Co. (U.S.P. 1,747,905, 18.2.30. Appl., 12.6.28).—Magnesite bricks without an iron or other foreign bond are composed of granular calcined magnesite to which has been added up to 10% of colloidal or finely-divided magnesium carbonate or basic carbonate. The bricks are moulded under high pressure and burned until the magnesium carbonate is converted into periclase. C. A. KING.

Preparation of aluminous materials [corundum from bauxite]. R. R. RIDGWAY and J. B. GLAZE, Assrs. to NORTON Co. (U.S.P. 1,719,131, 2.7.29. Appl., 15.10.26).—A mixture of 100 pts. of calcined bauxite, 5–10 pts. of coke, 5 pts. of pyrites, and 20 pts. of iron borings is fused in a carbon-lined electric furnace of the submerged-arc type and the aluminous slag is separated from the ferrosilicon and ferrotitanium alloy regulus. This slag, which should contain aluminium carbide and less than 3% of aluminium sulphide surrounding crystallised grains of alumina, is roughly broken up and caused to disintegrate by treatment with steam for several hours. The washed product is dried and heated in chlorine to decompose carbides and to volatilise iron and titanium, and the resulting alumina is again fused. A. R. POWELL.

Process and plant for casting rough plate glass. COMP. RÉUNIES DES GLACES & VERRES SPÉCIAUX DU NORD DE LA FRANCE (B.P. 317,820, 3.7.29. Fr., 23.8.28).

Methods and apparatus for colouring hollow glass articles. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of J. T. FAGAN (B.P. 329,940, 15.10.29. U.S., 15.10.28).

Enamelling furnace (B.P. 308,613).—See I. **Joining glass to wood etc.** (B.P. 303,855).—See V. **Torch furnace for mullite** (U.S.P. 1,747,756).—See XI.

IX.—BUILDING MATERIALS.

Measuring viscosity of setting cement (immersion-filter method). K. E. DORSCH and A. DEUBEL (Kolloid-Z., 1930, 51, 180–186).—Measurements of the viscosity of cement pastes carried out by the usual methods did not give reproducible values, but a method of measurement is described which allows determinations to be made with an accuracy of about 5%. The course of setting of all the cements examined is marked by a sharp rise in viscosity during the first 60 min., then a region of constant viscosity for about 30 min., and finally by another slow rise in viscosity. E. S. HEDGES.

Gypsum plaster in the ceramic industries. J. E. WISS, T. P. CAMP, and R. B. LADOO (J. Amer. Ceram. Soc., 1930, 13, 287–314).—The qualities of set plaster are examined. Effects of varying consistency, mixing time, etc. are described and illustrated by graphs, indicating the importance of the water:plaster ratio. In a workable mix the ratio is 0.6:1; on setting, the product has a compressive strength of 2500 lb./in.², a total absorption of 25% by wt., and a dry density of 73.5 lb./cub. ft. Such troubles as pinholes, hard spots, cracking, efflorescence, etc., met with in practice, are considered and methods for their control or prevention are indicated. Physical and chemical tests for the raw materials are also given. R. J. CARLIDGE.

Penetration of water vapour into wood. L. M. PIDGEON and O. MAASS (Canad. J. Res., 1930, 2, 318–326).—The rate of diffusion of water vapour through wood fibre was measured by means of a quartz spring balance completely enclosed in an atmosphere of constant vapour pressure, the process being a reversal of the natural drying of wood. Considerably higher rates of diffusion were observed for sapwood than for heart-

wood, and also when tested in a longitudinal direction with the fibres. At concentrations below the saturation point the movement appears to take place as vapour diffusion through the spaces of the wood structure, due to the pressure gradient caused by the difference of moisture content. C. A. KING.

Analysis of bituminous materials. HYATT.—See II. **Maizolith.** HARTFORD. **Wood-pulp board.** BOEHM. **Naval stores from waste wood.** PALMER.—See V.

PATENTS.

[Continuous-type] tunnel kilns. H. M. ROBERTSON (B.P. 328,265, 23.1.29).—A kiln suitable for lime burning and similar purposes is constructed so that the pre-heating is effected by the products of combustion passing countercurrent to the goods, and the cooling by fresh air which is drawn through the goods then used for combustion. A form of carrier is described which comprises closely coupled trough-like trucks on which the material forms a continuous elongated heap. B. M. VENABLES.

Manufacture of material for [heat] insulation and constructional purposes. H. C. M. INGEBERG and A. VESTERLID (B.P. 327,407, 2.1.29).—A plastic mass, the solid constituents of which consist of 99–50% by wt. of defibrated straw or hay and 1–50% of chemical fibrous cellulose, e.g., sulphite-cellulose, is shaped and dried. The articles may be surfaced with an aqueous suspension of cellulose or asbestos containing dyes, fillers, or reinforcing agents, and treated with the usual waterproofing, fire-resisting, and preservative materials. F. G. CLARKE.

Manufacture of cement. A. CARPMAEL. From Soc. POUR L'EXPLOIT. DES PROC. PETRUS (B.P. 305,170, 28.1.29).—The cement comprises a mixture of 5–27% of Portland cement, 2–22% of lime, and 60–80% of puzzolana prepared from vitrified blast-furnace slag. Plaster of Paris (1–10%) may be added to control the setting. A. R. POWELL.

Cement manufacture. R. W. HYDE, Assr. to DWIGHT & LLOYD METALLURG. Co. (U.S.P. 1,746,944, 11.2.30. Appl., 26.3.27).—The finely-divided raw material for cement manufacture is mixed with pulverised fuel, sintered, and finally heated for a short time to the finishing calcining temperature. C. A. KING.

Cement compositions. H. and N. CADMAN (B.P. 329,107, 6.4.29).—Approx. 1 lb. of hair is mixed with 1 cwt. of Portland or similar cement as a dry mixture for the preparation of a cement plaster for external use. No free lime is used. C. A. KING.

Cement composition. M. E. GRUNEWALD (U.S.P. 1,748,839, 25.2.30. Appl., 4.6.28).—A grouting composition for use under very wet conditions consists of 100 pts. of Portland cement, 0.05–10% (1 pt.) of sodium carbonate, and approx. 100 pts. each of water and sand. C. A. KING.

Cement powders and mortar powders. I. G. FARBERIND. A.-G. (B.P. 328,538, 2.10.29. Ger., 2.10.28).—Sodium silicate cementing compositions are made by using pulverised sodium silicate or other silicates which contain hydrated silicic acid and which are easily soluble in water. C. A. KING.

Manufacture of cellular aggregate. R. L. LLOYD, Assr. to DWIGHT & LLOYD METALLURG. Co. (U.S.P. 1,746,860, 11.2.30. Appl., 18.1.27).—The content of combustible matter in waste products is increased and the mixture is spread evenly on a pervious bed and ignited. Combustion of the mass is allowed to proceed until a thin semi-plastic layer is formed which is immediately blown by the current of gas into thin-walled cells and chilled, thus forming a homogeneous, cellular, rigid cake for use as filler in concrete floors, etc.

C. A. KING.

Production of sand-lime bricks. H. W. CHARLTON (U.S.P. 1,745,601, 4.2.30. Appl., 18.11.26).—Subsequent to the usual hardening process and after the steam is blown off, a solution, *e.g.*, a 2–3% solution of sodium stearate, is introduced into the cylinder to impregnate the bricks, which are then withdrawn and dried.

C. A. KING.

Preparation of bituminous masses. N.V. MIJNBOUW-EN CULTUURMAATSCHAPPIJ "BOETON" (B.P. 310,923, 9.3.29. Holl., 3.5.28).—The addition of a small quantity of water when preparing a cold-plastic bituminous mass by mixing a low-boiling solvent with an artificial or natural mixture of bitumen and a filler, *e.g.*, Boeton asphalt-limestone, temporarily reduces the adhesiveness of the bitumen and prevents clotting. The water and solvent may, if desired, be added as an emulsion. The product is applicable to moist surfaces, *e.g.*, soft concrete.

F. G. CLARKE.

Treatment of stones or stony materials, cement, concrete, bricks, road surfaces, etc. C. G. FOX (B.P. 328,969, 7.2. and 19.7.29).—Previous to the coating of road aggregate with tar or bituminous emulsions, the aggregate is treated with a dilute (0.5–3%) solution of the emulsifying agent.

C. A. KING.

Road-making materials and construction of roads. C. G. J. LEFEBVRE and E. E. F. BERGER (B.P. 307,751, 8.3.29. Fr., 12.3.28).—A tar filler composition for coating macadam road material or for dressing roads consists of about equal proportions of a tar binder and a mineral filler of low porosity, excluding clay and lime. The filler may be quartz sand, quartzites, clinkers, etc. pulverised to pass 300-mesh, and the viscosity of the mixture should be about the same as that of the tar.

C. A. KING.

Preservation and disinfection of wood. I. G. FARBERIND. A.-G. (B.P. 305,943, 11.2.29. Ger., 9.2.28).—A mercurised aliphatic hydrocarbon, *e.g.*, mercuric ethyl bromide, is used as the active ingredient, being 10–20 times as active as the inorganic mercury compounds. Such organic compounds are sparingly soluble in water and may be converted into the colloidal state by dissolution in a mixture of water, acetone, and dextrin, or used in solution in organic solvents, *e.g.*, alcohol, phenol. Alkaline salts also increase the solubility of these compounds in water.

C. A. KING.

Transfer of wood grain. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTLICHER OBERFLÄCHEN (B.P. 303,804, 9.1.29. Ger., 9.1.28).—Light is allowed to pass through an extremely thin section of the wood on to a sensitised surface in contact with the wood, by a process similar to

the ordinary light-copying process. The wood section may be stained and mounted on glass before copying.

C. A. KING.

Manufacture of [heat-]insulating material [from felt]. UPSON Co., Assees. of C. A. UPSON (B.P. 306,559, 23.2.29. U.S., 23.2.28).

[Rotary apparatus for] manufacture of plates from asbestos or fibro-cement. R. BINDSCHEDLER (B.P. 329,850, 28.5.29).

Wall and ceiling coverings and the like. A. HIRSCHFIELD (B.P. 328,985, 8.2.29).

Process and [hot-air] device for drying timber. T. C. GROOT (B.P. 329,842, 21.5.29. Holl., 25.1.29).

Cellular products (U.S.P. 1,746,648). **Joining wood to metal etc.** (B.P. 303,855).—See V. **Polluted liquids** (U.S.P. 1,747,803).—See XXIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Heat economy in metallurgical furnaces. (See) R. HADFIELD and R. J. SARJANT (Fuel, 1930, 9, 219–224).—Improvement in the heat economy of furnaces can be effected (a) by suitable control of the load; (b) by control, preferably automatic, of the fuel supply; and (c) by the recovery of heat from the waste gases and the furnace structure. Instances from works' practice illustrating quantitatively the advantages of such measures are discussed, and attention is directed to the possibility of using heat-resisting steel in recuperator practice and in the construction of regenerators.

A. B. MANNING.

Separation of carbide from rustless non-magnetic chrome-nickel steel on heating. B. STRAUSS, H. SCHORTKY, and J. HINNÜBER (Z. anorg. Chem., 1930, 188, 309–324).—For the heat treatment of chrome-nickel steel alloys having the composition $C < 0.16$, $Ni\ 8.5$, $Cr\ 18\%$, a temperature above 1100° is employed, followed by rapid cooling without subsequent annealing. In the hot-working of such steels a range of temperature exists, near 700° , in which the mechanical and chemical properties are profoundly altered, the metal becoming weak and oxidisable. Measurements of the tensile strength, extensibility, and electrode potential, in conjunction with photomicrographic examination, show that the change in properties is due to the separation of carbide from the initially homogeneous mixed crystals. The increased oxidisability is due partly to surface corrosion and partly to intercrystalline corrosion brought about by the formation of local galvanic elements. Although direct observation shows that the separation of carbide at 800 – 900° is considerably greater than at 600 – 700° , the modification of properties is less marked at the former temperatures. This is attributed to the fact that at the higher temperature the carbide is less highly dispersed.

F. L. USHER.

Determination of chromium and vanadium after oxidation with potassium bromate; separate titration of chromate and vanadate in the same solution and application of the method in steel analysis. I. M. KOLTHOFF and E. B. SANDELL (Ind. Eng. Chem. [Anal.], 1930, 2, 140–145).—The deter-

mination of chromium and vanadium in a steel is carried out by oxidising the solution of the steel in sulphuric acid by potassium bromate. The chromium can then be selectively reduced by means of a slight excess of standard arsenious acid and this excess determined by permanganate. After this titration the vanadium is determined by ferrous sulphate, using diphenylbenzidine as indicator. Since tungsten would interfere with this latter titration it must be removed, if present, by the method of Willard and Young (B., 1928, 643). Manganese does not interfere with the determinations. Results obtained in the analysis of steels of known composition agreed well with the known values.

H. INGLESON.

Determination of tantalum, tungsten, vanadium, and molybdenum in high-speed steel. K. SWOBODA and R. HORNÝ (Z. anal. Chem., 1930, 80, 271—288).—The conditions for the quantitative precipitation and determination of the above elements have been studied, and the following procedure is recommended for their determination in high-speed steels. The finely-divided steel (1.2 g.) contained in a nickel crucible is roasted in oxygen at 400—500° for 5 min.; 12 g. of potassium hydroxide are then added, and the whole is heated to redness for $\frac{1}{2}$ hr. in a current of oxygen. The melt is dissolved in water, any manganate formed is destroyed with hydrogen peroxide, and the solution is made up to 300 c.c. After filtration, 250 c.c. of the filtrate (\approx 1 g. of steel) are neutralised with sulphuric acid, using phenolphthalein as indicator. The liquid is boiled for 3 min., 10 c.c. of ammonia are added, and the whole is kept at 100° for $\frac{1}{2}$ hr. The precipitate of tantalic acid is filtered, washed with ammoniacal ammonium nitrate, ignited, and weighed after removal of silica with hydrofluoric and sulphuric acids. The filtrate is evaporated to dryness after addition of 20 c.c. of concentrated hydrochloric acid and 20 g. of manganese nitrate, and the residue heated until the nitrates are decomposed. The residue from this is treated with 50 c.c. of concentrated hydrochloric acid, the solution diluted, and the precipitated tungstic acid filtered off and weighed after treatment with hydrofluoric acid to remove traces of silica. The filtrate is evaporated to 300 c.c., 20 g. of ammonium chloride are added, and the liquid is neutralised with ammonia; 12 drops of ammonia in excess are then added, and the whole is set aside for 6 hrs. The precipitate containing the vanadium is washed with hot water, redissolved in hot hydrochloric acid, and the solution evaporated to fuming with excess of sulphuric acid. In the resulting diluted solution the vanadium is determined volumetrically by the usual permanganate method, using an excess of permanganate and back-titrating with arsenious acid solution. The filtrate from the manganese vanadate and chromium hydroxide is evaporated to 100 c.c., treated in a pressure flask with 5 c.c. of concentrated sulphuric acid, diluted, and the molybdenum precipitated by treatment with hydrogen sulphide under pressure. The precipitated molybdenum sulphide is filtered off, roasted in a silica crucible at 450°, and weighed as the oxide.

H. F. HARWOOD.

Separation of nickel and cobalt by means of hypochlorite. F. H. RHODES and H. J. HOSKING (Ind. Eng. Chem. [Anal.], 1930, 2, 164—166).—Nickel and

cobalt can be separated by fractional precipitation with sodium hypochlorite in a hot alkaline solution. By allowing sufficient time of contact between the precipitate and the hypochlorite it is possible to precipitate most of the cobalt free from nickel and *vice versa*. A small intermediate fraction is also produced which contains both elements. A semi-large-scale experiment showed that the method might be suitable for industrial operation.

H. INGLESON.

Corrosion and protective films. A. C. HANSON (Ind. Eng. Chem., 1930, 22, 554).—From visual observation of the progress of corrosion, caused by the contact of a drop of water saturated respectively with oxygen and carbon dioxide with polished steel, it is considered that electrolytic action begins in the presence of water with the formation of a film of ferrous hydroxide. This film is protective until carbon dioxide reacts to dissolve ferrous carbonate which later is precipitated in the form of ferric hydroxide with the recommencement of electrolytic action.

C. A. KING.

Cleaning of oily metal parts on the works' scale.

H. STADLINGER (Chem.-Ztg., 1930, 54, 354—355).—The preparation recommended for this purpose consists of a mixture of sodium silicate and trisodium phosphate, for use either in the form of powder or as a 3—4% aqueous solution. It has a powerful emulsifying action on grease of all kinds. It does not corrode aluminium, zinc, or their alloys, and is best applied as a hot spray.

C. IRWIN.

Series system of electrolytic copper refining at Nichols Copper Co. F. R. CORWIN and C. S. HARLOFF (Amer. Electrochem. Soc., May, 1930. Advance copy. 10 pp).—Details are given regarding the equipment and operation of the series system of copper refining at the plant of the Nichols Copper Co., one of the two plants where this system is still in use (cf. Merriss, Min. and Met., June, 1925; Robie, Eng. and Min. J., Feb., 1925). Wooden electrolysis tanks are being replaced by larger cells of concrete. The anode weight has been gradually increased and has now reached 45 kg. The current density has also been raised, the limiting values during 1929 being 2.25 and 2.63 amp./dm.² and the corresponding energy yields 138 and 123 kg./kw.-day, as compared with 109 kg./kw.-day in best practice by the multiple system. The proportion of anode scrap is only about half that in the multiple system and the amount of electrolyte to be circulated per ton of copper about a quarter of that used in the latter system. It is concluded that the plant installation cost for a given tonnage is notably lower for the series system, since less space is required, the power plant will be smaller, the copper tied up in conductors and bus-bars will be less, and the number of cells fewer. Labour costs are lower for the series system and the value of material tied up in process is less. The series system has now been developed to deal with less pure metal without lowering the quality of the product, and the precious metal loss has been controlled so that it compares favourably with that in the multiple system.

H. J. T. ELLINGHAM.

Bent-cathode test for control of cyanide copper baths. W. L. PINNER and E. M. BAKER (Amer. Electrochem. Soc., May, 1930. Advance copy. 7 pp).—

The bent-cathode test previously developed as a means of controlling chromium-plating baths (B., 1929, 480) is now applied to cyanide copper-plating baths. The effects of varying the ratio of copper to free cyanide, the concentrations of sodium carbonate and thiosulphate, the temperature, and the mean cathodic current density on the appearance of the deposit on the bent cathode have been investigated. By means of the test the onset of unsatisfactory plating conditions can be anticipated and hence avoided.

H. J. T. ELLINGHAM.

Electrodeposition of copper-nickel alloys. L. E. STOUT, O. G. BURCH, and A. S. LANGSDORF (Amer. Electrochem. Soc., May, 1930. Advance copy. 15 pp.).—Determinations have been made of the composition of copper-nickel alloys electrolytically deposited under various conditions from solutions of the complex cyanides of the two metals. The ratio of copper to nickel in the deposit is always greater than that in the bath. Increase in the copper content of the bath increases the copper content of the deposit, but to a much smaller extent than corresponds with the change in metal ratio of the bath. The ratio of copper to nickel in the deposit increases linearly with rise in temperature and also increases with rise in current density. Empirical formulæ representing the effects of these factors on the composition of the deposit are given. Increasing the free cyanide concentration in the bath lowers the current efficiency of deposition.

Cadmium as a plating material. H. KURREIN (Chem.-Ztg., 1930, 54, 333—335).—An account is given of the properties and uses of deposited cadmium and of the methods of deposition, with a *résumé* of recent literature.

S. I. LEVY.

Production of continuous and seamless tubing by electrodeposition. J. BILLITER (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.).—The metal is deposited on a short, slightly tapered mandrel of chrome or silicon steel. At regular time intervals the deposit is pulled along the mandrel so as to expose a short section of the latter on which is formed a new deposit joined to that previously produced. Continuous tubes of copper, zinc, and iron have been made in this way. In a second method the metal is deposited on the exterior of a continuous lead tube, which is passed slowly through the bath. The lead may afterwards be melted out.

H. J. T. ELLINGHAM.

Preparation of metal powders by electrolysis of fused salts. I. Ductile uranium. F. H. DRIGGS and W. C. LILLIENDAHL (Ind. Eng. Chem., 1930, 22, 516—519).—Uranyl salts are unsatisfactory for electrolytic reduction since they appear to ionise as the bivalent uranyl ion and deposit the dioxide. Salts of quadrivalent uranium, e.g., uranous chloride and potassium uranous fluoride, yield the quadrivalent uranium ion and deposit the metal. A quantity was prepared from potassium uranous fluoride (KUF_3) in a molten mixture of equal parts of sodium and calcium chlorides, using a carbon crucible as anode and a molybdenum wire as cathode. As removed on the cathode the spongy mass was protected from oxidation by the cooled salts, which were dissolved, and the metal was washed and dried.

Uranium thus prepared was silver-grey in colour and only the finer particles tended to ignite spontaneously, but pellets pressed from the grains ignited more readily. The carefully fused metal drained from any impurities contained 99.9% U, 0.055% Fe, 0.06% C, and 0.015% Si. Hydrochloric acid dissolved the metal readily, sulphuric acid more slowly, whilst nitric and acetic acids appeared to have negligible effect. Properties of uranium include d 18.9 (fused) and 19.07 (rolled), m.p. 1690°, sp. resistance 32.1×10^{-6} ohm per cm.³, hardness (fused) B92—94 (Rockwell).

C. A. KING.

Indium: recovery by electrodeposition. L. R. WESTBROOK (Amer. Electrochem. Soc., May, 1930. Advance copy. 7 pp.).—In the purification of liquors obtained in the acid leaching of roasted zinc ores indium is precipitated with cadmium and nobler metals by treatment with zinc dust. In working up this precipitated sponge for cadmium, indium is removed with the other impurities and can be partially separated from the latter by adding specially pure zinc shavings to their sulphate solution. The crude indium was purified by successive precipitations as hydroxide, and an oxide of 99.5% purity (In_2O_3) was eventually obtained. 200 G. of this oxide were dissolved in 600 c.c. of water containing 120 c.c. of 96% sulphuric acid, 250 g. of sodium citrate added, and the solution was made up to 1 litre. This solution was electrolysed at room temperature, using a platinum anode and an indium or steel cathode at a cathodic current density of 2 amp./dm.² The indium deposit was compact and could be built up to 1 in. or more in thickness. Over 1 lb. of the pure metal was recovered in this way. The electrodeposition of indium from other types of bath was investigated and previous work on the subject is reviewed.

H. J. T. ELLINGHAM.

Status of electrolysis as a metallurgical process. T. H. DONAHUE (Amer. Electrochem. Soc., May, 1930. Advance copy. 20 pp.).—Dominant and alternative metallurgical processes for the extraction and refining of the principal metals are tabulated, and statistics are collected to show the extent to which electrolysis is employed in the extraction of copper, zinc, and cadmium, and in the refining of copper, lead, nickel, and silver. The importance of the recovery of precious metals in the economy of electrolytic processes is discussed. Consideration is given to other processes such as treatment with chlorine, cementation on iron, and physical methods of separation, which are actual or possible competitors with the electrolytic processes, and the future of electrolysis in metallurgy is discussed in the light of these considerations. Expansion in the electrolytic extraction of zinc and refining of copper is expected, and electrolytic nickel refining is increasing rapidly in importance. Factors limiting the development of electrolytic copper extraction and lead refining are discussed, and the employment of electrolytic methods in the refining of tin, antimony, cobalt, iron, and the rarer metals is referred to. The possibility of the electrolytic production of oxides or basic salts of metals which cannot be electrodeposited in the free state is considered.

H. J. T. ELLINGHAM.

Metallurgical coke. PURVES. **Submerged com-**

bustion. HAMMOND.—See II. Ajax-Wyatt induction furnace. ADAM.—See XI.

See also A., June, 669, Recrystallisation of metals (BERGER). 670, Testing of monocrystalline wires (DE HAAS and VAN ALPHEN). 680, Nickel-chromium system (MATSUNAGA). 681, System chromium-carbon (WESTGREN and PHRAGMÉN). X-Ray analysis of iron and iron-manganese alloys (SHIMURA and WADA). Barium-tin alloys (RAY and THOMPSON). Silver-cadmium alloys (FRAENKEL and WOLF). System bismuth-tellurium (KÖRBER and HASCHIMOTO). 682, Diffusion of molybdenum and iron in the solid state (GRUBE and LIEBERWIRTH). System aluminium-magnesium-zinc (NISHIHARA). 707, Electrodeposition of copper (MARIE and GÉRARD; FOERSTER and GÄBLER). Deposition potential of alumina (DROSSBACH). 715, Preparation of thallium (ANDRIEUX). 719, Behaviour of silver and of silver-copper alloys on ignition in oxygen (LEROUX and RAUB). Preparation of definitely oriented single crystals of metals (GLOCKER and GRAF).

PATENTS.

Metallurgical furnace. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 316,986, 15.2.29. U.S., 11.5.28.).—The indirect heating of a reducing chamber situated above a smelting furnace is effected by completing the combustion of the furnace gases in a combustion chamber and passing the hot gases through a number of tubes which pass through the reduction chamber. Alternatively, the tubes may be lengthened and heated externally in the combustion chamber, in which case air is passed through the tubes to be later used for primary or secondary combustion.

C. A. KING.

Apparatus for reducing ores. G. S. HOWELL (U.S.P. 1,747,716, 18.2.30. Appl., 14.11.28).—A reducing shaft furnace has a funnel-shaped base which acts as a bosh and into which tuyères are fitted. Smelted material flows through the bottom into a lower chamber which acts essentially as a separator for slag and metal which may be drawn off at different levels. Suitable means for condensing fumes from the smelting are provided.

C. A. KING.

Ore-reduction furnace [for mercury ores]. J. M. SMITH (U.S.P. 1,747,740, 18.2.30. Appl., 5.6.28).—The furnace consists of a lower circular chamber surmounted by a cupola. A circular table of slightly frusto-conical shape forms the roasting bed in the lower chamber and is fed from a series of superposed, hollow, conical baffles in the cupola above. During operation the roasting table rotates and discharges the hot residue down an exit pipe, which serves as an entry for air for combustion.

C. A. KING.

Blast furnace. H. KOPPERS (U.S.P. 1,746,705, 11.2.30. Appl., 29.9.22. Renewed 1.3.27).—The uppermost part of a blast furnace consists of a double-walled steel cylinder in line with the refractory walls below. The jacket is water-cooled and the inner wall preferably corrugated to strengthen it against the wear of the charge. An expansion joint between this portion and the brickwork below is made by flanged plates angularly disposed and packed with asbestos.

C. A. KING.

Utilisation of the heat of slag. COMP. DES FORGES DE CHATILLON-COMMENTRY ET NEUVES-MAISONS (B.P. 327,732, 10.1.29. Fr., 6.12.28).—Hot slag cast into blocks is introduced into either a vertical or horizontal chamber provided also with a discharge opening, and a fluid is circulated through the chamber in countercurrent to the movement of the slag.

C. A. KING.

Treatment of ferrotitanium [ilmenite] ores. G. J. BANCROFT (U.S.P. 1,745,732, 4.2.30. Appl., 19.2.29).—Titaniferous iron ores are ground with fluorspar and carbonaceous material and the mixture is heated in a reducing atmosphere to produce iron sponge and calcium fluotitanate. The reduced mass is discharged through steam into water containing a small proportion of sodium hydroxide, and the granular product is ground wet, screened to remove coarse iron, and concentrated on Wilfley tables or by means of a magnetic separator.

A. R. POWELL.

Dephosphorisation of iron. P. RIES and F. BICHE-ROUX (B.P. 310,468, 11.4.29. Belg., 26.4.28).—The lime is introduced into the converter partly as lumps at the beginning of the blow and partly as powdered lime in the blast during the decarburisation stage of the blow. The lime-laden air-blast is supplied at the bottom of the converter from a chamber which is provided with a tangential entry for the air and which communicates with the bottom tuyères by way of a plate having counterbored inlets to the tuyères. The bottom of the converter is closed with a cover pressed up to a conical shape on the inner side to prevent accumulation of lime powder in the feed chamber.

A. R. POWELL.

Pickling of iron and steel. VER. F. CHEM. U. MET. PROD. (B.P. 312,996, 29.5.29. Czechoslov., 4.6.28).—A small quantity of a sulphonation product of crude carbazole or an alkali or alkaline-earth sulphonate derived therefrom is added to the pickling bath as an inhibitor.

A. R. POWELL.

Recovery of nickel and other metals from ores containing nickel. F. L. DUFFIELD (B.P. 328,696, 8.2.29).—The ore is roasted at 750–1000° until conversion of the iron into oxide and the nickel and copper into sulphates occurs, sodium, magnesium, or calcium chloride is then added, and the mixture heated at 650° to convert the nickel and copper into chlorides which are subsequently removed by raising the temperature to cause them to volatilise. The residual iron oxide is reduced with a gaseous reducing agent and separated from the gangue magnetically. The volatilised chlorides are dissolved in water and separated by chemical or electrolytic means.

A. R. POWELL.

Carbonisation of nickel and nickel alloys. WESTINGHOUSE ELECTRIC & MANUF. CO., Assees. of H. MCK. ELSEY (B.P. 305,465, 25.1.29. U.S., 4.2.28).—Nickel or nichrome for the manufacture of radio-valve grids is oxidised by heating in the air, then carbonised by heating at 700–1000° in an atmosphere of nitrogen and a hydrocarbon gas free from hydrogen.

A. R. POWELL.

Treating [lead sulphate] ores and the like. F. S. MULOCK, Assr. to U.S. SMELTING, REFINING, & MINING CO. (U.S.P. 1,745,945, 4.2.30. Appl., 8.1.24).—The ore

(native lead sulphate or roasted sulphide) is leached at 100° with hot, acid brine containing sufficient calcium chloride to convert all the sulphate present into calcium sulphate. After filtration the solution is cooled to 0°, preferably after dilution with 5–25% of its volume of water, whereby pure lead chloride is obtained. This is separated, dried, and smelted with lime, sodium chloride, and carbon to produce metallic lead and calcium chloride for use again in the process. The mother-liquor from the lead chloride is treated with lime to precipitate other metals which may be present and the clarified solution is re-acidified for use again.

A. R. POWELL.

[Lead bearing-metal] alloy. J. V. O. PALM, Assr. to CLEVELAND GRAPHITE BRONZE Co. (U.S.P. 1,741,733, 31.12.29. Appl., 30.9.26).—An alloy of 70–75% Pb, 5–6% Cu, 4% Hg, 4–8% Sb, and about 12% Sn is claimed; it has a fine crystal structure and increases in hardness and strength after 24 hrs. at the ordinary temperature.

A. R. POWELL.

Welding lead. W. SMITH, and EXPANDED METAL Co., LTD. (B.P. 329,023, 11.2.29. Addn. to B.P. 285,995; B., 1928, 373).—The surfaces to be joined are amalgamated and then welded together by running a fan-tailed gas flame along the joint. [Stat. ref.]

A. R. POWELL.

Flux [for autogenous welding]. W. B. MILLER and A. R. LYTLE, Assrs. to ONWELD ACETYLENE Co. (U.S.P. 1,748,785, 25.2.30. Appl., 28.8.28).—The flux, particularly adapted for use with aluminium bronzes, consists of 10–50% of a fluoride (e.g., of an alkali metal) and a borosilicate glass, preferably containing more boric acid than silica. The fluoride is added either to the powdered or the molten glass, and the mixture is pulverised after cooling.

S. K. TWEEDY.

Obtaining chromium [powder]. M. N. RICH, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,741,955, 31.12.29. Appl., 9.11.27).—Chromic oxide obtained by ignition of ammonium dichromate is heated at 900° in a rapid current of hydrogen which has been rigorously purified from oxygen, nitrogen, hydrocarbons, and water vapour. The product is suitable for pressing and sintering into bars which may be swayed and drawn into wire.

A. R. POWELL.

[Chromium-nickel] alloys. INTERNAT. NICKEL Co., and P. D. MERICA (B.P. 328,362, 16.5.29).—Nickel alloys containing 0.1–10% Fe, 10–20% Cr, 0.1–3% Mn, 0.03–0.5% C, and 0.1–5% Al, 0.03–0.15% Mg, or 0.03–0.15% Ca are claimed. The aluminium, magnesium, or calcium is added just prior to casting to effect deoxidation and to ensure the production of an alloy which may readily be hot-worked, rolled, or forged.

A. R. POWELL.

Tungsten-tantalum alloy. J. H. RAMAGE, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,741,953, 31.12.29. Appl., 28.5.27).—Tungsten alloys with 0.5–1.5% Ta for the manufacture of lamp filaments are prepared by precipitating a mixture of potassium tungstate and tantalate with a slight excess of hydrochloric acid and heating the mixture with 0.15–0.4% of lampblack for 3 hrs. in a current of coal gas at 800°, then for 5–6 hrs. at 1200–1400° in a current of hydrogen until the carbon is completely removed.

A. R. POWELL.

Silver extraction [from ores]. E. HAEHRE, Assr. to T. ROBERTS & Co. (U.S.P. 1,747,072, 11.2.30. Appl., 15.2.28. Norw., 19.9.27).—Ores containing native silver are agitated in a pulp containing an alkali sulphide and the mixture is heated with steam to convert the silver wholly or superficially into sulphide, which is then separated by flotation.

A. R. POWELL.

Treatment of platinum ores, concentrates, etc. containing chromite. A. R. POWELL, E. C. DEERING, and JOHNSON, MATTHEY, & Co., LTD. (B.P. 328,564, 28.1.29).—Chromite concentrates containing platinum are roasted with lime and litharge to produce lead chromate and ferric oxide, silica is added, and the roasting continued until a sintered, granular mass is obtained, and finally this mass is smelted in a lead blast-furnace to obtain platiniferous lead bullion from which the precious metals are recovered by cupellation. Alternatively the concentrates are roasted with lime and sodium carbonate and the product, with or without a previous leaching to remove soluble chromates, is smelted with matte-forming materials to produce a matte from which the platinum is recovered as described in B.P. 316,063 (B., 1929, 822).

A. R. POWELL.

Flotation apparatus. J. P. RUTH, JUN. (U.S.P. 1,746,682, 11.2.30. Appl., 8.10.27).—An elongated tank having sloping sides or a concave bottom is divided in the upper part into three sections by two vertical, perforated walls. Compressed air is injected near the bottom between two L-shaped partitions extending the length of the tank below the normal pulp level and the froth is carried over into launders.

C. A. KING.

Metal polishes. W. HESSELGRAVE (B.P. 327,783, 26.1.29).—Precipitated chalk and turpentine are mixed to form a semi-liquid mass, ammonia solution is added, and the whole diluted with water.

H. ROYAL-DAWSON.

Method and means for electrically heating metals. F. W. THOMPSON (B.P. 328,270, 18.10.28).—Metal articles are heated electrically at a predetermined rate to a definite temperature; the applied voltage is then automatically changed to a lower value by operation of a switch controlled by the resistance of the article, so that the heating is continued at a different rate.

J. S. G. THOMAS.

Manufacture of electrolytic iron. R. D. PIKE (U.S.P. 1,746,903, 11.2.30. Appl., 4.1.26).—Sponge iron produced by the reduction of leached pyrite cinders is dissolved in a solution of ferrous and ferric chlorides containing 8–10% Fe and the solution is purified from copper and zinc by treatment with scrap iron and hydrogen sulphide. The purified solution is then electrolysed at 70° in a divided cell to obtain electrolytic iron and an anolyte containing ferric chloride which is returned to the leaching tanks.

A. R. POWELL.

Electrodeposition of a coating of zinc [on iron or steel]. Q. MARINO (B.P. 305,035, 28.1.29. Fr., 28.1.28).—The electrolyte comprises a solution of 200–250 g. of zinc sulphate, 20–25 g. of aluminium sulphate, 50–60 g. of zinc chloride, and 20–50 g. of sodium or potassium borobenzoate per litre.

A. R. POWELL.

Cleansing or scouring iron and steel by electrochemical means. Q. MARINO (B.P. 305,036, 28.1.29. Fr., 28.1.28).—The bath comprises a solution of sodium or potassium sulphate in the liquor obtained by treating calcium phosphate with sulphuric acid (*d* 1.1—1.2). The article is made first the cathode, then the anode.

A. R. POWELL.

Protection of cuprous materials. I. T. BENNETT, ASSR. to T. E., J. B., T. E., JUN., and J. F. MURRAY (U.S.P. 1,746,987, 11.2.30. Appl., 6.3.25).—Copper is plated first with iron, then with chromium, and finally with nickel, and the whole is heated at 850—900° for 4—5 hrs. to cause the three plated metals to diffuse into one another to make a corrosion-resistant nickel-chromium-iron alloy coating on the copper.

A. R. POWELL.

Electrolytic production of insulating layers on articles of aluminium and its alloys. E. W. KÜTTNER (B.P. 329,190, 6.6.29).—The articles are subjected to anodic oxidation in a bath containing oxalic acid and a strong mineral acid, *e.g.*, hydrochloric acid, or a powerful oxidising agent, *e.g.*, potassium permanganate or dichromate or derivatives thereof.

A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 328,574, 29.1.29).—A salt mixture for the preparation of a cadmium-plating bath consists of 2.5—3 oz. of cadmium oxide, 6 oz. of sodium hydrogen sulphate, 4 oz. of ammonium sulphate, and 1—10 g. of a sugar, preferably cane-sugar caramel. This mixture is dissolved in 1 gal. (Amer.) of water for use in the bath.

A. R. POWELL.

Manufacture of pure iron. (A—C) A. MITTASCH and C. MÜLLER, (A, C) E. LINCKH, and (B) W. SCHUBARDT, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,759,658—1,759,660, 20.5.30. Appl., [A] 21.12.25, [B] 4.3.26, [C] 2.7.27. Ger., [A] 15.12.24, [B] 23.5.25, [C] 5.7.26).—See B.P. 262,938, 269,677, and 281,963; B., 1927, 144, 527; 1928, 127.

Finely-divided metals from metal carbonyls. C. MÜLLER and W. SCHUBARDT, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,759,661, 20.5.30. Appl., 2.7.27. Ger., 6.7.26).—See B.P. 284,087; B., 1928, 270.

Working up iron ores in a direct way. P. H. LEDEBOER, ASSR. to STAALSYNDICAAT DR. LEDEBOER II (U.S.P. 1,759,753, 20.5.30. Appl., 17.3.28. Holl., 30.3.27).—See B.P. 287,925; B., 1929, 984.

Blast furnace for reduction and recovery of volatilisable metals as oxides and their operation. W. JOB, ASSR. to AMER. LURGI CORP. (U.S.P. 1,759,559, 20.5.30. Appl., 29.6.26. Ger., 2.7.25).—See B.P. 268,201; B., 1927, 415.

Removal of incrustation formed on metallic surfaces. R. SELIGMAN (U.S.P. 1,759,762, 20.5.30. Appl., 1.8.27. U.K., 4.11.26).—See B.P. 284,778; B., 1928, 271.

Enamelling furnace (B.P. 308,613). Separation of materials (B.P. 328,283).—See I. Joining metal to wood etc. (B.P. 303,855).—See V. Treating mixtures of alkali stannates, arsenates, and antimonates (U.S.P. 1,747,709). Lyes containing copper and

zinc (B.P. 329,225). Recovering vanadium from solutions (U.S.P. 1,747,403).—See VII. Magnesite bricks (U.S.P. 1,747,905).—See VIII. Furnace for iron ores (U.S.P. 1,748,805). Controlling electro-deposition (B.P. 328,057).—See XI.

XI.—ELECTROTECHNICS.

The Ajax-Wyatt or vertical-ring induction furnace. W. ADAM, JUN. (Amer. Electrochem. Soc., May, 1930. Advance copy. 22 pp.).—The ideal characteristics of an electric furnace for melting non-ferrous metals and alloys are set out and the extent to which these characteristics are possessed by the vertical-ring induction furnace is discussed in detail. The electrical and thermal efficiencies may be as high as 85%. Efficient circulation of the metal produced by the combined action of the motor effect, the pinch effect, and the Joule effect ensures homogeneity of the melt and avoids local overheating, thus keeping metal losses very low. Since the heat is produced in the metal itself, there is no heat lag. The necessity of keeping a "heel" of molten metal in the ring is a disadvantage in changing over from one alloy to another. The power factor is high, and current surges cannot occur. Operating data are given, including a typical melting cost analysis: an induction furnace working 9 hrs. per day, making pressure castings of red brass. Furnaces are available with power inputs ranging from 45 to 150 kw., and pouring capacities from 400 to 2200 lb. per heat.

H. J. T. ELLINGHAM.

Contact rectification. I. Classification of contact rectifiers. M. BERGSTEIN (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.).—The literature on contact rectifiers is reviewed. Such rectifiers may be divided into integral junctions, in which the dissimilar electrodes are formed on one another, as in the copper-cuprous oxide rectifier, and non-integral junctions, where the electrodes are brought into intimate contact by pressure, as in the cupric sulphide-magnesium rectifier. Further classification is based on the chemical character of the electrodes. In general, rectified current flows from the electronegative to the electropositive member of the junction, but in some cases the reverse is reported. For the cupric sulphide-magnesium rectifier this reversal is shown to be related to the voltage across the junction. H. J. T. ELLINGHAM.

Magnetic concentration of certain natural and artificial manganese dioxides. T. J. MARTIN (Amer. Electrochem. Soc., May, 1930. Advance copy. 13 pp.).—Manganese dioxide is weakly magnetic, and this property has been utilised in concentrating pyrolusite ores, particularly in obtaining the grades of dioxide required for dry cells. Concentration tests at various magnetic intensities have now been carried out on a number of natural and artificial manganese dioxides ground to various degrees of fineness. Highly magnetic impurities can be separated, but efficient separation of weakly magnetic impurities is not always possible at the flux densities employed. Efficient separation is less possible with finely-ground than with coarsely-ground ores. Even when a manganese dioxide product separates well, no predictions as to the composition of the fractions

can be made. Artificial manganese dioxides differ as widely from each other in magnetic susceptibility as do the ores. The possible cause of these differences is discussed. H. J. T. ELLINGHAM.

Humidity recorder. ROSECRANS; WALKER and ERNST. **Ultrafiltration funnels.** ZINZADZE.—See I. **Maizolith.** HARTFORD.—See V. **Splitting of mica.** OBRIMOV.—See VII. **Abrasives.** NOBES.—See VIII. **Strontium-tin alloys.** RAY. **Analysis of white metals and solder.** MATTHIJSEN. **Copper refining.** CORWIN and HARLOFF. **Control of cyanide copper baths.** PINNER and BAKER. **Copper-nickel alloys.** STOUT and others. **Cadmium plate.** KURREIN. **Seamless tubing.** BILLITER. **Uranium.** DRIGGS and LILLIENDAHL. **Indium.** WESTBROOK. **Electrolysis in metallurgy.** DONAHUE.—See X.

See also A., June, 681, **Electrical conductivity etc. of lead-antimony alloys** (STEPHENS). 707, **Electrodeposition of copper** (MARIE and GÉRARD; FOERSTER and GÄBLER). **Deposition potential of alumina** (DROSSBACH). **Mechanism of a simple voltaic cell** (LATTEY and PERKIN). **Mercury-basic mercury sulphate cell** (VOSBURGH and LACKEY). **Pyrochemical Daniell cells** (OPPENHEIMER). 715, **Preparation of arsine and stibine** (HLASKO and MASLOWSKI). **Preparation of thallium** (ANDRIEUX). 725, **Analysis of nitrating acid** (MÜLLER and KOGERT).

PATENTS.

Induction furnaces. P. E. BUNET, and ACIÉRIES DE GENNEVILLIERS (B.P. 306,144, 14.2.29. Fr., 17.2.28).—A very low-resistance, air- or water-cooled, primary circuit is arranged as close as possible to the secondary circuit and is fed with current at a voltage which is substantially equal to that desired in the secondary, or is a low multiple thereof. J. S. G. THOMAS.

Electric furnace [for smelting iron ores]. A. STANSFIELD (U.S.P. 1,748,805, 25.2.30. Appl., 22.3.29).—The charge is partly reduced and prepared for final treatment in the furnace, by gases given off in the furnace as the charge descends in thin layers over a series of inclined surfaces over which the gases flow in counter-current to the charge. J. S. G. THOMAS.

Electric torch furnace [for melting ceramic materials, especially mullite]. T. S. CURTIS, Assr. to VITREFRAX CORP. (U.S.P. 1,747,756, 18.2.30. Appl., 30.7.28).—A preheated bar of ceramic material is fed in line with, but in the opposite direction to, one of a pair of electrodes arranged at right angles, and provided below with quenching means, and is fused by the arc between the electrodes. J. S. G. THOMAS.

Electric furnace for continuous production of cyanogen compounds. O. STALHANE (B.P. 318,548, 28.8.29. Swed., 5.9.28).—In a furnace constructed in accordance with B.P. 272,996 (B., 1927, 602), comprising an inner and an outer tube, insulated at one end and in metallic connexion at the other, current is supplied from a source of two- or poly-phase current to both tubes at the insulated end. One end of the inner tube is connected to the earth-connected zero-point of the current system. J. S. G. THOMAS.

Current-rectifying device. W. O. SNELLING (U.S.P. 1,745,723, 4.2.30. Appl., 24.2.23).—A homogeneous mixture of copper sulphide and lead sulphide is claimed. J. S. G. THOMAS.

Alternating-current rectifying element. S. J. M. ALLEN (U.S.P. 1,746,511, 11.2.30. Appl., 26.5.27).—A copper plate, coated with copper oxide and heated to 650–705°, is quenched in an inert liquid, *e.g.*, water. J. S. G. THOMAS.

Rectifying device and its production. W. D. DOOLEY (U.S.P. 1,748,012, 18.2.30. Appl., 12.9.28).—An aluminium plate coated electrolytically with a film of oxide is heated at 150–280° and exposed to vapours, whereby a superposed coating of aluminium sulphate is formed. The plate is then heated until by decomposition a second film of oxide is formed upon the first. J. S. G. THOMAS.

Forming copper-copper oxide rectifier plates. A. K. CROAD. From HANOVIA CHEM. & MANUF. Co. (B.P. 328,701, 11.2.29).—A prepared copper plate heated to a high temperature in an oxidising atmosphere is quenched by the vapour of an active substance, *e.g.*, quinol, in an inactive base liquid, *e.g.*, liquid paraffin wax, at between 70° and 169°. J. S. G. THOMAS.

[Controlling electrodeposition in] electrolytic processes. K. BREUSING and U. GOTTESMANN (B.P. 328,057, 30.1.29).—The direction and density of current flow are controlled by magnetic, electromagnetic, and/or electrostatic means. J. S. G. THOMAS.

[Wehnelt cathode for] electric-discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 328,641, 1.1.29).—A coherent, relatively thin sheath of tungsten is formed on a core of a material, *e.g.*, molybdenum, which becomes brittle at a higher temperature than tungsten. If desired, thorium oxide or other material of high electronic emission may be incorporated in the core. J. S. G. THOMAS.

Source of light. [Glow-discharge gas-filled lamp.] T. W. CASE, Assr. to CASE RES. LAB., INC. (U.S.P. 1,747,287, 18.2.30. Appl., 19.11.27. Renewed 9.7.29).—The gas-filling of a glow-discharge lamp having a pair of spaced electrodes, the negative one being coated with an alkaline-earth metal, preferably barium oxide, consists of a mixture of hydrogen with 1–25% (preferably 5% or less) of nitrogen. J. S. G. THOMAS.

Electrical method and apparatus. [Electrical conductor.] J. KREMER (U.S.P. 1,748,927, 25.2.30. Appl., 1.12.25. Renewed 10.9.29).—Current is passed through molten sodium maintained in circulation in a conduit provided with cooling means. J. S. G. THOMAS.

[Cooling and insulating medium for] electrical apparatus. L. R. WILDER (U.S.P. 1,746,977, 11.2.30. Appl., 6.11.26).—The use of diphenyl ether, maintained in liquid form by a heating coil, is claimed. J. S. G. THOMAS.

Insulating material for electric condensers etc. J. E. G. LAHOUSSE (B.P. 329,604, 10.10.29. Fr., 22.10.28).—Paper is impregnated with diphenyl ether or its equivalent, *e.g.*, benzyl oxide, either alone or containing dissolved wax, resin, resinsates, amides, *e.g.*, acetanilide, urea, etc. J. S. G. THOMAS.

Electrical insulating materials. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HADGOOD, and H. C. CHANNON (B.P. 328,025, 14.11.28).—For submarine telephones or cables, a mixture of dirt-free gutta-percha and/or balata, with rubber from which all proteins and other non-rubber compounds have been removed, is claimed. (Cf. B.P. 307,390; B., 1929, 400.)

J. S. G. THOMAS.

[Electrodes for] electric accumulators. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 329,514, 22.5.29).—A combination of porous lead anodes produced by eliminating admixed metal from an alloy of lead with an alkali or alkaline-earth metal, and cathodes consisting of the usual commercial negative electrodes is claimed.

J. S. G. THOMAS.

Manufacture of active masses for [alkaline] electric accumulators. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 328,096, 22.3.29).—A mixture composed of the active material and a highly viscous liquid soluble in water, *e.g.*, a mixture composed of nickel hydroxide (80 pts.), graphite (20 pts.), and glycerin (15 pts.), is pressed by a matrix having a relatively long extension orifice into the form of a band, thread, or small tube, which is subdivided, worked up into the desired shapes, and pressed directly into containers.

J. S. G. THOMAS.

Salvaging of storage battery plates. C. B. WHITE (B.P. 328,123, 30.4.29).—The plates are heated so as to liquefy only the metal (antimonial lead) forming the grid frame, which is collected; the mixture of hot, active or filler material and enclosed liquid metal is then disintegrated by projection upon a rough cooler surface and collected, the metal thereby adhering to the roughened surface.

J. S. G. THOMAS.

Irradiation of substances. E. BUIITZ (B.P. 328,410, 3.4.29).—Suspensions or fine powders in thin layers to the centre of a flat, slowly rotating disc are irradiated by a source, preferably annular in shape, arranged above the disc. The process is applicable to milk.

J. S. G. THOMAS.

Stabilisation of non-crystalline, piezo-electric microphone plates. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 316,628, 1.8.29. Ger., 1.8.28).—A direct-current potential is continuously applied to the electrodes of plates produced by the action of high-tension direct current upon a cooling mass of resin, wax, etc.

J. S. G. THOMAS.

Manufacture of laminated magnetic bodies and the like. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRICITÄTS-GES. (B.P. 314,084, 20.6.29. Ger., 22.6.28).—Metal laminations are held together by reaction products of polyhydric alcohols and polybasic acids or their respective anhydrides.

J. S. G. THOMAS.

Electrode compositions for electron-discharge devices. S. DUSHMAN, ASSR. to GEN. ELECTRIC CO. (U.S.P. 1,747,061—4, 11.2.30. Appl., 2.7.23).—See B.P. 218,630; B., 1925, 794.

Electric-discharge tubes. N.V. PHILIPS' GLOEI-LAMPENFABR. (B.P. 309,584, 26.3.29. Holl., 13.4.28).

[Envelope for] electric-discharge tubes. M.-O. VALVE CO., LTD., and W. H. PETERS (B.P. 328,763, 9.4.29).

Electron-discharge devices. CLEARTRON (1927), LTD., and J. R. SINCLAIR (B.P. 329,802, 27.4.29).

[Electrodes for] luminous electric-discharge tubes. A. E. CHAPMAN and J. VIELLE (B.P. 329,639, 18.12.28).

Long-duration arc-lamps. SIEMENS-PLANIAWERKE A.-G. F. KOHLEFABRIKATE (B.P. 328,071, 18.2.29. Ger., 16.11.28).

[Reducing the tendency to "flashing" of] gas-filled electric incandescence lamps. GEN. ELECTRIC CO., LTD., J. T. RANDALL, and L. C. JESTY (B.P. 329,446, 26.3.29).

Construction of photo-electric cells. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 328,451, 4.5.29).

Construction of light-sensitive [selenium] cells. A. E. JONES (B.P. 327,674, 8.10.28).

[Accumulators or other] galvanic batteries. OLDHAM & SON, LTD., and W. D. WILDE (B.P. 329,831, 15.5.29).

Pasting of grid strips for the manufacture of accumulator plates. FRIEMANN & WOLF GES.M.B.H. (B.P. 329,889, 8.7.29. Ger., 16.3.29).

Heat for chemical reactions (B.P. 328,635). Temperature regulator (B.P. 299,714).—See I. Marking material by electrodeposition (B.P. 329,258).—See V. Phosphoric acid (B.P. 319,656). Acid-resisting materials (U.S.P. 1,732,140). Treating mixtures of alkali stannates etc. (U.S.P. 1,747,709). Fluorine (B.P. 315,768—9).—See VII. Nickel etc. from ores (B.P. 328,696). Carbonisation of nickel (B.P. 305,465). Tungsten-tantalum alloy (U.S.P. 1,741,953). Heating of metals (B.P. 328,270). Electrolytic iron (U.S.P. 1,746,903). Coating zinc on iron or steel (B.P. 305,035). Cleansing iron or steel (B.P. 305,036). Protecting cuprous materials (U.S.P. 1,746,987). Insulating layers on aluminium etc. (B.P. 329,190). Cadmium plating (B.P. 328,574).—See X. Rendering victuals radioactive (B.P. 329,292).—See XIX. Removing oxygen from water (G.P. 456,459).—See XXIII.

XII.—FATS; OILS; WAXES.

Detection of hardened fats. H. P. KAUFMANN and M. KELLER (Chem. Umschau, 1930, 37, 142—144).—By the use of Grossfeld's improved lead salt separation (B., 1930, 466) the authors were successful in detecting isooleic acid due to admixtures of 2% of hardened arachis and blubber oils, or 20% of hardened coconut oil; addition of 3% of hardened coconut oil or 5% of fully hardened maize oil (both of which contain very little isooleic acid) could not be detected by this method. The detection of nickel in milk chocolate cannot be taken as an indication of the presence of hardened fats (nickel content 0.001—0.01 mg./kg.), as 0.005—0.05 mg. of nickel per kg. was found in dried milk powder (cf. Martini, A., 1930, 565; Bertrand and Nakamura, A.,

1927, 992, whose figures for the occurrence of nickel in animal and vegetable materials are collected).

E. LEWKOWITSCH.

Determination of the iodine value of oils and fats by thermometric titration. T. SOMIYA (J. Soc. Chem. Ind., Japan, 1930, 33, 174—176 B).—The thermometric titration of oils etc. by solutions of chlorine in acetic acid and carbon tetrachloride must be conducted under standard conditions, the end-point being determined graphically (cf. B., 1929, 274). The values obtained were rather below those found by the usual methods.

E. LEWKOWITSCH.

Polymerised soya-bean oil and its soap. M. HIROSE and T. SHIMOMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 169—172 B).—The characteristics of soya-bean oil polymerised by heating at 290—295° in carbon dioxide are given. The rate of decrease of iodine value is considerably smaller than in the case of fish oils, but in both cases this rate decreases with the duration of heating. The yields of hexabromide and solid tetrabromide decrease to nil as polymerisation progresses, but the amount of viscous brownish-black bromide insoluble in light petroleum (which is regarded as an unsaturated polymerised product of linolenic acid) increases to an amount nearly equal to that of the hexabromide in the original oil. The increase in mol. wt. (Rast) of the fatty acids is smaller than that of the original glycerides. The surface tension of a solution of the soap increases, and the (Hillyer) drop number and the lathering power (Stiepel) decrease with increasing polymerisation. Thus polymerisation reduces the detergent power of the soap, but as the soap does not deteriorate on keeping and has remarkable tenacity it may with advantage be added to soaps of better washing power but of a brittle nature, e.g., hydrogenated soya-bean oil soap.

E. LEWKOWITSCH.

Spectrographic analysis of the fluorescence of some vegetable oils observed under ultra-violet rays. H. MARCELET (Compt. rend., 1930, 190, 1120—1122).—Olive, soya-bean, sesamé, maize, grape-seed, arachis, tea, cottonseed, and argan oils produce different emission and absorption spectra in ultra-violet radiation. The addition of 5% of olive-pulp oil to refined or virgin olive oil may be detected by means of the sharply defined regions characteristic of its emission spectrum. Mixtures of olive oil with 10% of soya-bean, maize, or grape-seed oil gave inconclusive results. J. GRANT.

Neutralisation of castor oils. M. T. FRANÇOIS (Compt. rend., 1930, 190, 1308—1310).—The acids present in commercial castor oils, which are detrimental to their use in aviation and to their keeping properties, can be neutralised by industrial "triethanolamine" (tri- β -hydroxyethylamine), consisting of a mixture of the corresponding primary (0.5%), secondary (20—25%), and tertiary (75—80%) amines, using methyl-orange as an indicator. The neutralised product has a slightly higher viscosity. A. I. VOGEL.

Composition of almond oil. Comparison of almond oil and apricot-kernel oil. A. HEIDUSCHKA and C. WIESEMANN (J. pr. Chem., 1930, [ii], 124, 240—260).—The following data have been obtained, the two figures referring to almond oil and apricot kernel oil, respectively,

in each case: d_{4}^{15} 0.9180, 0.9181; refractivity at 25° 65.3, 65.9, and at 40° 57.4, 58.0; $[\alpha]_D$ —0.007°, 0.007°; iodine value 99.36, 101.60; saponif. value 188.80, 190.15; Hehner value 94.22, 93.07; Reichert-Meissl value 0.00 0.06; Polenske value 0.00, 0.00; acid value 1.24, 3.26; unsaponifiable matter 0.50, 0.43%; glycerol (Shukov and Schestakov) 10.00, 10.73%; fatty acids: palmitic 3.10, 2.08; oleic 77.00, 79.39; linoleic 19.90, 18.53%; linolenic acid not detectable. A modification of Twitchell's lead salt-alcohol method for the determination of saturated acids is used in which the lead salt is crystallised three times from 95% alcohol; in this way the salts of unsaturated acids are completely removed without appreciable loss of saturated acids. The unsaturated acids are determined by bromination by Hazura's method. The behaviour of the two oils on oxidation in the air has also been examined by determining the refractivities and the iodine, acid, and oxidation values at intervals; only minor differences are observed. There are thus no readily detectable differences between the two oils, except the slight ones in taste and appearance, and the colour reactions, which are applicable only to fresh oils. R. K. CALLOW.

New consistometer and its application to greases and to oils at low temperatures. R. BULKLEY and F. G. BITNER (J. Rheology, 1930, 1, 269—282).—A consistometer is described, speedy in operation and suitable for routine determinations, which depends for its action on the measurement of flow through a capillary tube under a variable pressure-head of mercury. The apparatus is suitable for materials the consistency of which changes with time, since for each determination the volume of flow need not exceed 0.5 c.c. The accuracy claimed is $\pm 2\%$. Certain oils and greases behave as thixotropic gels and, after working, require several hours at 0° to attain a constant and reproducible state. By the use of this apparatus consistency-pressure curves may be obtained for materials in the completely worked and completely unworked condition. T. H. MORTON.

Determination of the so-called refined white montan wax in beeswax compositions. G. BUCHNER (Chem. Umschau, 1930, 37, 144—145).—The so-called refined montan "wax" consists almost entirely of free fatty (montanic) acids and unsaponifiable matter, and may be determined as follows (in a mixture, e.g., of stearic acid, beeswax, montan "wax," and paraffins): The acid value, ester value, and Buchner value (cf. B., 1911, 907) of the mixture are first determined, the last-named corresponding to the amount of stearic acid present, since the montanic acids are insoluble in the 80% alcohol used. The proportion of montan "wax" (acid value may be taken as 100) is calculated from the difference between the acid value actually found for the residue from the Buchner test and that corresponding (i.e., 1/5 of the original ester value) to the beeswax present. It is assumed that other saponifiable substances (e.g., tallow) which would influence the ester value are absent: their presence would be indicated if the separated paraffins were considerably greater in amount than that calculated (1 pt. of tallow appears by calculation as 2.6 pts. of beeswax).

E. LEWKOWITSCH.

Stability of emulsions. AUERBACH.—See I. **Submerged combustion.** HAMMOND. **Lubricating oils.** DAMIAN and DIXMIER.—See II. **Solvents.** FIFE and REID.—See III.

See also A., June, 692, **Systems soap-cresol-water** (ANGELESCO and POPESCU). 693, **Influence of soap solution on rate of saponification of triglycerides** (McBAIN and KAWAKAMI). 702, **Soap boiling** (McBAIN and others). 803, **Mixed fatty acids present in glycerides of cod-liver and other fish-liver oils** (GUHA and others). 804, **Insect oils and fats** (TIMON-DAVID). **Bull-frog oil** (IWAMOTA and KISEGAWA). **Saturated fatty acids of Japanese great-herring oil** (UENO and IKUTA). **Oils and fats of Japanese birds** (UENO and IKUTA).

PATENTS.

Manufacture of margarine. K. P. WANKLYN (B.P. 327,519, 21.2.29).—Wheat germ is dried in a deoxidised atmosphere and ground to a powder, or is crushed to produce an extract in the form of a pulp or jelly, and the powder or pulp is incorporated in margarine. E. LEWKOWITSCH.

Preservation of fats [in raw materials]. J. A. S. VAN DEURS (B.P. 303,059, 11.12.28. Denm., 27.12.27).—The p_H within the animal or vegetable tissues surrounding the fat is maintained at not above 5 during the period of preservation, by the addition of (aqueous) acid or acid-forming micro-organisms. E. LEWKOWITSCH.

Production of soaps possessing intensive detergent power. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 327,393, 1.1.29).—Unesterified monoalkyl ethers of the glycols or polyglycols are incorporated with soaps after or during the course of their manufacture; hydrocarbons or chlorinated hydrocarbons may be added as desired. Solid, pasty, or jelly-like soaps are formed which are almost odourless and are not injurious to the skin. E. LEWKOWITSCH.

Cooling of fatty substances in liquid condition. H. G. R. NAUMANN (B.P. 328,196, 22.12.28. Holl., 6.11.28).—The rotating cooling drum used consists of an outer and an inner (concentric) cylinder, interconnected by partitions; the enclosed annulus contains the circulating, evaporating, liquid refrigerant. E. LEWKOWITSCH.

Extraction of neutral grease from wool-scouring water and treating same. E. MERTENS (B.P. 328,606, 30.1.29. Belg., 6.11.28).—The wool-washing liquors are circulated through channels in which they are churned by pairs of paddle-wheels rotating in opposite directions; the scum collected by these is washed by spraying with water from atomisers, and is treated finally in a digester at 90–120° under 2–3 atm., yielding an almost anhydrous, odourless grease. E. LEWKOWITSCH.

Apparatus for separation of oil from oil-containing substances. F. KRUPP GRUSONWERK A.-G. (B.P. 297,816, 28.7.28. Ger., 30.9.27. Addn. to B.P. 272,118).—Claim is made for an arrangement of a series of horizontal presses. A. R. POWELL.

Production of oil from palm fruit and other oil seeds. VEGETABLE OIL MACHINERY SYND., LTD., and

J. MOORES (B.P. 328,734, 14.3.29).—The fruit is sterilised by cooking out of contact with water in a boiler fitted with a false bottom and pressed, the oil obtained being heated to about 100° and filtered; if desired, the fruits may be depulped after sterilising, the mashed pulp being re-cooked before pressing. E. LEWKOWITSCH.

Decoloration of [palm] oils. CO-OPERATIVE WHOLESALE SOC., LTD., A. GLOVER, and C. W. COUCHE (B.P. 327,990, 14.12.28).—Palm oil is bleached by heating with the exclusion of air (*in vacuo*, but without atomisation, steam-injection, etc.) at 200–300° until the oil may be saponified without return of the yellow colour; fatty acids in the oil are retained. E. LEWKOWITSCH.

Deacidification and bleaching of oils and fats. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,276, 24.12.28).—The oil, which has been mixed with a finely-divided adsorption agent, *e.g.*, fuller's earth or carbon, is subjected to a distillation treatment at elevated temperatures with wet steam, or a mist of finely-divided liquids, *e.g.*, alcohol or benzene, dispersed in an inert gas (cf. B.P. 213,267 and 235,792; B., 1925, 619, 680). E. LEWKOWITSCH.

Separation of waxes into their acids and mixtures of their alcohols and hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 304,150, 14.1.29. Ger., 14.1.28).—The mixture obtained by saponifying beeswax, montan wax, wool fat, etc. is atomised, and the dry powder so produced is extracted with solvents to remove the unsaponifiable matter. E. LEWKOWITSCH.

Modifying isocolloid materials (B.P. 321,689, 321,691–4, 321,699, 321,722–7).—See III. **Bleaching materials** (B.P. 328,544).—See VI. **Heating hydrogen for hydrogenation** (B.P. 329,045).—See VII. **Products resembling waxes** (B.P. 328,190). **Plastic masses** (B.P. 304,612).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oil of white pine seeds from Cadore. V. MASSERA (Giorn. Chim. Ind. Appl., 1930, 12, 180).—A sample of this oil, obtained in about 8% yield, gave: d_{4}^{15} 0.8568, α_D^{20} –82° 30', n_D^{20} 1.4742, acid value 0.429, ester value 5.103, esters as bornyl acetate 1.786%, acetyl ester value 8.342, total alcohols as $C_{10}H_{18}O$ 2.31%, complete solubility in 90% alcohol in the proportion 1 : 9; Schiff's reagent gives after about 15 min. a faint blue coloration, which deepens after 2 hrs. The oil consists mostly of *d*-limonene, with small proportions of a sesquiterpene (? cadinene), and of borneol, partly free and partly esterified. T. H. POPE.

Qualitative reactions for pine oil. J. KOR'A (Chem. Listy, 1930, 24, 150–153, 169–179).—The only reliable reactions for the detection of pine oil in turpentine are the Grimaldi modification of Halphen's method (B., 1910, 963) and Wolff's method (B., 1923, 730 A).

R. TRUSZKOWSKI.

Ultramarine blue-white lead mixtures. H. WAGNER and E. MARTZ (Farben-Ztg., 1930, 35, 1553–1554).—A comprehensive series of tests showed that ultramarine blue-white lead mixtures in various vehicles are quite stable, no formation of lead sulphide being recorded in oil or varnish vehicles. A slight danger

exists in the presence of water (either from aqueous media or moisture retained in the film), but this is avoidable by good working conditions and thorough drying. It is advisable, although not essential, to avoid the use of white lead containing lead acetate. It is pointed out that Brilliant Yellow, a mixture of cadmium-yellow and Kremnitz White, has long been in use without decomposition troubles. S. S. WOOLF.

Minium. J. HERITES (Chem. Listy, 1930, 24, 218—220).—The covering power of minium depends on the fineness of division of the grains, not on the actual dioxide content. Thus three specimens of minium, containing 29.65, 29.88, and 33.55% PbO_2 , and the diameters of the grains of which were, respectively, 0.7—15.5, 0.8—27.9, and 1.0—24.8 μ , had a covering power of 250, 80, and 133 cm^2 per g.

R. TRUSZKOWSKI.

Cadmium pigments. SEDLACZEK (Farben-Ztg., 1930, 35, 1661—1662).—The properties, composition, and preparation of cadmium sulphide and selenide pigments are discussed, with references to the patent literature on the subject. S. S. WOOLF.

Cadmium-selenium red. H. E. MARLEY (J. Amer. Ceram. Soc., 1930, 13, No. 5, ii, 44—47).—Tests were carried out to determine the proportions of cadmium, sulphur, and selenium required to produce a brilliant red, the sources of cadmium being the carbonate, oxide, and sulphide. Cadmium carbonate produced a better red colour than either the oxide or the sulphide. The limits in composition for the production of a brilliant red were: cadmium carbonate 58—70%, sulphur flowers 21—31%, selenium metal 9—15%. F. SALT.

Slippage correction in the equation of plastic flow. R. V. WILLIAMSON (J. Rheology, 1930, 1, 283—287).—The flow-pressure diagrams of pigmented dispersions have been determined to test the validity of Buckingham's (Proc. Amer. Soc. Test. Mat., 1921, 21, 1154) equation for plastic flow. With a plastic dispersion made by grinding 2 pts. of zinc oxide with 1 pt. of raw linseed oil, the apparent fluidity decreases rapidly to a minimum as the shearing stress is decreased and again rises slightly. At this point the medium continues to flow, but the pigment becomes flocculated and stationary. The product of the minimum fluidity and the radius of the capillary is constant. A dispersion of "Titanox" in equal parts of linseed oil and a petroleum hydrocarbon, which showed little plasticity, yielded a linear flow-pressure curve passing through the origin. The slight change in the slope of this curve for different capillary radii is in the direction opposite to that expected from the equation, which, however, represents with fair accuracy the behaviour of the zinc oxide dispersion at low shearing stress down to the point where flocculation begins. The minimum on the curve corresponds to the formation, in the capillary, of a core of weak gel surrounded by liquid medium.

T. H. MORTON.

Production of coumarone resins. I. I. BURDA (Ukraine Chem. J., 1929, 4, [Tech.], 249—255).—Coumarone resins can be prepared directly from the heavy benzol fraction of coal tar, without the necessity for any purification from phenols and pyridine compounds. The

yield of resin varies with the quantity of sulphuric acid added (1—4% by vol.); with less acid the yield is smaller, but the resin is of a lighter colour and is softer. The repeated treatment of the heavy benzol fraction with 0.5—1% of acid, with distillation of volatile hydrocarbons following each addition of acid, yields at first hard resins and later soft ones. The latter can be converted into the former by heating at 150—180° for 20—90 min. R. TRUSZKOWSKI.

Artificial copals [Albertols] as raw materials for modern varnishes. ANON. and E. FONROBERT (Farben-Ztg., 1930, 35, 1506—1510, 1554—1557, 1606—1608, 1658—1661).—A dissertation on the use of Albertols. Their general properties and the suitability of various grades for particular purposes are discussed. The introduction of driers into oil varnishes is considered at some length, the characteristics of various forms of lead, manganese, and cobalt driers being detailed. The remaining sections deal with the use of Albertols in primers, fillers, and surfacers, carriage varnishes and japans, and "4-hr." varnishes, formulations and full manufacturing instructions for 14 typical products being included. S. S. WOOLF.

Solvents from natural gas. CLARK. Solvents. FIFE and REID.—See III. Naval stores from waste wood. PALMER.—See V. Phosphorescent substances. RHODE.—See VII. Casein plastics. SIMMONS.—See XV. [Pigment from] *Lantana camara*. PILLAI and others.—See XVI. Ambergris. MORRISON.—See XX.

See also A., June, 746, Formaldehyde-urethane resinification (GIUA and RACCIU). 750, X-Ray investigations of cellulose derivatives (TROGUS and others). 781, Dehydrogenation of boswellic acid (BEAUCOURT). 783, Phenolcamphorein and its derivatives (DEULOFEU).

PATENTS.

Coating compositions and the like. IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 327,981, 3.10.28).—A mixture of rubber solution and oil with or without drier is thinned with a volatile solvent to a suitable consistency, and an inhibitor, e.g., 1—10% of quinol on the rubber content, is incorporated in order to retard the catalytic depolymerisation of the rubber. S. S. WOOLF.

Coating compositions and vehicles therefor. BRIT. CELANESE, LTD. (B.P. 299,782, 29.10.28. U.S., 31.10.27).—Cellulose derivatives, preferably organic, e.g., cellulose acetate, are dissolved in a low-boiling vehicle consisting of alcohols, e.g., ethyl alcohol, aromatic hydrocarbons, e.g., benzene, and halogen addition products of unsaturated hydrocarbons, e.g., ethylene dichloride, which in the cold are non-solvents for the cellulose derivatives. Synthetic or natural resins, high-boiling solvents, plasticisers, pigments, fillers, etc. may be incorporated. S. S. WOOLF.

Manufacture of resinous products. I. G. FARBEININD. A.-G. (B.P. 328,728, 5.3.29. Ger., 24.12.28).—Adipic acid, its higher homologues having a branched chain, or mixtures of these with each other or with other mono- or di-basic carboxylic acids, are condensed with

polyhydric alcohols in the absence of monohydric alcohols and, if desired, in the presence of a catalyst. The products may be incorporated with other resins, *e.g.*, "glyptals," for which they act as plasticisers.

S. S. WOOLF.

Synthetic resin varnishes and synthetic resins.

IMPERIAL CHEM. INDUSTRIES, LTD., H. H. MORGAN, A. A. DRUMMOND, and G. C. ATTFIELD (B.P. 327,946, 6.11.28).—A polyhydric alcohol, *e.g.*, glycerol, a dibasic acid or its anhydride, *e.g.*, phthalic anhydride, and a fatty oil, *e.g.*, tung oil, are made to react under heat with aliphatic alcohols of b.p. not above 175°, *e.g.*, butyl alcohol, cyclohexanol, alkyl monoethers of ethylene glycol, ethyl lactate. The reaction may be carried out in the presence of hydrocarbon or ester solvents, *e.g.*, hydrogenated naphthalenes, solvent naphtha, butyl acetate, benzyl benzoate. The aliphatic alcohol may be used in quantity just sufficient to bring about the incorporation of the oil, in which case the process may occupy 20–24 hrs., or in excess, when the time taken is shortened considerably, the excess alcohol being removed by distillation after the preliminary reaction has occurred. Driers may be added at any time in the process, and dyes or pigments may be incorporated.

S. S. WOOLF.

Production of polybasic acid–polyhydric alcohol resin. J. H. SCHMIDT, ASSR. to BAKELITE CORP. (U.S.P. 1,739,771, 17.12.29. Appl., 16.3.28).—Glycerin is heated to 205° with excess of phthalic anhydride, substantially toward the point of slag formation, *e.g.*, until an extracted sample "strings" at 180–190°. The mass is then chilled to arrest the reaction by pouring into thin slabs, preferably in metal trays, and the cooled product is hardened by baking at 125–145° for 12 weeks.

S. S. WOOLF.

Synthetic resinous compositions. IMPERIAL CHEM. INDUSTRIES, LTD., W. BAIRD, R. HILL, and E. E. WALKER (B.P. 328,003, 9.1.29).—Polyhydric alcohols are condensed with polybasic acids in the presence of monocarboxylic acids preferably of high mol. wt., *e.g.*, drying oil acids, and drying oils are incorporated before or during the condensation.

S. S. WOOLF.

Obtaining aldehyde condensation products. M. MELAMID (U.S.P. 1,727,076, 3.9.29. Appl., 8.12.24. Ger., 4.12.23).—Hydroxy-derivatives of aromatic hydrocarbons are treated with a mixture of aldehyde and an electrolyte. *E.g.*, 100 pts. of phenol are heated on the water-bath with a mixture of 60 pts. of 30% formaldehyde solution and 300–400 pts. of 10% sulphuric acid. The product is a soluble resin, m.p. about 90°.

A. R. POWELL.

Production of artificial compositions from the condensation solutions of urea, thiourea, or their derivatives and formaldehyde. L. N. REDDIE. From KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 328,040, 11.12.28).—The condensation of 1 mol. of urea, thiourea, or a derivative thereof with less than 2 mols. of formaldehyde in the presence of acids is interrupted before the stage at which the resin would separate from the solution in a solid form on cooling, and the resin solution is concentrated at 40–50°, *e.g.*, by spraying it against a warm current of air or by the

use of heated rollers, vacuum drying drums, etc., until a gelatinous mass containing about 5% of water and capable of swelling in water is obtained. The mass is comminuted during or after the concentration process and moulded by hot-pressing in the presence, if desired, of ammonium sulphate or other acid salts which accelerate polymerisation. The press powder may be washed with ammonia or other reagents which fix or remove formaldehyde.

S. S. WOOLF.

Melting and esterification of resins or the like.

J. SOMMER (B.P. 319,651, 28.2.29. Ger., 25.9.28).—Resins, *e.g.*, copals, are esterified with glycerin etc. in apparatus comprising a heated melting pot fitted with cover, foam distributor, stirrer, and inlets for glycerin, inert gas, and air, the vapours from the pot, with the exception of water vapour, being condensed in a tubular cooler, and led to a separator from which glycerin separating out is returned to the pot. Esterified resin is discharged by pressure.

S. S. WOOLF.

Manufacture of products of the nature of resins, waxes, balsams, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,190, 20.10.28).—Resin acids, *e.g.*, abietic acid, natural resins containing them, or artificial resins still containing free carboxylic acids are treated with alkylene monoxides, at 60–200° and preferably at elevated pressures, in the presence or absence of acid condensing agents, *e.g.*, boric acid, zinc chloride, the products obtained being acylated if desired.

S. S. WOOLF.

Manufacture of a moulding mixture. V. H. TURKINGTON, ASSR. to BAKELITE CORP. (U.S.P. 1,728,378, 17.9.29. Appl., 11.12.22).—A mixture of furfuraldehyde with hexamethylenetetramine is incorporated with wood pulp or similar filling material and the heated mass is thoroughly mixed with a soluble phenol–formaldehyde condensation product. After moulding into shape the mass is hardened by suitable heat-treatment.

A. R. POWELL.

Moulding mixtures. H. WADE. From BAKELITE CORP. (B.P. 327,969, 12.1.29).—Such mixtures comprise a resin of the urea–aldehyde type, a filling material, *e.g.*, wood flour, a cellulose ester stable up to 165°, *e.g.*, cellulose acetate, and, if desired, a plasticiser preferably containing a dialkyl ester of an organic acid.

S. S. WOOLF.

Production of plastic masses. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS, Assecs. of W. PLINATUS (B.P. 305,238, 2.2.29. Ger., 2.2.28).—Moist cellulose esters, *e.g.*, nitrocellulose containing 30% of water or cellulose acetate containing 10–120% of water, the esters comprising or containing a proportion of low-viscosity material, are converted into solid solutions by the addition of high-boiling solvent and gelatinising agents, *e.g.*, ethyl phthalate, and the use of mechanical energy and heat, in the absence of volatile solvents.

S. S. WOOLF.

Manufacture of artificial [plastic] masses. I. G. FARBENIND. A.-G. (B.P. 304,612, 21.1.29. Ger., 20.1.28).—The use of a relatively small proportion of synthetic rubber, *e.g.*, that obtained from dimethylerythrene, as binding agent in otherwise customary processes for the

production of artificial masses resembling linoleum, ebonite, celluloid, etc. is claimed. S. S. WOOLF.

Preparation of coating materials and plastic masses. IMPERIAL CHEM. INDUSTRIES, LTD., Assees. of R. T. HUCKS (B.P. 298,914, 15.10.28. U.S., 15.10.27).—Nitrocellulose is treated with a colloidizing agent, *e.g.*, butyl phthalate, which is substantially immiscible with water, and the plastic thus produced is kneaded with water-wet pigment pulp, preferably in the presence of a small quantity of a substance which is an active solvent for nitrocellulose, but also substantially immiscible with water, *e.g.*, ethyl acetate. After incorporation of the pigment the separated water is decanted.

S. S. WOOLF.

Manufacture of lithopone. W. C. HOOEY, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,759,115—6, 20.5.30. Appl., 15.12.25).—See B.P. 263,119—20; B., 1928, 341.

Modifying isocolloid materials (B.P. 321,689, 321,691—4, 321,699, 321,722—7).—See III. **Zinc sulphide** (U.S.P. 1,742,030).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Testing of automotive rubber parts assembled under compression. I, II. F. D. ABBOTT (Ind. Eng. Chem. [Anal.], 1930, 2, 145—159).—I. The current specifications relating to the testing of automotive rubber parts are adversely criticised. It is shown that high tensile strength is no criterion of relative resistance to compression-set and that hardness is not a measure of deflectability. Deflection and permanent set under compression are influenced by the size and shape of the test piece, the method of cutting the sample, the methods of measuring it, as well as by the condition and kinds of surface in contact with the test piece.

II. An instrument is described with which tests of deflection, hysteresis, and set can be made under constant load on constant distortion. Details are also given of a method of carrying out a compression-set test.

H. INGLESON.

See also A., June, 782, **Colloidal nature of caoutchouc, gutta-percha, and balata** (STAUDINGER).

PATENTS.

Manufacture of porous bodies, filters, diaphragms, etc. consisting of porous or porous and non-porous parts made of soft to hard rubber and the like. M. WILDERMAN (B.P. 328,273—4, 20.12.28).

—(A) Porous rubber articles are formed from semi-vulcanised particles of a soft to hard mixture, which, while being spread, are kept at a temperature such that they are maintained in a state of fine division; they are then vulcanised. The particles may be sifted before use and, if stored, the storage vessel should be provided with an effective stirrer. The heat for vulcanisation may be provided by steam, but all moisture must be kept from the rubber. Articles only partly porous may be manufactured either by forming in separate moulds and vulcanising together, or by spreading the non-porous parts and compressing, then spreading the porous parts without much compression. In (B) machines for continuous and intermittent operation are described for the above processes. It is preferred to use metal foils in the

moulds so that the articles can be formed in one press and vulcanised in another. The non-porous parts of the articles may be produced by laying strips of non-porous rubber on the spread material before vulcanising.

B. M. VENABLES.

Applying and/or attaching or securing rubber or the like to metal [by surface beating]. DUNLOP RUBBER CO., LTD., H. C. YOUNG, and C. HEMM (B.P. 327,856, 10.4.29).

Heat-transfer for vulcanisation (B.P. 305,577).—See I. **Modifying isocolloid materials** (B.P. 321,689, 321,691—4, 321,699, 321,722—7).—See III. **Insulating materials** (B.P. 328,025).—See XI. **Coating compositions** (B.P. 327,981). **Plastic masses** (B.P. 304,612).—See XIII.

XV.—LEATHER; GLUE.

Action of neutral salts on the enzyme activity of tryptic bates. II. V. KUBELKA and K. DOUSA (Collegium, 1930, 66—72; cf. B., 1929, 1025).—The activity of "Standard Oropon" was determined by the Schneider-Ulcek method, using different amounts of the tryptic bate and of ammonium sulphate, respectively. Maximum activity varied with the relative proportion of neutral salt to bate and also proportionally as the amount of neutral salt. The activity of the enzyme bate at the point of maximum activity was the greater, the larger was the amount used in the determination. The maximum activity of the "Standard Oropon" on a casein substrate was three times that of the material without the addition of ammonium sulphate.

D. WOODROFFE.

Action of trypsin on the properties of collagen. R. O. and A. W. PAGE (Ind. Eng. Chem., 1930, 22, 545—547).—In addition to removing a certain amount of hide proteins in the process of bating, trypsin acts on the collagen, which has been considerably pretreated with alkali in the unhairing and liming process, to reduce its plumping, to lighten the colour and increase the mellowness of the resulting leather, and to diminish its power of combining with vegetable tanning materials.

F. R. ENNOS.

Action of papayotin and papain on hide powder. W. ACKERMANN (Collegium, 1930, 74—79).—When hide powder was treated with papain and papayotin, respectively, at 37° under different conditions of acidity and alkalinity, they had a bating action similar to that of trypsin. Their action was much stronger on hide powder and pelt than was indicated by the figures obtained with the Fuld-Gross casein method of testing enzyme activity. Papayotin was stronger than papain in its action.

D. WOODROFFE.

Colloid-chemical investigations of some tanning processes. I, II. W. SCHINDLER and K. KLANFER (Kolloidchem. Beih., 1930, 31, 100—148, 149—184).—The processes of dyeing, neutralisation, oiling, and pickling in the manufacture of chrome leather are discussed from the colloid-chemical point of view. The adsorption of dyestuffs by chrome leather has been investigated experimentally for Orange II, Patent Blue A, induline, and Diphenyl Brown BBN, and the influence of amount of adsorbent, concentration of the dye, and temperature have been studied. The relation

between the final concentration of dye and the relative adsorption is given by three exponential equations, each of which is valid for a certain range of concentration. When plotted logarithmically the curves show two breaks. The velocity of taking up acid, substantive, and basic dyes was measured; the last-named were found to be the most reactive. The values of $1/n$ in the adsorption isotherm are lower for the basic dyes than for the other two types. The neutralisation of the chrome leather with alkalis also proceeds in accordance with an adsorption process over a wide range. Measurements were also made of the rate at which chrome leather takes up oils emulsified by soap solution or sulphonated oils. The sulphonated oils were taken up the more rapidly. The rate of adsorption of acid in the pickling process has been investigated, and sulphuric and hydrochloric acids are taken up at the same rate, but formic acid is adsorbed more slowly. E. S. HEDGES.

Hydrogen-ion concentration and tanning effect. H. MACHON (Collegium, 1930, 49—63)—Crude and sulphited quebracho extracts were analysed by the official shake method of tannin analysis, but at different p_H values, and maximum results for tannin were obtained at p_H 6.0 and 5.9, respectively. More tannin was combined with the hide powder in the presence of weak than of strong organic acids. Similar effects were produced by acids of similar dissociation constants. The tanning value (amount of tannin absorbed by hide powder from more concentrated solutions) varied inversely as the p_H value of solutions of sulphited quebracho extract, but showed maxima at p_H 2—3 and 6.5—8.0 and a minimum at p_H 5.0. The greatest tanning value was obtained with acetic acid in solutions brought to the same p_H value with different organic acids. The p_H of maximum tanning value increased as the concentration of the solution decreased. Precipitation of the tan liquors was dependent on their p_H value, and independent of the particular acid present. The amount of the precipitate was affected by the nature of the acid. Tannin analysis should be made on the natural solutions and the p_H reported. It is shown that in the early stages of tanning at different p_H values there is no relation between the water and tannin contents of the tanned pelt. D. WOODROFFE.

Loss of tannin in liquors due to fermentation. J. M. SELTZER and F. F. MARSHALL (J. Amer. Leather Chem. Assoc., 1930, 25, 168—173).—Liquors (d 1.035) were prepared from various tanning materials and extracts left 12 weeks, during which period they were analysed at intervals. The loss in tannin (%) after 12 weeks was as follows: wattle bark 2.91; valonia cups and beards 27.5; myrobalans 60.24; solid mangrove-bark extract 0.48; liquid quebracho extract (heavily sulphited) 4.32, (lightly sulphited) 6.13; sulphite-cellulose extract 6.33; hemlock extract 14.88; chestnut (liquid) 17.0; oak-bark extract 21.44; valonia extract (powder) 40.04. The acidity of the liquors increased at first and then diminished slightly in most of them. No difference was shown in the acidity of the liquors by stoppering the containing vessel. D. WOODROFFE.

Manufacture of casein plastics. W. H. SIMMONS

(Ind. Chemist, 1930, 6, 206—208).—The combination of casein with formaldehyde with prevention of the drying out and splitting which otherwise occur is probably not capable of any simple chemical explanation. The early wet process is nowhere used commercially, having been completely replaced by the dry process. The casein used should be precipitated from skimmed milk collected under antiseptic conditions by rennet; the fat content should not exceed 2%, ash content should be 8%, and moisture 10—12%. Formaldehyde is used as a 40% solution, neutral to litmus, and should be stored in a warm place. Acid dyes are used for colouring. Water used should be free from iron, softened by zeolite or distillation, and degassed. C. IRWIN.

Maizolith. HARTFORD.—See V. Xylose. EMLEY. —See XVII.

PATENTS.

Production of thermoplastic material [resembling synthetic horn]. P. C. CHRISTENSEN (U.S.P. 1,746,070, 4.2.30. Appl., 20.8.26).—A thick aqueous paste of (preferably) equal parts of casein and "leather gum," a product obtained by successively treating scrap leather with dilute sulphuric acid, boiling with water, and removing, cooling, and grinding the residue, is passed between rollers at about 55—100° and then extruded or moulded under pressure; the products are toughened by treatment with an aldehyde solution.

L. A. COLES.

Manufacture of glues from yeast which are stable for storing. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,645, 30.1.29).—The products comprise intimate mixtures of yeast or the products of yeast autolysis with dry alkali hydroxides or salts having an alkaline reaction (e.g., di- or tri-sodium phosphate); aldehydes or substances which liberate them (e.g., paraldehyde, bishydroxymethylcarbamide) may also be added before, during, or after the mixing operation.

L. A. COLES.

Production of solidified globules ["pearl glue"] from liquid gelatinous substances. T. K. LOWRY (U.S.P. 1,746,543, 11.2.30. Appl., 25.1.28).—Glue etc. liquefied by heat is forced through adjustable jets into the lower end of a vertical, water-jacketed cylinder which receives, just above the jets, a stream of a non-inflammable liquid (trichloroethylene, carbon tetrachloride), the solidified globules are subsequently screened at the top of the cylinder and the liquid collected for use again.

L. A. COLES.

Manufacture of products for tanning and tawing industries. M. KAHN, E. LE BRETON, and G. SCHAEFFER (U.S.P. 1,759,700, 20.5.30. Appl., 22.4.26. Fr., 20.11.23).—See B.P. 247,977; B., 1926, 557.

XVI.—AGRICULTURE.

Soil survey of the Nalkantha district (Limbdia State) and its significance. C. V. R. AYYAR (J. Indian Inst. Sci., 1930, 13A, 43—56).—A method of mechanical analysis is adopted in which the usual pretreatment of the soil sample with acid and hydrogen peroxide is omitted and suspensions for the sedimentation process are prepared in distilled water without the

addition of dispersive agents. A system of soil classification based on such analyses can be correlated with field observations of soil texture and with chemical analyses.

A. G. POLLARD.

Influence of stones in soil on plant growth. E. BLANCK [with H. KEESE and F. KLANDER] (J. Landw., 1930, 78, 1—8).—The proportion of stones in soil affects plant growth only as a result of its influence on the volume-water capacity of the soil. Such effects are small, being significant when the proportion of stones approaches 40%.

A. G. POLLARD.

Potent factors in soil formation. C. F. SHAW (Ecology, 1930, 11, 239—245).—The influence of climate, vegetation, parent material, and erosion on soil formation is discussed.

A. G. POLLARD.

Biological *Azotobacter* method for determining the phosphate requirement of soils. H. NIKLAS and H. POSCHENRIEDER (Superphosphat, 1930, 6, 69—72).—Modifications are described in the technique of the *Azotobacter* test. Results agree well with those of the seedling method, especially where the phosphate content of the soil is small.

A. G. POLLARD.

Reaction and phosphate content of soils. F. TERLIKOWSKI (Rocz. Nauk. Roln. i Leśn., 1929, 22; Proc. Internat. Soc. Soil Sci., 1930, 5, 21—22).—Correlation between soil reaction and the easily-soluble phosphate content is only possible on certain soil types. All weakly acid soils benefit from phosphatic fertilisers, and the failure of growth of *Azotobacter* in these soils is mainly due to phosphate deficiency. General relationships between the phosphate content of soils and their reaction are to be anticipated if the latter value is expressed in terms of the degree of saturation with bases.

A. G. POLLARD.

Effects of potash and phosphorus on tip-burn and mildew of cabbage. C. CHUPP (Phytopath., 1930, 20, 307—318).—Heavy applications of superphosphate to soils increased the proportion of tip-burn in cabbage, especially on soils deficient in nitrogen and potash. Sodium nitrate was without effect on tip-burn unless large amounts were applied, when the disease increased somewhat. Potassium chloride reduced the disease and is recommended as a control measure. Potassic and nitrogenous fertilisers tended to increase the proportion of downy mildew in cabbage, whilst phosphates had the reverse effect.

A. G. POLLARD.

Relationship between the nitrogen and carbon metabolism of *Clostridium acetobutylicum*. P. W. WILSON, W. H. PETERSON, and E. B. FRED (J. Bact., 1930, 19, 231—260).—*Clostridium acetobutylicum* can utilise protein, peptone, or beef aminoids as sources of nitrogen with but small changes in the proportion of solvents produced during fermentation. Peptone tends slightly to increase the yield of acetone at the expense of ethyl alcohol, whilst beef aminoids favour the formation of butyl alcohol. The organism utilises part of the amino-acids as such. In the presence of aminoids there is a large increase in synthesised protein. Ammonia alone cannot be used, but in the presence of protein ammonium salts are preferentially utilised, the maximum being reached with ammonium carbonate. The ratio of

solvents produced is altered by the use of ammonium salts, there being a decreased acetone yield at the expense of ethyl alcohol unless the total yield falls, in which case the proportion of acetone increases. These variations in yield are attributed to changes in p_H of the fermenting liquor. Nitrates are not utilisable by the organism, whether alone or in the presence of undegraded protein.

A. G. POLLARD.

Reduction of nitrates to nitrites by *Salmonella pullorum* and *S. gallinarum*. R. P. TITSLER (J. Bact., 1930, 19, 261—267).—Examination of numerous strains of *Salmonella pullorum* and *S. gallinarum* shows both to be capable of reducing nitrates to nitrites. There is no appreciable difference between the strains. Wittes' peptone is unsuitable for media for this work, and "Difco" is recommended. Dimethyl- α -naphthylamine is preferred to α -naphthylamine for the detection of nitrites.

A. G. POLLARD.

Spot-plate test for nitrate-nitrogen in soil and other extracts. M. F. MORGAN (Science, 1930, 71, 343—344).—To one drop of the liquid to be tested are added 4 drops of a solution of 0.05 g. of diphenylamine in 25 c.c. of concentrated sulphuric acid and the colour is compared with a standard. The nitrate concentration of the liquid under test (as nitrogen) must be adjusted to within the range 1—25 pts. per million for the test to be approximately quantitative.

L. S. THEOBALD.

The plant as a factor in the action of Bordeaux mixture as an insecticide. D. M. DE LONG, W. J. REID, JUN., and M. M. DARLEY (J. Econ. Entom., 1930, 23, 383—390).—Copper can be dissolved from Bordeaux mixture residues by solutions of sugar and by expressed plant juices, but not by distilled or rain water. The sugar content of vigorously growing plants sprayed with Bordeaux mixture decreased for about 2 days after spraying, but later rose to, and maintained, a higher concentration than in unsprayed plants.

A. G. POLLARD.

Influence of Bordeaux mixture on the efficiency of lubricating-oil emulsions in the control of the San Jose scale. B. A. PORTER and R. F. SAZAMA (J. Agric. Res., 1930, 40, 755—765).—The efficiency of lubricating oil emulsion at low concentrations in reducing San Jose scale (*Aspidiotus perniciosus*) is lowered by the addition of ordinary strengths of Bordeaux mixture. For dormant spraying the oil content of such mixtures should be greater than 1.5%; for summer spraying 2.0%.

F. O. HOWITT.

Toxicity of copper to the potato leaf hopper. D. M. DE LONG, W. J. REID, JUN., and M. M. DARLEY (J. Econ. Entom., 1930, 23, 390—394).—Copper sulphate in dilutions up to 1 in 6500 and containing 5% of sugar was toxic to the nymphs of the potato leaf hopper. The plant juice of beans the roots of which were immersed in dilute copper sulphate solutions contained copper and caused high mortality among leaf hoppers feeding on the plants.

A. G. POLLARD.

Two arsenical substitutes [insecticides]. S. MARCOVITCH and W. W. STANLEY (J. Econ. Entom., 1930, 23, 370—376).—Cryolite and barium fluosilicate are highly toxic to insects, and are but little harmful

to foliage. Used as a spray (1 lb. to 50 gals. of water), both substances give effective control of the Mexican bean beetle, and as a dust (five-weekly treatments) were successful against hornworm and flea-beetle on tobacco. Dusted, in the proportion of 6 lb. per acre, neither material affected bean foliage, and at 30 lb. per acre caused only moderate scorching. The addition of 25% by wt. of fish oil improved the sticking quality of these dusts, which were also successful when mixed with twice their weight of lime.

A. G. POLLARD.

Fungicidal efficiency of chemical dusts containing furfuraldehyde derivatives. C. S. REDDY (Phytopath., 1930, 20, 147—168).—Numerous trials of mercury compounds of furfuraldehyde derivatives for controlling dry-rot seedling blights of maize are recorded. The most successful preparations were (1) compound from mercuric chloride and hydrofurfuramide in aqueous solution, (2) compounds formed by addition of mercuric chloride and nitrate to furfuraldehyde pretreated by the Cannizzaro reaction, (3) mercury furylacrylate. Judged by the increased yields of grain, these preparations were most efficient against *Diplodia* followed in order by *Basisporium* and *Giberella*.

A. G. POLLARD.

Egg-killing washes at the Long Ashton Research Station. L. N. STANILAND, F. TUTIN, and C. L. WALTON (J. Pomology, 1930, 8, 129—152).—Eggs of the capsid bugs (*Plesiocoris rugicollis*) and of the winter moth may be destroyed by the stifling or smothering action of the oily covering applied, an action performed satisfactorily by petroleum products such as heavy medicinal paraffin; the eggs of *Aphis* and *Psylla* are more resistant, but may be killed by the definite toxic action of tar distillates. It is considered that the inefficiency of the older tar washes made from "total" distillates was due to their relatively high content of volatile material which evaporated before the time at which the insect eggs were most readily killed, and to the fact that they contained tar acids and bases which had relatively low ovicidal powers and were easily washed from the eggs by rain. The "neutral" washes are far superior.

E. HOLMES.

Nature of "Kuhlerde" and its action on marsh soils. E. BLANCK and W. DÖRFELDT (J. Landw., 1930, 78, 9—30).—The amelioration of marsh soils by the surface treatment or ploughing-in of certain lower soil strata ("Kuhlerde") is examined. The physical and chemical effects of Kuhlerde depending on their sand and chalk contents are discussed.

A. G. POLLARD.

Utilisation of waste vegetation. I. Preliminary study of *Lantana camara*. L. T. R. N. PILLAI, P. K. DE, C. V. PARAMASIVAN, M. G. RAO, S. A. RAFAY, and T. R. SATHE (Agric. J. India, 1930, 25, 143—149).—A preliminary examination of *Lantana*, including ultimate chemical analyses of the roots, stems, leaves, flowers, and fruits, suggests that the stems and leaves of this plant, which thrives under poor soil conditions, are well suited for the production of synthetic farmyard manure. A hydrochloric acid-soluble pigment has been isolated from the flowers.

E. HOLMES.

Manuring of paddy in Lower Burma. D.

HENDRY (Agric. J. India, 1930, 25, 126—137).—The immediate manurial requirements of rice soils in Lower Burma are nitrogen and phosphoric acid; potash has little if any effect. Experiments with various proprietary ammonium phosphate fertilisers containing N and P_2O_5 in the ratios of 1:1 and 1:3 approx. indicate the economic superiority of those containing equal amounts of the two nutrients. The highest economic benefits follow the application of approx. 250 lb. per acre of fertiliser containing about 20% each of N and P_2O_5 , rather than the equivalent mixtures of ammonium sulphate and superphosphate.

E. HOLMES.

Xylose. EMLEY.—See XVII.

See also A., June, 725, **Colorimetric determination of phosphoric and arsenic acids** (ZINZADZE). 811, **Repellents against the Japanese beetle** (METZGER). 823, **Distribution of nickel and cobalt in plants** (BERTRAND and MOKRAGNATZ). **Distribution of titanium in cryptogams** (BERTRAND and VORONCA-SPIRT). **Rôle of chlorine in nutrition and growth of the tobacco plant** (GARNER and others). 824, **Determination of sulphur in plants** (FREAR). **Determination of nitrate in green tomato and lettuce tissues** (EMMERT). 825, **Evaluation of plant products** (NIETHAMMER).

PATENTS.

Conversion of ammonia into fertilisers. G. F. UHDE (B.P. 299,896, 3.11.28. Ger., 3.11.27).—Crude calcium phosphate containing chalk, preferably suspended in calcium nitrate solution, is treated with sufficient nitric acid or oxides of nitrogen, obtained by the oxidation of ammonia, to convert the carbonate alone into calcium nitrate; the enriched phosphate, after separation from the liquor, is converted by treatment with sulphuric acid into calcium sulphate and phosphoric acid, these being worked up to calcium carbonate and ammonium sulphate and to ammonium phosphate, respectively. Potassium salts etc. may be added to the liquor used in the initial nitric acid treatment so that mixed fertilisers are obtained on evaporating the solution after removal of the undissolved phosphate.

L. A. COLES.

Manufacture of mixed fertilisers. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,620, 29.10.28).—The fertilisers comprise isomorphous mixed potassium-ammonium sulphate-nitrate crystals prepared from solutions containing ammonium nitrate and sulphate and potassium nitrate and/or sulphate, one of which may be formed *in situ* by the interaction of two other salts; other nitrogenous or potassium salts or phosphates may also be present.

L. A. COLES.

Calcium arsenate (U.S.P. 1,727,306).—See VII. **Treatment of agricultural residues** (U.S.P. 1,747,538).—See XVIII.

XVII.—SUGARS; STARCHES; GUMS.

Economic and industrial prospects of xylose. W. E. EMLEY (Chem. Met. Eng., 1930, 37, 238—285).—Xylose is obtained by hydrolysis of xylan, which is present in cottonseed hulls (40%), corn cobs (32%), peanut shells (26%), and other agricultural wastes,

with dilute acid. The method of manufacture, including the removal of ash, gum, and protein material, is fully described, as are also the potential uses of xylose. These include oxidation to trihydroxyglutaric acid, which may be used as a substitute for citric acid in foods and beverages, or in place of acetic-lactic acid mixtures for the vegetable-tan bath in leather tanning; the preparation of nitro-derivatives of xylose or "xylite"; fermentation to xylonic, propionic, or lactic acid; its employment by diabetics and for the preparation of syrups for certain artificial silk spinning processes. Probable costs are outlined. E. H. SHARPLES.

Sulphur dioxide in fruit syrups. WOIDICH.—See XVIII. **Determination of sugar in condensed milk.** DE GRAAFF.—See XIX.

See also A., June, 720, **Acid clays of Japan** (OKUNO). 731, **Phthaleinoscope for acidimetry in coloured media** (BRUÈRE).

PATENTS.

Sugar-cane and like mills. A. MCA. LANG. From S. H. LEACH (B.P. 329,755, 26.3.29).

Evaporation of sugar solutions (B.P. 304,670).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Downy mildew of the hop in British Columbia. W. NEWTON and C. YARWOOD (Sci. Agric., 1930, 10, 508—512).—For use in conjunction with Bordeaux mixture on hops the most efficient "spreader" was found to be a potash-resin soap, prepared by dissolving 1 pt. of caustic potash and 2 pts. of resin in 3 pts. of water and used in the proportion of 1 pt. of soap to 200 pts. of Bordeaux mixture. A similarly prepared soda-resin soap was of little value. (Cf. B., 1930, 299.) A. G. POLLARD.

Source of diastase. C. H. ORIS (Science, 1930, 71, 221).—The trade product, "Pangestin," is a suitable source of starch- and sugar-free enzyme which is strongly amylolytic and proteolytic. L. S. THEOBALD.

Micro-determination of sulphur dioxide in wine and fruit syrups. K. WOIDICH (Mikrochem., 1930, 8, 147—150).—The liquid (5 c.c.) is acidified with 5 drops of dilute hydrochloric acid in a pear-shaped bulb which is heated with steam inside a specially designed Erlenmeyer flask. The distillate is collected in 5 c.c. of 0.01*N*-iodine solution and the excess iodine is titrated with thiosulphate. A second portion of the liquid (5 c.c.) is treated with dilute iodine until a blue colour is obtained with starch and the distillation is repeated after addition of 5 drops of acid. The first titration gives the total, and the second the combined, sulphur dioxide. A. R. POWELL.

Determination of aldehydes in rectified spirits. E. WOJCIECHOWSKA-STRUSZYŃSKA (Przemysł Chem., 1930, 14, 222—227).—The intensity of the coloration given by reagents of the Schiff type with aldehydes is the greater the greater the fuchsin content and the smaller the sulphur dioxide content of the reagent; for this reason such reagents become more sensitive on keeping. The sensitivity of the reagent also increases with increase in

alcohol concentration, until, at very high concentrations of alcohol, the reaction is positive even in the absence of aldehyde. The following minimum aldehyde contents (%) in absolute alcohol can be detected by the following reagents: Girard's and Mohler's, about 0.0005; Schiff's, Schiff and Gayon's, and Villavecchia's, about 0.00005. The following method is proposed for the determination of aldehyde in absolute alcohol. One c.c. of 10% *m*-phenylenediamine hydrochloride solution, kept in a dark glass bottle over animal charcoal, is added to 10 c.c. of the alcohol, and the fluorescence produced in the solution by the light of a mercury-vapour lamp is compared after 4 hrs. with that of a sample of alcohol of known aldehyde content. This standard is best prepared by fractional vacuum distillation of alcohol over *m*-phenylenediamine hydrochloride, and then adding to the aldehyde-free distillate a known quantity of acetaldehyde. Aldehyde-free alcohol gives a violet coloration in ultra-violet light under the above conditions, whilst a blue fluorescence appears at concentrations of 0.00002% of aldehyde; as the aldehyde concentration increases the fluorescence becomes yellowish-green. Accurate results are given by this method for concentrations of 0.00002—0.001%.

R. TRUSZKOWSKI.

Coils for stills. BUTCHER.—See I.

See also A., June, 731, **Phthaleinoscope for acidimetry in coloured solutions** (BRUÈRE).

PATENTS.

Manufacture of malt liquids. C. A. MITCHEL, Assr. to H. E. FREES Co. (U.S.P. 1,747,785, 18.2.30. Appl., 13.2.28).—The volatile constituents are distilled from hops at a pressure less than 1 atm., and the residual hop material is boiled with malt wort. After separation from the hop residues the wort is concentrated and, after cooling, is incorporated with the volatile constituents of the hops. C. RANKEN.

Tinting of beer. H. FINLAY (B.P. 328,999, 3.1.29).—A range of tinted glasses are so coloured that the progressive numbers which they bear correspond to the number of units of colouring matter in a unit volume of caramel solution. The glasses are mounted on a rotatable disc, so that they may be viewed side by side with a glass-sided tank containing the solution to be matched. The colour number of the wort and that to which it is to be raised are determined, and the difference between the two numbers on the glasses gives the number of colour units to be added to unit volume of the wort. C. RANKEN.

Apparatus for determining alcohol in liquids. B. STEIN (U.S.P. 1,747,742, 18.2.30. Appl., 21.12.28).—Two conical receptacles, one of which contains water and the other the liquid to be tested, are placed side by side and heated by a single source of heat situated in a flue between the two vessels. Thermometers are mounted in both vessels to indicate the b.p. of the contained liquid, and the second receptacle is also fitted with a condenser for the alcoholic vapours.

C. RANKEN.

Treatment of distiller's wash, and other by-products and agricultural or industrial residues.

M. BERNIER, A. and F. DURIEZ, and H. SCHOTSMANS (U.S.P. 1,747,538, 18.2.30. Appl., 29.3.23. Fr., 19.1.23).—Distiller's wash and other fermentable by-products are sterilised and fermented in a closed vessel by bacteria of the type of *B. aminophilus*. Before and during fermentation the p_H of the solution is adjusted to between 6 and 8.

C. RANKEN.

Glues from yeast (B.P. 328,645).—See XV.

XIX.—FOODS.

Wheat and flour studies. XVI. Effect of yeast fermentation on viscosity of flour-water suspensions. A. H. JOHNSON and J. GREEN (Cereal Chem., 1930, 7, 117—132).—Fermentation lowers the viscosity of flour-water suspensions, the rate of decrease depending on the hydrogen-ion concentration during the process, high concentrations increasing and low decreasing this rate. The presence of sodium chloride and phosphates prevents the decrease in viscosity during fermentation periods extending over 10 hrs. The viscosity cannot be restored by addition of salts after fermentation.

W. J. BOYD.

Certain effects of varying the p_H of the tempering water on the wheat proteins. M. C. MARKLEY (Cereal Chem., 1930, 7, 154—161).—For tempering wheat 0.1*N*-sulphuric acid and 0.1*N*-sodium hydroxide, respectively, do not differ from water at 92 hrs., but at 20 hrs. they depress the amino-acid content and increase both the water-soluble nitrogen and the copper-non-precipitable nitrogen. Sodium sulphate (0.05*N*) depresses these three values at all times. Sodium hydroxide combines more freely with the proteins than does sulphuric acid, but dilute solutions of these as tempering agents have nearly identical effects on the nitrogen distribution.

W. J. BOYD.

Effect of severe weathering on the protein and ash contents of wheat and flour. W. O. WHITCOMB and A. H. JOHNSON (Cereal Chem., 1930, 7, 162—168).—No change was observed in the protein and ash contents of wheat subjected to severe weathering throughout the winter, although marked changes occurred in physical properties (*e.g.*, in weight per bushel) and in germination.

W. J. BOYD.

Water imbibition of frosted wheat. C. H. BAILEY and E. G. BAYFIELD (Cereal Chem., 1930, 7, 108—116).—Unripe wheat kernels frozen when their moisture content is above 40% subsequently show higher water imbibition and higher amino-nitrogen content than normal kernels. Freezing in the unripe stage appears to arrest protein formation from the amino-acids. On rewetting, such kernels tend to revert to the moisture content at the time of freezing.

W. J. BOYD.

Sundry means of hastening the determination of protein in wheat. H. L. WILKINS (Cereal Chem., 1930, 7, 168—188).—Various devices are described for handling reagents and samples in the routine determination of the protein in a large number of wheat samples. A reliable modification of the method recommended by the American Association of Cereal Chemists is described, whereby the protein in a sample may be determined in 25 min. In this, 0.5 g. of sample is digested

in a 500-c.c. Kjeldahl flask for 12 min. on a 600-watt pre-heated Gilmer heater. Half portions of concentrated sulphuric acid, alkali thiosulphate solution, and mercuric oxide-potassium sulphate mixture are used, with the other reagents similarly reduced in amount, and 75 c.c. of liquid are distilled over.

W. J. BOYD.

Adaptability of the quinhydrone electrode to cereal work. L. V. SORG (Cereal Chem., 1930, 7, 143—153).—The quinhydrone electrode gives p_H values in close agreement with those obtained with the hydrogen electrode, and by its use a constant potential is more readily reached (about 1 min.). The temperature coefficient is -0.00074 volt per degree. Under ordinary laboratory conditions variations of pressure have no measurable effect. Duplicate determinations agree within 0.000802 volt. For flour-water extracts bare gold wire is the best inert electrode, but bare platinum wire may be used for most solutions free from suspended matter. There is no error due to the water-soluble proteins in determining the p_H of flour-water extracts by the quinhydrone electrode.

W. J. BOYD.

Fermentation of bread. J. SPOUSTA and A. KUBÁSEK (Chem. Listy, 1930, 24, 121—129, 145—150).—Dough made from wheat and rye flours, and from mixtures of the two, with the addition of different quantities and strengths of yeast, is allowed to ferment for different periods of time before baking. For each case a certain optimum time is found, above or below which the porosity of the bread obtained is smaller.

R. TRUSZKOWSKI.

Speed of germicidal action of chlorine compounds on bacteria commonly occurring in milk. C. K. JOHNS (Sci. Agric., 1930, 10, 553—563).—The effects of certain chlorine preparations used as sterilising rinses on *Esch. coli*, *Aer. aerogenes*, *S. lactis*, and *B. subtilis* are recorded. Chloramine-T preparations were uniformly too slow in action, whereas liquid hypochlorite preparations were very effective against all but sporing organisms. None of the materials examined affected spores of *B. subtilis*. Losses of available chlorine during storage were least in chloramine-T solutions. Home-made (prepared from bleaching powder) were more stable than proprietary preparations.

A. G. POLLARD.

Determination of sugar content of condensed milk. J. DE GRAAFF (Chem. Weekblad, 1930, 27, 339—340).—The error introduced in the polarimetric determination by the mutarotation of the lactose is discussed, and a modified procedure for preparing the solution is described.

S. I. LEVY.

Cholesterol as a measure of egg yolk in milk products. L. M. LAMPERT (Ind. Eng. Chem. [Anal.], 1930, 2, 159—162).—A colorimetric method based on the Liebermann-Burchard procedure is described for the determination of cholesterol in dairy products, *e.g.*, ice-cream, by which the amount of egg used in the preparation may be estimated. A determination of the total cholesterol content of the ice-cream is made and a correction is applied for that introduced by the other constituents. It is found that whole, fresh egg-yolk contains 1.36% of cholesterol.

H. INGLESON.

Method of Uglow and Schapiro for determination of caffeine in tea. F. C. VAN EEKHOUT (Chem. Weekblad,

1930, 27, 189—191).—Numerous methods for the determination of the caffeine content of tea are surveyed. The method of Uglow and Schapiro (B., 1928, 544) is considered inferior to that of Romburgh, Nanninga, and Deuss, and is criticised on account of the employment of sodium carbonate solution for the extraction: the caffeine obtained has usually a lower m.p. than that obtained by the other method. H. F. GILLBE.

Vitamin-C in fresh and canned tomatoes. B. CLOW and A. L. MARLATT (J. Agric. Res., 1930, 40, 767—775).—Various methods of canning cause only a slight loss, or none at all, in the vitamin-C content of field-ripened tomatoes. The amount of vitamin-C in field- or greenhouse-matured green tomatoes allowed to ripen by exposure to the air or ethylene is equal to that of tomatoes ripened naturally on the plant. This result is in agreement with that of House, Nelson, and Haber (A., 1929, 610). The vitamin-C content increases with maturation of the tomato. F. O. HOWITT.

Freezing of bacon. C. R. BARNICOAT (New Zealand J. Sci. Tech., 1930, 11, 409—410).—Bacon stored at -10° freezes slowly, giving large ice crystals which disrupt the flesh, but rapid freezing at lower temperatures, if anything, improves the quality. C. W. GIBBY.

Oxidation-reduction indicators as a means of determining overheating in walnuts during dehydration. A. R. C. HAAS (Bot. Gaz., 1930, 89, 200—204).—Overheating during the dehydration of walnuts is marked by the increased reducing power of the products. The latter is determined by the extent of the colour change in suitable oxidation-reduction indicators (e.g., an α -naphthol-2-sulphonate-indophenol, methylene-blue, or the mono-, di-, or tetra-sulphonates of indigo) when added to an infusion of mashed walnut in a neutral buffer solution.

A. G. POLLARD.

Detection of hardened fats. KAUFMANN AND KELLER.—See XII. **Casein plastics.** SIMMONS.—See XV. **Xylose.** EMLEY.—See XVII.

See also A., June, 825, **Evaluation of plant products** (NIETHAMMER). 827, **Composition of odoriferous compounds of roasted chicory** (REICHSTEIN and BEITER).

PATENTS.

Rendering victuals radioactive. H. STRATMANN and F. G. WERNER (STRATMANN & WERNER) (B.P. 329,292, 11.2.29).—Victuals coated with an electrically conducting film of water vapour are exposed at a high negative potential in the open air, until atmospheric radium emanation is transferred to them.

J. S. G. THOMAS.

Preparation of tapioca and apparatus therefor. R. L. M. MORIN (B.P. 316,291, 16.5.29. Fr., 28.7.28).—Cassava fecula after treating with water until the moisture content is 50—55% above normal is spread out in layers 1 cm. thick on non-ferrous metal, then heated by steam for 2—3 min., and the product dried in a kiln. E. B. HUGHES.

Preparing an infusion of coffee from which the poisonous constituents have been removed. J. LOCKHOVEN (B.P. 327,662, 20.8.29).—Before infusion

the coffee is mixed with granulated active carbon, e.g., in the proportion of 4:1. A small quantity of boiling water is added to the mixture, and a further quantity after 5—10 min. F. G. CLARKE.

Pretreatment of materials used in the preparation of pectin. PECTINERIE DU KERVOR, S.A.R.L. (B.P. 302,734, 21.12.28. Fr., 21.12.27).—In the preliminary lixiviation of the pulp used for the extraction of pectin, hot dilute acid is used instead of organic acid. It is claimed that this treatment gives a larger yield and purer pectin. E. B. HUGHES.

Preservation of meat and fish refuse and the like. W. CONNEMANN (B.P. 328,547, 21.12.28. Ger., 23.11.28).—The material, e.g., raw shrimps, is mixed with sufficient quick lime, plaster of Paris, etc. to remove chemically the greater part of the water, and most of the hydrated lime etc. is removed from the product by screening. The product may be dried further by treatment with hot gases which, when lime has been used and the product is required for the manufacture of fodder, may contain carbon dioxide to convert residual calcium hydroxide into the carbonate. L. A. COLES.

Treatment of [edible] animal matter. D. THOMSON (U.S.P. 1,758,987, 20.5.30. Appl., 30.11.25. Renewed 11.10.29).—See B.P. 252,754; B., 1926, 689.

Apparatus for production of fish meal or the like. C. DOWNS and R. A. BELLWOOD (B.P. 329,723, 4.3.29).

Freezing cream etc. (B.P. 328,434).—See I. **Bleaching materials** (B.P. 328,544).—See VI. **Irradiation of substances** (B.P. 328,410).—See XI. **Margarine** (B.P. 327,519).—See XII. **Increasing the vitamin content of foodstuffs** (B.P. 328,942).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pharmaceutical specialities and secret remedies.

VII. Identification of pharmaceutical and technical solvents. C. A. ROJAHN, M. GREISS, and A. LINK (Pharm. Zentr., 1930, 71, 337—346).—Tables are given for the separation and identification of 61 of the more common organic solvents. The fractions of the product soluble, and insoluble, in water are distilled through a column and the constituents identified by the special tests described previously (cf. B., 1927, 617).

H. E. F. NOTTON.

Employment of the Seitz filter for pharmaceutical solutions. A. JACOBSEN (Dansk Tidsskr. Farm., 1930, 4, 146—154).—Experiments on the filtration of solutions of (a) arsenious oxide buffered with sodium phosphate, (b) morphine hydrochloride, (c) caffeine sodium benzoate, using the above filters, both of the pressure and suction type, showed that only 2—3% of the dissolved constituent was adsorbed by the filter; in the case of a 1:10,000 solution of methylene-blue, 59% of the latter was retained. The material of the filter was found to yield magnesia to solutions passing through it, hence if solutions of alkaloids are being filtered these must either contain free acid (0.001N) or be buffered (p_H 5.5), otherwise a separation of the free base occurs in the filtrate.

H. F. HARWOOD.

Industrial extraction of santonin. F. CHEM-NITIUS (Chem.-Ztg., 1930, 54, 335).—An account of a small plant for the production of 40 kg. per month of the pure substance is given. S. I. LEVY.

South African pepper-tree oil. G. W. B. VAN DER LINGEN (Perf. Ess. Oil Rec., 1930, 21, 154).—Steam-distillation of fresh pepper-tree leaves with green berries gave 0.42% of a practically colourless oil having: d_{4}^{20} 0.8486, α_D +58° 24', n_D^{20} 1.4732, acid value below 0.1, ester value (phenol-free oil) 46.70, ester value after acetylation (phenol-free) 115.80. On addition of absolute alcohol the solution immediately becomes turbid. Thymol could not be detected in the oil, but a phenol (carbanil ester, m.p. 140°), probably carvacrol, is present and also 26.3% of phellandrene. E. H. SHARPLES.

Leaf oil from *Dacrydium Franklini*, Hooker. A. R. PENFOLD and J. L. SIMONSEN (J. Proc. Roy. Soc. New South Wales, 1930, 63, 95–101).—The air-dried leaves of *D. Franklini* gives 0.3–0.76% of essential oil having d_{4}^{15} 0.8752–0.8911, α_D^{20} +8.4° to +18.6°, n_D^{20} 1.4805–1.4871, ester value 2.7–4.5, ester value after acetylation 16.6–36.9, insoluble in 10 volumes of 80% alcohol. l - α -Pinene, β -pinene, and d -limonene are present in the oil as also is phyllocladene, m.p. 95°. The hydrocarbon "dacrydene" stated to be present in this oil (Hooker, Baker, and Smith, "Pines of Australia," p. 397) is probably identical with d - Δ^4 -carene. E. H. SHARPLES.

Australian "tea trees" of economic value. I. A. R. PENFOLD and F. R. MORRISON (Bull. Tech. Mus., Sydney, 1929, No. 14, 15 pp.).—The following essential oils were obtained by steam-distillation of the leaves and terminal branchlets. *Melaleuca linariifolia* gave 1.5–2.0% of oil having d 0.8927–0.8992, α +3.3° to +6.8°, n 1.4752–1.4780, ester value 1.3–2.7, ester value after acetylation 58–82, and consisting principally of α - and γ -terpinene, cymene, cineol (16–20%), Δ^1 -terpineol-4, and sesquiterpenes. *M. alternifolia* yielded about 1.8% of oil having d 0.8958–0.8961, α +6.8° to +7.4°, n 1.4782–1.4790, ester value 4–7, ester value after acetylation 80–84, and containing d - α -pinene, α - and γ -terpinene, cymene, cineol (8%) Δ^1 -terpineol-4, and sesquiterpenes. Both the above oils have pleasant odours, are non-poisonous, and are 11–13 times as powerful in destroying typhoid germs as is carbolic acid. From *Leptospermum citratum* is obtained 1–1.5% of oil having d 0.8792–0.8856, α +3.5°, n 1.4688–1.4757, solubility in 70% alcohol 1 in 1–1.2 vols., and containing about 50% of citral and 35% of citronellal. E. H. SHARPLES.

Essential oils of *Melaleuca decora* (Salisbury) Druce, and *M. nodosa*, var. *tenuifolia* (de Candolle), from the Port Jackson district. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1930, 63, 102–110).—Steam-distillation of the leaves and terminal branchlets of *M. decora* yielded 0.12–0.31% of bright yellow essential oils having d_{4}^{15} 0.8895–0.9069, α_D^{20} +12.2° to +13.25°, n_D^{20} 1.4785–1.4872, ester value 8.1–11.9, ester value after acetylation 30.4–52.6, solubility in 80% alcohol insoluble in 10 vols., and consisting chiefly of d - α -pinene (50–60%), sesquiterpenes (25–30%), with small quantities of α -terpineol

(?), dipentene, and a sesquiterpene alcohol. Leaves and terminal branchlets of *M. nodosa*, on steam-distillation, yielded 0.9–1.0% of lemon-yellow coloured oils having d_{4}^{15} 0.9073–0.9125, α_D^{20} +5.15° to +7.75°, n_D^{20} 1.4641–1.4649, ester value 3.5–5.8, ester value after acetylation 40.7, solubility in 70% alcohol 1 in 1.3–5.5 vols., and containing cineol 40–55%, α -pinene, dipentene, α -terpineol, and sesquiterpenes. E. H. SHARPLES.

Occurrence of a number of varieties of *Eucalyptus dives* as determined by chemical analysis of the essential oils. III. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1930, 63, 79–84; cf. B., 1929, 537).—Essential oils from *E. dives* grown in Victoria are described. Most of the trees examined were Type with an admixture of var. "A," and this was reflected in the varying piperitone contents of the oils (2–54%). Previous results have been confirmed, and all forms of *E. dives* are well established as separate varieties according to the chemical composition of the respective oils. Where the piperitone content fluctuates between 26% and 36% it is due almost invariably to admixture of var. "A" with Type. Four representative *E. dives*, Type oils from Victoria had d_{4}^{15} 0.8866–0.9062, α_D^{20} –57.3° to –68.7°, n_D^{20} 1.4783–1.4810, solubility in 70% alcohol 1 in 1.4–10 vols., piperitone 36–53%. The yields were 2.9–4.0%. E. H. SHARPLES.

Determination of citronellal and citral in Australian essential oils. A. R. PENFOLD and W. G. ARNE-MAN (J. Proc. Sydney Tech. Coll. Chem. Soc., 1927–1929, 4, 53–60).—The method of Holtappel (B., 1928, 587) for the determination of citronellal gives low values in the examination of oils rich in this aldehyde, and the following modification is described. About 2 g. of the oil are cooled to –10°, 20 c.c. of reagent [5 g. of hydroxylamine hydrochloride in 9 c.c. of water, 80 c.c. of neutral rectified spirit, 2 c.c. of bromphenol-blue indicator (0.1 g. in 3 c.c. of 0.05N-sodium hydroxide and 22 c.c. of water), made up to 100 c.c. with rectified spirit] and 30 c.c. of 0.5N-potassium hydroxide are mixed and also cooled to –10°, and the two solutions are mixed, kept at –10° for 15 min., and then allowed to attain room temperature. The excess alkali is then titrated with 0.5N-alcoholic hydrochloric acid. A good end-point is obtained and results, using pure citronellal and essential oils rich in this compound, are more accurate than those given by the original method, by Kleber's phenylhydrazine method, or by Dupont and Labaune's oximation method. Oils from *Eucalyptus citriodora* gave aldehyde contents of 70–85%, *Leptospermum citratum* 70–85%, and *Backhousia citriodora* 94%. Oils containing citral and citronellal soon change in aldehyde content on keeping. E. H. SHARPLES.

Hydrogen peroxide. SCHWYZER. **Antiformin.** GJALDBÆK. **Bismuth salts.** MAUERSBERGER.—See VII. **Testing of ampoules.** PRITCHARD.—See VIII. **Tobacco pest.** AYYAR.—See XVI. **Xylose.** EMLEY.—See XVII. **Determination of caffeine in tea.** VAN EEKHOUT.—See XIX.

See also A., June, 741, **Bactericidal dialkylacetic acids** (ARMENDT and ADAMS). **Bactericidal olefinic acids** (BROWNING and others). 765, **Mono- and**

polyalkoxy- or halogeno-substituted derivatives of benzhydroxylamine and $\alpha\beta$ -diphenylethylamine (VALETTE). 772, Ethyl esters of chaulmoogric acid (LORA Y TAMAYO). 786, Substituted piperidinoalkyl benzoates (BAILEY and McELVAIN). Catalytic reduction of 1-phenyl- and 1-benzyl-4-piperidones (BOLYARD). 788, 5:5-Substituted barbituric acids (VOLWILER and TABERN). Microscopical identification of barbituric acid (DENIGES). 793, Synthesis of thiazoles containing phenol and catechol groups (SUTER and JOHNSON). 794, The alkaloid coclaurine (KONDO and KONDO). 795, Decomposition of berberine in aqueous solution (DIETZEL and SOLLNER). 796, Microchemical reactions of veratrine (WAGENAAR). Fritzsche's reagent for alkaloids (DE LISSNER). 803, Preparation of lecithin (MALTANER). 825, Evaluation of plant products (NIETHAMMER).

PATENTS.

Vehicle for acetylsalicylic acid tablets. W. C. BAUSCH (U.S.P. 1,746,984, 11.2.30. Appl., 31.10.27).—The tablets contain 3–4 pts. of acetylsalicylic acid and 1 pt. of stearic acid, which latter inhibits hydrolysis in the stomach, but not in the intestine. L. A. COLES.

[Medical] preparation of solid compounds, capable of being spread, containing iodine and potassium iodide. R. GELLER (B.P. 310,869, 8.1.29. Ger., 2.5.28).—A solution of sodium hydroxide or ethoxide in alcohol is added to a hot alcoholic solution of benzoic acid, iodine, and potassium iodide are added to the neutral mixture, and the whole is poured into moulds and cooled. The benzoic acid may be replaced by salicylic, phthalic, palmitic, or stearic acid, or by mixtures of these. H. ROYAL-DAWSON.

Production of extracts for direct application as medicaments and for increasing the vitamin content of foodstuffs, medicaments, and the like. G. M. CLARK. From MATRO GES.M.B.H. (B.P. 328,942, 4.2.29).—Rootlets of germinated grain or maize from the drying kilns in maltings contain up to about 0.15% of sterol which is antirachitically active even in the unirradiated condition. The rootlets, either alone or mixed with substances such as caustic soda, lime, etc., are extracted with alcohol, the extract is freed from the solvent *in vacuo*, and the residue extracted with a solvent for fat. The solvent-free extracts so obtained are used for increasing the vitamin content of foods etc. E. H. SHARPLES.

Manufacture of solutions of cholesterol or its esters or mixtures of these substances. F. PASSEK (B.P. 328,922, 4.2.29).—Solutions of cholesterol or its esters etc. are prepared by dissolving these substances in a small amount of chloroform, ethylene dichloride, or similar type of chlorinated hydrocarbon and adding sufficient aqueous alcohol to ensure the production of a permanently clear solution. E. H. SHARPLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of grain size in photographic emulsions on failure of reciprocity law and a theory of its origin. A. P. H. TRIVELLI and R. P. LOVELAND (J.

Franklin Inst., 1930, 209, 639–648).—The results of Jones and Hall for the effect of difference in grain size on the intensity-time function of exposure of a photographic emulsion (B., 1926, 1030) have been confirmed and supplemented. If the larger grains contain most of the larger sensitivity specks, the small grains will require more light energy than the larger grains to convert their specks into centres large enough to be developable, which accounts for their being more sensitive to variations in intensity. If photographic sensitivity depends on the photo-conductivity of the silver halide lattice, the observed rate of increase of the photo-electric current with illumination adequately explains the failure of the reciprocity law at both low and high intensities.

R. CUTHILL.

Reactions of photographic materials to light. S. E. SHEPPARD (Ind. Eng. Chem., 1930, 22, 555–563).—A lecture. The applications of photochemistry to the problems of photography are discussed.

H. INGLESON.

Properties of [photographic] fixing baths. J. I. CRABTREE and H. A. HARTT (Brit. J. Phot., 1929, 76, 743–745, 758–761, 777–778; 1930, 77, 4–6, 19–22).—Various properties of fixing baths have been investigated and special tests are outlined for the determination of the velocity of fixation, the acidity, the sulphurisation life, *i.e.*, the time which elapses before deposition of sulphur, the hardening action on the gelatin film, and finally the developer capacity, *i.e.*, the quantity of standard developer which may be added before precipitation of aluminium sulphite and undue loss in hardening action occur. For a given emulsion a maximum velocity of fixation is reached with 30–40% sodium thiosulphate solution, and this velocity increases with temperature and with the degree of agitation of the film, being most markedly accelerated when the film surface is brushed. The hardening action is dependent on the balance between the potash alum, acetic acid, and sodium sulphite contents of the bath, and is unaffected by its age or thiosulphate content. With constant quantities of sulphite and alum the hardening decreases with addition of acid, whilst with increasing quantities of sulphite in presence of constant alum and acid concentrations the hardening rises to a maximum and then falls away, *i.e.*, for maximum hardening there is a critical sulphite concentration for each acid concentration. The maximum hardening with any combination is increased by addition of alum. Extension of time of fixation increases the hardening action, and over the temperature range 5–27° a slight increase is shown at the higher temperatures. For equal concentrations of alum and acid the sulphurisation life is rapidly increased by addition of sulphite, whilst the addition of acid to baths containing equal quantities of alum and sulphite shortens the life. Alum itself also slightly shortens the life. The application of these results to the compounding of fixing baths of definite properties is discussed; formulæ for satisfactory non-hardening and acid-hardening baths are also given, together with methods by which such baths can be revived during continuous use. The causes and means for avoiding various defects in the fixing process are described.

J. W. GLASSETT

Photographic chemistry. I. Dispersion of silver halides through the aqueous medium. T. SUZUKI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1930, 12, 285—293).—Ability to cause ready dispersion of freshly precipitated silver bromide in water appears to be the property of aqueous or alcoholic solutions of various dyes and the common alkaloids. Amongst the specific dyes are included the usual sensitizers, and desensitizers for gelatin-silver bromide plates. The dispersed silver halide is not stable, but after flocculation redisperses easily on agitation. Silver chloride and iodide, mercurous chloride, and thallous chloride can also be dispersed in the same manner. Substances which disperse silver bromide do not necessarily alter the sensitivity of the silver bromide plate, but plates made from dispersed silver bromide are suitable for photographic use. J. O. CUTTER.

See also A., June, 716, **Photomicrography of silver halide grains** (TRIVELLI and LOVELAND). **Photochemical properties of silver bromide in aqueous gelatin or gum arabic** (REYCHLER). **Blackening of photographic plates by electron rays** (NACKEN). 717, **Photothermometry** (HENCKY and NEUBERT).

PATENTS.

Kinematograph films. P. REHLÄNDER (B.P. 303,794, 8.1.29. Ger., 9.1.28).—The deleterious effect of scratches is minimised by the use of a support for the positive film comprising a mixture of celluloid with material of high albedo (magnesia, white lead, lithopone), the support having a transparency of about 55—60% and a diffusion value of 0.11—0.16. L. A. COLES.

[Fireproofing of] cellulose [kinematograph] films. FILM OZAPHANE (B.P. 305,653, 10.1.29. Fr., 10.2.28. Addn. to B.P. 301,878; B., 1930, 457).—Ammonium bromide is used alone without other admixture. H. ROYAL-DAWSON.

Marking material by electrodeposition (B.P. 329,258).—See V. **Transfer of wood grain** (B.P. 303,804).—See IX.

XXII.—EXPLOSIVES; MATCHES.

Duration of the explosion flame of quarry and coal-mine explosives. P. NAOUM and A. BERTHMANN (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 193—197).—For these experiments the authors made certain modifications in the Mettegang apparatus, and used 100-g. cartridges of 30 mm. diam., which were fired in a steel mortar, whereas Kast and Selle used 25-g. cartridges and fired them in a lead mortar with consequent loss of efficiency. The explosives examined comprised Wetter-Detonit A and B, Wetter-Siegrit A, Wetter-Nobelit A and B, Gelatit 1, Ammonit 1, 2, and 5, Ammon-Gelatine 1, Chloratit 3, Dynamit 1, and Kieselguhr Dynamit. The duration and size of flame, explosion temperature, and specific pressure are tabulated, and photographic reproductions of the flames are given. The lengths of the flames varied, but not proportionately, with their duration. No relation could be found between explosion temperature and duration of flame. Contrary to the experience of Kast and Selle, it was found that the flame

duration of coal mine explosives differed appreciably from that of quarry explosives, and even amongst the former there were obvious divergences.

W. J. WRIGHT.

Direct determination of nitric nitrogen in colloidal [explosive] powders. Acetone method. H. MURAOUR (Bull. Soc. chim., 1929, [iv], 45, 1189—1192).—The powder (about 0.8 g. for a nitric acid content of 12.5%) is agitated for 2 hrs. with 40 c.c. of acetone in a pyrex glass. 50 C.c. of 12-vol. hydrogen peroxide and 4 c.c. of 5*N*-sodium hydroxide are added, and, after agitating until dissolution is complete, 4 g. of sodium perborate. The mixture is left for 12 hrs., heated for 20 min. at 60°, and after adding 50 c.c. of water, at 50° till the perborate is destroyed. After cooling, 30 c.c. of 10*N*-sodium hydroxide, 80 c.c. of water, and 4 g. of Devarda alloy are added. The process is completed as in the ordinary Devarda method, about half the distillate being collected in 25 c.c. of *N*/3-sulphuric acid. The mixture gives concordant results with mixtures of nitrocellulose and nitroglycerin and the results are in agreement with those obtained by the Dumas method on guncotton etc. which is free from centralite and similar nitrogen compounds. Low results obtained by the ordinary Devarda method are due to the reducing action of organic matter in presence of soda on the nitrate in solution. R. BRIGHTMAN.

Xylose. EMLEY.—See XVII.

See also A., June, 708, **Explosion regions in gas mixtures** (POSTHUMUS). 725, **Analysis of nitrating acid.** (MÜLLER and KOGERT). 750, **X-Ray investigation of nitrocellulose** (TROGUS and others).

PATENTS.

Delay powder. F. OLSEN (U.S.P. 1,748,455, 25.2.30. Appl., 30.10.23).—Such powders contain a deterrent, such as triphenyl phosphate, to regulate the rate of burning. A suitable mixture contains charcoal 15.3%, sulphur 8.4%, potassium nitrate 74.1%, and triphenyl phosphate 2.2%. W. J. WRIGHT.

Manufacture of tracer compositions. W. S. HEITMANN (U.S.P. 1,748,288, 25.2.30. Appl., 8.9.25).—To obtain intimate admixture of the components of tracer composition, the combustible ingredients are first mixed together, and the binder (e.g., a metallic resinat) is dissolved in a solvent and incorporated with the mixture. The solvent is then driven off by evaporation, and the resultant material is ground, graded, and pressed. W. J. WRIGHT.

Preventing the formation of copper azide in copper lead azide detonators. W. ESCHBACH (B.P. 304,144, 14.1.29. Ger., 14.1.28).—Air is excluded from electric detonators with copper shells by fixing the fusehead in its cardboard case by means of sulphur or non-combustible materials such as chlorinated hydrocarbons, with or without other non-inflammable substances. After the whole has been inserted in the detonator, the free space above is sealed by a cast fusible material. W. J. WRIGHT.

Blasting cartridges. D. HODGE (B.P. 329,636, 20.2.29).

XXIII.—SANITATION; WATER PURIFICATION.

Bacterial population during [sewage-]sludge digestion. C. C. RUCHHOFF, J. G. KALLAS, and G. P. EDWARDS (J. Bact., 1930, 19, 269—294).—Changes occurring during the digestion of activated sludge, fresh sewage, and mixtures of these are examined. Reduction of the total organic solids was least in activated sludge and greatest in mixtures containing 20% of activated sludge. Changes in p_H of sludges during digestion were small, the extremes for all tests being 6.4—7.4. The total bacterial population increased for the first 10—15 days of digestion, after which there was a steady decline throughout the process.

A. G. POLLARD.

Germicidal efficiency of chlorine and the N-chloro-derivatives of ammonia, methylamine, and glycine against anthrax spores. F. W. TILLEY and R. M. CHAPIN (J. Bact., 1930, 19, 295—302).—Nitrogen trichloride, chlorine in neutral solution, and chlorine in solution in 0.02*N*-hydrochloric acid were effective against spores of *B. anthracis* in 15 min., with available chlorine concentrations of 10 p.p.m. or less. Corresponding values for chlorine in 0.02*N*-sodium hydroxide were 100 p.p.m., 2 hrs.; for monochloroamine 80 p.p.m., 45 min.; dichloroamine 80 p.p.m., 30 min.; dichloromethylamine 50 p.p.m., 30 min.; and dichloroglycine 50 p.p.m., 45 min. Monochloro-methylamine and -glycine were not effective in 2 hrs. with 200 and 240 p.p.m., respectively, of available chlorine.

A. G. POLLARD.

Toleration of fresh-water protozoa to increased salinity. H. E. FINLEY (Ecology, 1930, 11, 336—347).—Several among many species of protozoa examined survived transference from fresh to sea-water and others withstood lower concentrations of sodium chloride.

A. G. POLLARD.

Correlation of sulphite reducing and colon bacteria in water. I. M. LEWIS, T. C. GREEN, and V. HAMILTON (J. Amer. Water Works' Assoc., 1930, 22, 667—672).—Numerous samples of filtered river water and raw spring water were examined for the presence of *B. coli* of faecal and non-faecal origin and for organisms capable of reducing sulphites. The results obtained failed to show satisfactory correlation. Thus 20% of the samples returned as *coli* free, and therefore "good," contained over 11 sulphite reducers per 100 c.c., which would class them as "bad"; again, sulphite reducers were absent in 15% of the total, which samples, according to the *coli* test, would be "questionable" or "bad." It is considered that the sulphite-reduction test is of little or no value as a supplement to the standard test for *B. coli*.

C. JEPSON.

Influence of chlorine treatment on acidity of water. A. MASSINK (Chem. Weekblad, 1930, 27, 304—307).—The publications of Haase (cf. B., 1928, 466; 1929, 341) are criticised, and it is shown both experimentally and on theoretical grounds that with normal waters no variation in p_H sufficient seriously to affect the properties of the treated water is to be expected.

S. I. LEVY.

Determination of nitrates [in water]. G. W. BURKE, M. LEVINE, and G. H. NELSON (J. Amer. Water Works' Assoc., 1930, 22, 679—684).—Concentration of the alkaline sample prior to reduction, as recommended in "Standard Methods of Water Analysis," does not cause any loss of nitrite. Nitrites are destroyed if ammonium chloride (up to over 3500 p.p.m.) be added and the mixture evaporated to dryness before making alkaline. If the nitrites be destroyed in this way and the ammonia then driven off by concentration of the solution in presence of sodium hydrate, the nitrates may be determined directly by reduction in the usual way.

C. JEPSON.

Prevention of nuisance from fumes and dust. TRAVERS.—See I. **Determination of phenol in effluent waters.** PIETERS.—See II.

See also A., June, 741, **Bactericidal dialkylacetic acids** (ARMENDT and ADAMS). **Bactericidal olefinic acids** (BROWNING and others).

PATENTS.

Purification of polluted liquids. J. T. TRAVERS, ASSR. to OHIO SANITARY ENG. CORP. (U.S.P. 1,747,803, 18.2.30. Appl., 19.8.27).—The putrescible matter, mostly present in colloidal form, is converted into a coagulated suspensoid by agitating the liquid with a slowly dissolving reagent containing as a main constituent (a) an electrolyte-producing material, together with (b) an alkali, and (c) a coagulant. On adding (a), the colloidal putrescible matter acquires a negative charge and is then enabled to adsorb the positive ions offered by (a). Accompanying this adsorption nascent oxygen is generated which assists in destroying bacteria and renders re-aeration of the liquid unnecessary. By way of example, (a) may be calcium or magnesium sulphate, or certain waste products from cement manufacture or alkali plants; (b) lime; and (c) ferrous or aluminium sulphate or calcium monophosphate. Apparatus for carrying out the process is described. S. K. TWEEDY.

Apparatus for treating impure water with chemical solutions. O. WALTER (G.P. 456,611, 25.3.26).—The water is admitted through a float-controlled valve, and an adjustable apparatus to compensate for the hydrostatic head of the chemical solution is provided.

W. G. CAREY.

Purification and softening of water. F. SCHMIDT (F.P. 631,530, 28.3.27).—A mixture of caustic soda or potash, sodium or potassium carbonate, and water is finely ground and to it are added silicates, aluminates, or aluminosilicates. The mixed solutions are boiled before use.

W. G. CAREY.

Removal of oxygen from water. II. MANZ (G.P. 456,459, 21.3.26).—The water is passed over the cathode of a low-tension electrolytic cell using an easily oxidisable anode, with a slight cathodic polarisation.

W. G. CAREY.

Gas mask and like appliances generating their own oxygen. W. W. TRIGGS. From INHABAD-GES. M.B.H. (B.P. 304,248, 17.1.29).

Ferric alumina ((U.S.P. 1,747,177). **Oxygen preparations** (U.S.P. 1,745,465).—See VII.

B.—APPLIED CHEMISTRY

AUG. 1 and 8, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Tests on a Birmingham [town's gas-fired oven-] furnace installation. C. M. WALTER (Gas J., 1930, 190, 576—577).—The developments in oven-furnace design which have occurred during recent years and the improvement of working efficiencies by the employment of special insulating materials, improved recuperator elements, the exact control of primary and secondary air, and the reduction of losses by leakage through doors and dampers are described. C. B. MARSON.

Critical relations between water and steam. H. L. CALLENDAR (Proc. Inst. Mech. Eng., 1929, 811—838).—Equations hitherto proposed to represent the pressure-volume relations of gases near the critical point are based on the theory of the continuity of state. Discrepancies occur in all cases between theory and observation, and it is suggested that these are due to an error in the fundamental assumption of molecular identity between the two states. As the latent heat of fusion of ice is so much less than that of the vaporisation of steam, the author considers it reasonable to suppose that in water linkings exist approximating to the hexagonal-plate structure of ice on Bragg's model. On this view the Joule-Thomson equation for water becomes $V - b = RT(V - c)$, in which c represents the defect of volume due to co-aggregation and is a function of V and T . The combination of this with the relation between total heat and volume gives very close values of the saturation pressures up to the critical point. Observation of the point of disappearance of the meniscus in a fresh quartz-glass tube gave a value for the critical temperature of 374.0° , a figure higher than that previously recorded. This figure was verified by latent heat measurements in the critical region, and the results obtained were found to be in accordance with the co-aggregation theory. Beyond the critical point there are no longer distinct phases, but two mixtures of different density can still be observed and the latent heat does not entirely vanish at the critical temperature. Pressure-heat diagrams are given and the advantage to be gained by increasing pressure is discussed. This is less than has been generally supposed, and steam near the critical point would require a very high degree of superheat to be fitted for direct use in a turbine. The constants and equations on which an international system of defining the properties of steam can be based are discussed. C. IRWIN.

Submerged combustion. HAMMOND. Coal-tar oil for furnaces. LUHN. Graphite lubrication. CORDEBAS.—See II. Rubber linings. COURBIER and BOUSSAND.—See XIV. Boiling of worts. WORSSAM.—See XVIII.

PATENTS.

Furnaces. I. W. FOLTZ (B.P. 329,010, 5.2.29).—In a boiler furnace having a fire-box, fire-bridge, and further combustion chamber, air is taken from the ashpit and preheated above the ignition point of the fuel in passages formed in the walls or floor of the combustion chamber and is then returned to the fire-box above the burning fuel and near the stoking door. B. M. VENABLES.

[Boiler] furnace and process of operating the same. H. J. KERR, Assr. to FULLER LEHIGH Co. (U.S.P. 1,747,676, 18.2.30. Appl., 8.10.27).—It is considered that the presence of incombustible dust in the combustion gases promotes transmission of heat to the boiler tubes (or other object to be heated). A dust catcher, of the type having vertical tubes which are slit longitudinally, facing the oncoming gases is placed in the flue and the dust collected therein transferred to a hopper, whence it is mixed with the pulverised fuel going to the boiler. B. M. VENABLES.

Apparatus [boiler furnace] for heating fluids. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 329,316, 9.1.29).—The combustion space has walls and roof formed of water-tubes, with or without external walls of non-conducting brick; in the lower part an internal lining of good conducting material such as silicon carbide is provided. B. M. VENABLES.

Furnace fronts. J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 329,879, 29.6.29).—A pulverised-fuel burner fitted to a furnace door is described. B. M. VENABLES.

Handling and removing furnace residues and other solid materials. ASH Co. (LONDON), LTD. (B.P. 329,840, 18.5.29).—The ash drops (continuously) into a large volume of water contained in a hopper; to remove the ash (intermittently) the water is first drained off, in order to prevent a sudden rush choking everything, and the self-supporting residue is sluiced away by jets of water. Suitable apparatus is described. B. M. VENABLES.

Continuous kilns. C. ROST (B.P. 311,246, 7.5.29. Ger., 7.5.28).—In a kiln of the Hoffmann type fired with powdered fuel, the mixture of fuel and air is admitted to the kiln at the lowest possible speed through a number of long narrow slots in the walls and roof and the passages leading thereto are preferably flared towards the chamber, with the object of obtaining a cloud of flame and consequent uniform heating. B. M. VENABLES.

Kiln or retort for continuous working. H. M. ROBERTSON (B.P. 317,066, 23.1.29. U.S., 10.8.28).—A tunnel kiln, stated to be suitable for recovering hydrocarbons from materials such as shale or briquettes, is

* The remainder of this set of Abstracts will appear in next week's issue.

constructed with preheating, heating, and cooling zones in line; each zone is provided with outlet conduits connecting with a suction device and is separately controllable. The walls of the preheating zone have projecting pillars built on them to cause the longitudinally travelling waste gases to eddy and circulate.

B. M. VENABLES.

Kiln bottoms. J. KNOWLES & CO. (WOODEN BOX), LTD., and H. J. TAYLOR (B.P. 329,443, 25.3.29).—The bottom of a downdraught kiln formed of perforated blocks has specially shaped perforations over the supporting walls so that gases may leave uniformly over the whole floor.

B. M. VENABLES.

Heat exchangers. GRISCOM-RUSSELL CO., ASSEES. OF K. B. RIS (B.P. 308,715, 19.2.29. U.S., 27.3.28).—In an exchanger of the bundle-of-tubes type, the outer fluid is caused to flow in an alternate rotating and axial manner by means of baffles strung on the tubes which are alternately (a) flat, having a clear passage through the centre; and (b) bent, having no perforations other than those occupied by the tubes and bolts, but being slit radially nearly to the centre; the sector-shaped blades thus formed are twisted so as to afford helical, circumferential passages for the outer fluid.

B. M. VENABLES.

Heat exchanger. L. S. QUENSEL and F. A. STEPHENS (U.S.P. 1,746,497, 11.2.30. Appl., 11.12.28).—A number of hollow, tapering drums are assembled on a hollow shaft forming a bellows-like structure which is rotated in the liquid to be heated (or cooled); the heating medium is supplied and removed through the hollow shaft, which has driving means and a stuffing box at the upper end and a bearing at the bottom.

B. M. VENABLES.

Heat interchangers. F. I. E. STENFORS (B.P. 329,714, 26.2.29).—In an exchanger of the conducting-plate-and-frame (filter-press) type, in which the two fluids flow at right angles to each other in alternate frames, the transfer ports are made as long as the internal width, and as wide as the thickness, of the frames, the latter being bevelled internally to permit free flow of the fluids at the return bends.

B. M. VENABLES.

Heat-interchange apparatus. G. CAHILL (U.S.P. 1,745,978, 4.2.30. Appl., 24.2.27).—An exchanger similar to a honeycomb radiator is provided with specially-shaped tubes for use round the sides to give a flat surface to which the frame may be attached. Adjacent the inlet and outlet connexions more space is provided for the fluid by reducing the size of the intermediate parts of the honeycomb tubes much below that of their polygonal ends, e.g., by flattening them to cruciform, crescent, or other shape.

B. M. VENABLES.

Plate apparatus for heat exchange. R. SELIGMAN (B.P. 310,411, 15.3.29. Holl., 25.4.28. Addn. to B.P. 327,377; B., 1930, 490).—In an apparatus as described in the prior patent, the flow of vapour is zigzag vertically, but passages are left at the lower ends of the ribs for direct flow of condensate, these passages being small and increasing in size towards the outlet.

B. M. VENABLES.

Catalytic apparatus. A. O. JAEGER, ASSR. to SELDEN CO. (U.S.P. 1,741,309, 31.12.29. Appl., 13.12.27).—In

an apparatus suitable for gaseous reactions which are strongly exothermic or need close regulation of the temperature, the catalyst is in elongated annular form and is contained in large-bore tubes surrounded by a bath of liquid. The axial spaces in the catalyst masses are occupied by twin concentric tubes through which at least a part of the incoming gases passes to and fro. The liquid may be metallic or not, and may boil at, or slightly above or below, the required reaction temperature; or it may be non-boiling, in which case it should be mechanically circulated.

B. M. VENABLES.

Gaseous exothermal catalyses. UNION CHIM. BELGE, SOC. ANON. (B.P. 329,551, 24.6.29. Belg., 13.11.28).—A portion only of the incoming gases is subjected to heat exchange with the outgoing gases, but the whole of the former is passed through the catalyst and through an annular space between the pressure-resisting wall and the heat-exchanging zone, the adjustable valve for dividing the gas coming after that pass. A heater is provided in the central catalysing zone, but is normally used only for starting.

B. M. VENABLES.

Devices for carrying out exothermic catalytic reactions. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, ASSEES. OF SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.) (B.P. 307,840, 13.3.29. Morocco, 14.3.28).—When the catalyst has to be cooled by the incoming gases (or other fluid) passing through an annular space round the catalyst chamber, that space is filled with metal gauze or other discontinuous material which improves heat transmission by forming eddies, steadies the flow, and prevents warping due to expansion and contraction.

B. M. VENABLES.

Apparatus for carrying out reactions continuously in the liquid phase under increased pressure. IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,260, 28.10.29. U.S., 29.10.28).—The apparatus comprises two concentric cylindrical chambers, the outer one being pressure-resisting and the inner having separate communication with external apparatus at the bottom, but opening at the top into the outer one, i.e., there is a vapour space common to them both. Part of the annular space is filled with Raschig rings (or similar device) and an inlet pipe is provided reaching to a distributor plate on top of the filling; pipes are connected to the bottom of each chamber for inlet or outlet according to circumstances, and at the top connexions are made for a pressure gauge and for drawing off permanent gases. A device to indicate when the central stand-pipe or chamber is full of liquid may be added.

B. M. VENABLES.

Water-cooling towers. L. G. MOUCEL & PARTNERS, LTD., and M. E. GERARD (B.P. 329,715, 26.2.29).—A cooling tower of hyperboloidal form is formed of straight timbers set at an angle (not perpendicular) to the ground with their lower ends on a circle and their intersections bolted together.

B. M. VENABLES.

Drying apparatus. W. M. STRONG, ASSR. to GEN. DRYING ENG. CORP. (U.S.P. 1,749,451, 4.3.30. Appl., 17.4.28).—The material, such as lucerne, is carried on a conveyor, which is enclosed above and below and is subjected to upward flows of hot air from a furnace and fan at the charging end. A proportion of the gases

when hottest pass through the material when wettest, but another regulable portion passes on through a door and percolates through the partly dried material at a lower speed, the length of conveyor forming the secondary zone being the longer. The moist gases from the primary zone are exhausted to a chimney, but the secondary gases are returned to the feed end and mixed with fresh gases from the heater. B. M. VENABLES.

Vacuum drying apparatus. A. E. JONSSON (B.P. 306,037 and 306,088, 11.2.29. Swed., [A] 14.2.28, [B] 15.2.28).—(A) A rotating or oscillating cylinder contains a number of hollow shelves arranged the full length of the cylinder and grouped in parallel on both sides of a diametral space, which affords communication to man-holes for charging and discharging. The material being dried is intended to slide rather than tumble from shelf to shelf. The shelves, also the shell, if desired, are heated by a fluid in the spaces or jackets. (B) An apparatus similar in principle to that described in (A) is intended for non-powdered goods which are supported clear of the heat-radiating shelves by non-conducting ribs. B. M. VENABLES.

Straining device for vacuum-drying apparatus. A. E. JONSSON (U.S.P. 1,748,955, 4.3.30. Appl., 21.3.28. Swed., 9.4.27).—A strainer for protecting the outlet pipe of a vacuum dryer is described. It comprises a cylindrical slotted surface, the slots being kept open by a comb that can be moved to and fro. B. M. VENABLES.

Centrifugal dryers. H. C. BEHR (U.S.P. 1,749,369 and 1,749,370, 4.3.30. Appl., [A] 3.8.28, [B] 23.8.28).—The cycle of operations of these centrifuges is controlled by a number of valves operated in sequence by a cam-shaft. A quantity of the pulp is drawn into a metering chamber by a vacuum and then propelled into the slowly rotating basket by air pressure. After centrifuging, a wash may be applied, and then, in (A), the basket, being cylindrical, is stopped and the material blown inwards off the screening surface by a pressure fluid, whence it drops out through bottom-discharge ports. In (B) the basket is the space between two cones of different angle, the upper one carrying the screening surface and the lower one the scrapers. It runs at the same speed as the upper one when centrifuging, but at a lower speed when discharging the collected cake, the change being effected by a hydraulic gear under control of the cam-shaft. B. M. VENABLES.

Absorption method and apparatus. F. G. WHEELER, Assr. to BLEACH PROCESS Co. (U.S.P. 1,747,687, 18.2.30. Appl., 5.12.25).—The apparatus may be constructed in stoneware and is suitable, e.g., for absorbing chlorine in milk of lime. The liquid flows downward from the narrow end of an expanding pipe with a velocity insufficient to form a jet; the expanding portion of the pipe is pierced by downwardly inclined holes through which the gas is admitted from a surrounding T-piece. The liquid tends to break away from the wall of the pipe at the perforations, thus producing eddy currents which carry bubbles of gas into the body of liquid. B. M. VENABLES.

Absorber. J. A. CAMPBELL (U.S.P. 1,748,409, 25.2.30. Appl., 17.12.27).—In a bubble tower having decks composed of twin perforated plates, the space between becoming filled by a mass of froth which breaks

down above the upper plate of a pair, provision is made for running the liquid down from the top of an upper plate to the lower plate of the deck below through an annular space formed by a double shell. To avoid intermingling of the streams from alternate plates the flow is only permitted down one side alternately and the space is divided into semi-annuli by means of vertical ribs. B. M. VENABLES.

Bubble tower. W. E. TETER (U.S.P. 1,748,855, 25.2.30. Appl., 18.2.28).—The upflow pipes are of inverted J-shape, and on top of these is a perforated plate from the upper surface of which the downflow is taken, preferably by a single pipe which branches lower down. The cross-section of the tower may increase in the upper part. B. M. VENABLES.

Digestors. SILLER & RODENKIRCHEN GES.M.B.H., and W. SILLER (B.P. 329,600, 25.9.29).—The digester, suitable for the extraction of glucose from wood by means of acid, rotates on a horizontal axis, the heating fluid to a jacket and the charge to the digester being admitted through one trunnion and exhausted through the other. The inlet pipe for material is taken to the bottom and the outlet comes from the top of the internal space, both being curved in such a way that the rotation tends to keep the openings clear. (Cf. B.P. 325,760; B., 1930, 399.) B. M. VENABLES.

Disintegrator. W. M. DUNCAN (U.S.P. 1,745,478, 4.2.30. Appl., 1.7.27).—A rotor drives two sets of balls or rolls which make contact with the casing, each element being in a separate pocket on the rotor. The disintegration is effected by a combination of three actions, viz., rolling between the rolls and casing, attrition between the rolls and driving pockets, and impact; provided the centrifugal force is not too great, the rolls will make contact with alternate faces of the driving pockets, according to whether they are rising or falling. B. M. VENABLES.

Apparatus for breaking and handling solid materials. ASH Co. (LONDON), LTD. (B.P. 312,057, 26.4.29. U.S., 19.5.28).—Under a boiler or similar furnace are a number of alined hoppers for discharge of ash, a transferable crushing unit is placed under each opening in turn, and the crushed product drops into a sluice, whence it is carried by water to an additional crusher which renders it fine enough for the pulp to be disposed of by pumps. B. M. VENABLES.

Crusher. W. I. BOWDEN, Assr. to AMER. ENG. Co. (U.S.P. 1,748,828, 25.2.30. Appl., 20.11.26).—An intermediate bearing for a long crusher roll suitable for boiler ash-pockets is described. B. M. VENABLES.

Crusher. R. BERNHARD, Assr. to TRAYLOR ENG. & MANUF. Co. (U.S.P. 1,748,102, 25.2.30. Appl., 24.5.29).—To prevent damage by uncrushable material, the upper end of the shaft of a gyratory crusher is yieldingly supported by a lever and spring. B. M. VENABLES.

Crusher. J. E. KENNEDY (U.S.P. 1,748,484, 25.2.30. Appl., 21.5.28).—The drive is transmitted to the shaft of a gyratory crusher by means of an electromagnetic friction clutch, to prevent damage by uncrushable pieces, to aid restarting, and to permit the use of a synchronous motor. B. M. VENABLES.

Reinforced crusher plate. C. E. HARRISON, Assr. to AMER. ENG. Co. (U.S.P. 1,748,879, 25.2.30. Appl., 22.7.25).—A crusher plate or jaw for severe service, such as in the ash-pockets of pulverised-fuel furnaces, is formed with steel or wrought-iron rods embedded in the cast metal and is deeply ribbed on the back.

B. M. VENABLES.

Pulveriser. G. D. HARRINGTON (U.S.P. 1,748,050, 18.2.30. Appl., 17.11.26).—The material is fed on to a horizontally rotating disc having upstanding radial beaters which fling the material against a toothed or corrugated stationary ring. Air enters with the feed, picks up fine material, and enters a fan above through a central port. Various adjustments are provided for the air current to determine the size of the delivered material.

B. M. VENABLES.

Pulverising machine. E. H. ELZEMEYER, Assr. to AMER. PULVERIZER Co. (U.S.P. 1,748,214, 25.2.30. Appl., 9.10.25).—In a disintegrator having hammers in the form of loose rolling rings, the edges of the rings are made wavy (when viewed edgewise, but not when viewed axially) to prevent packing of the material upon the grids or other stationary surfaces.

B. M. VENABLES.

Pulverising mills. INTERNAT. COMBUSTION, LTD., and F. H. ROSENCRANTS (B.P. 329,061, 1.3.29).—In an air-borne pulverising system, hot air is supplied by a heater of the type in which additional air is mixed with the products of combustion, the hot air passes through an explosion relief chamber which is constructed to serve also as a dust settler, and is drawn into the main grinding circuit by the action of the circulating fan, excess air being drawn from an earlier point of the same circuit through a dust separator by an additional fan. To prevent deposition of dew in the dust separator, hot air may be admitted thereto directly from the explosion chamber by means of a regulable by-pass.

B. M. VENABLES.

Pulverising mill. F. J. BULLOCK, Assr. to PAPEC MACHINE Co. (U.S.P. 1,748,046, 18.2.30. Appl., 5.7.28).—In a disintegrator the casing is supported in such a way that it can oscillate slightly in the plane of rotation of the beaters, and it is pulled back against the rotation by a spring; when grinding takes place the drag of the material will move the casing and extend the spring, this motion being utilised to vary the opening from a stationary feed hopper, so that the power required for grinding is held at a steady value.

B. M. VENABLES.

Pulverisation of materials. M. W. ARROWOOD (U.S.P. 1,747,922, 18.2.30. Appl., 25.5.28).—A ball-, pebble-, or rod-mill is provided with a drum of grid-like structure and is surrounded by a fixed casing; interrupted helical vanes are attached to the outside of the drum and push material to one end of the casing where it is picked up by scoops and returned to the drum. The fine material is carried away in a current of air, which is blown in at one end of the casing and exhausted at the other, and travels in a spiral direction round the drum. The pulveriser is particularly suitable for coal.

B. M. VENABLES.

Comminution of material. R. C. NEWHOUSE, Assr. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,748,920, 25.2.30. Appl., 23.2.28).—In order to obtain a product

containing a high proportion of very fine material from a tube-mill or similar grinder without correspondingly reducing the capacity of the air separators, the latter receives material from the last but one compartment of the mill and delivers its undersize to the last compartment for final grinding without subsequent air separation.

B. M. VENABLES.

Grinding machine. H. C. SCHNEIDER, Assr. to FAIRBANKS, MORSE & Co. (U.S.P. 1,745,646, 4.2.30. 18.11.26).—In a machine of the rotating hammer or knife type, the hammers, spacers, and inner races of the bearings are threaded on a shaft which is in the form of a bolt and the whole is clamped together by tightening up nuts on the end of the shaft.

B. M. VENABLES.

Grinding bodies for tube- or drum-mills. HELIPEBS, LTD., and R. McDUGALL (B.P. 329,131, 24.4.29).—The bodies comprise square helices formed from square or rectangular metallic wire or rod.

B. M. VENABLES.

Apparatus for testing materials [for hardness]. J. GOGAN (U.S.P. 1,746,891, 11.2.30. Appl., 28.2.27).—A machine suitable for rapidly effecting, without special preparation, a ball test on every piece of material manufactured, causes the test ball to advance under sufficient pressure to penetrate the skin and then from this point as zero to advance a predetermined distance, e.g., 0.01 in., the extra pressure being the criterion of the hardness and the dent too small to spoil the piece for use. In practice a few pieces are checked by known refined methods and are then used as standards in the machine, the dial being set to zero for the maximum load on the standards, and the production pieces being passed if the reading on the dial does not go beyond prescribed limits.

B. M. VENABLES.

Means for measuring the hardness and thickness of [non-rigid] objects. H. A. DAYNES, E. B. JOHNSON, and RES. ASSOC. OF BRIT. RUBBER MANUFERS. (B.P. 329,629, 18.2.29).—The apparatus comprises a broad foot within the area of which is a loaded plunger which indents the test-piece and of which the advance relative to the foot is measured while under a definite load. Both the foot and the plunger rest on the test-piece, or, when taking the zero reading, on a rigid surface such as a sheet of glass. For measuring the hardness of a curved object such as a rubber-covered roller a pair of cylindrical rods suitably held in grooves take the place of the foot and the zero must be obtained on a rigid body of equal radius. For measuring the average thickness of a sheet the foot alone rests on the test-piece and the plunger on the rigid support.

B. M. VENABLES.

Imparting compactness and coherence to a granular mass contained in a mould, and building block made therewith. R. LEJMARK (B.P. 329,063, 4.3.29).—The mould containing the mass (e.g., concrete) is subjected to jarring by allowing it to fall upon a heavy bed-plate and is then raised against a ram which effects smoothing of the upper portion. A machine is described which effects both motions by the same foot-operated mechanism.

B. M. VENABLES.

Manufacture of materials in thin sheet form. W. H. PEASE (B.P. 329,691, 25.1.29).—A fluid material is supplied from a hopper through a slot, with micrometer

adjustment, to a drum, and is spread thereon by adjustable knives. The temperature, humidity, etc. of the material, drum, and surrounding atmosphere (contained in an enclosure) are controlled so that the material leaves the drum in a solid or semi-solid state. An insoluble material may be interposed between the solution and the drum, thus forming a two-ply sheet. B. M. VENABLES.

Mixing machine. G. J. HICKS (U.S.P. 1,745,993, 4.2.30. Appl., 17.10.28).—A mixing machine suitable for confectioners' use comprises a trough-shaped container, one end of which is entirely removed for discharging purposes; the beaters are therefor journaled only at the other end. The solid material is fed from a hopper above by means of a screw-feeder and the liquid may be injected by compressed air either at the end of the feeder or upwards through the bottom of the trough. The temperature may be regulated by coils on the lid of the trough. B. M. VENABLES.

Automatic machines for mixing different products in specified proportions. SOC. DES SUCRERIES TERNYNCK (B.P. 316,862, 22.4.29. Fr., 3.8.28).—Tipping dishes are provided for each material which are supported on scale beams in such a way that they are locked upright for filling, but when the counterpoise is balanced they overturn through 90° and discharge, which act transfers the centre of gravity to the other side of the pivot and causes the pan to turn back for filling again. The actual filling of the dishes is effected by any known device that can be instantly stopped, and the dish for the primary material governs the operation of the others in such a way that the latter can only return to the filling position when the primary is emptying; interlocks are provided to stop the whole mechanism in the event of deficiency of any material or if the receiving container becomes too full. B. M. VENABLES.

Separator. C. G. HAWLEY, ASST. to CENTRIFIX CORP. (U.S.P. 1,746,253, 11.2.30. Appl., 29.6.26).—An entrained substance (fluid or solid) is separated from a carrier fluid (gas, vapour, or liquid) by projecting the mixture downwards, deflecting it laterally then downwards, and allowing the cleaned carrier fluid to turn inwardly and upwardly and to pass out through the original stream where it is flowing laterally. The apparatus comprises a collecting hopper, above which are arranged deflectors to effect flow as described above, the outlet being situated annularly round the inlet. B. M. VENABLES.

Separating apparatus and method. W. T. BIRD-SALL (U.S.P. 1,747,155, 18.2.30. Appl., 15.10.25. Renewed 25.6.29).—A separator for fluids, or fluids and solids, of different density or viscosity is based on the fact that the film on a rotating shaft has laminar motion with respect to the body of fluid which may or may not have eddying motion. The apparatus is intended to drag a film of a single constituent into another compartment; centrifugal force of the order of 200,000 times gravity may be developed tending to urge foreign matter out of the laminar film. Several forms of apparatus are described: one is like a plain journal bearing with one arc of bearing surface much longer than the opposite one; others have screw-threaded casings surrounding a plain or screwed shaft. B. M. VENABLES.

Separation of intermixed divided materials. R. PEALE and R. PEALE, JUN. (B.P. 328,663, 31.12.28. U.S., 18.12.28).—An apparatus for the dry separation of particles which differ greatly in size but not much in sp. gr. comprises a shaking table which has a bed of varying porosity to upward air currents which can be controlled in zones. After stratification the products are delivered by inclined guides. B. M. VENABLES.

[Wet] separators [for ores etc.]. J. and J. O. SPROUL (B.P. 329,030, 15.2.29).—A separator of the type in which a conveyor carries the heavier constituents against a stream or streams of water has the conveyor composed of a number of shallow buckets or skips of which the bottom from the leading end is gradually inclined and at the back rises in a steep curve over the edge of the succeeding bucket. B. M. VENABLES.

Separation of minerals [containing flat and granular particles]. J. BLAND (U.S.P. 1,749,371, 4.3.30. Appl., 20.9.26).—Mixed minerals, such as mica and feldspar, are dropped in an annular stream within the inner edges of a number of superposed, spaced, truncated, conical baffles, against an upward current of air which is insufficient not only to affect the downward flow of the granular particles, but also to lift the flakey particles of relatively large surface; it does, however, cause the latter to eddy and escape from the air current outwardly between the baffles. To obtain clean products of both constituents a number of units may be employed in series. B. M. VENABLES.

Separation apparatus. B. A. MILLER (B.P. 328,940, 7.1.29).—A pulp flowing down an inclined V-shaped sluice is given a side-to-side swirling motion by curved baffles attached to either side of the sluice alternately, thus causing the heavier concentrate to collect in the bottom of the sluice, whence it is removed through outlets controlled by internal lips. B. M. VENABLES.

Centrifugal separator. H. O. LINGREN, ASST. to DE LAVAL SEPARATOR Co. (U.S.P. 1,749,291, 4.3.30. Appl., 24.8.28. Swed., 26.10.27).—The separator is suitable for the continuous separation of materials such as wool scourings, both dirt and fats being separated from the water. The bowl is slightly conical, and has an outlet for dirt (and water) leading from the largest circumference to a point nearer the axis, through which the water etc. passes before being finally discharged. The outlet for middle fluid (water) leads from the smallest circumference above the top separating disc to outlet jets at an intermediate radius. The lightest liquid leaves the spaces between the discs at their inner edges and flows upwards to an inner neck. B. M. VENABLES.

Apparatus for separating solids from liquids. A. L. ARMENTROUT (U.S.P. 1,749,057, 4.3.30. Appl., 11.11.26).—The solid is settled centrifugally in a truncated conical rotor (or by gravity in a tank with sloping bottom), a carrier liquid of intermediate sp. gr. being added at the smaller circumference in such a manner that it will not emulsify with the lighter liquid. Separate outlets are provided for carrier liquid with dirt, excess carrier liquid, and lighter liquid, and the supply of carrier liquid is maintained by a pump rotating with the centrifuge. B. M. VENABLES.

Apparatus for the clarification of liquids and separation of finely-powdered solid substances [by flotation]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,013, 7.2.29).—The suspended particles are removed in a foam produced by the downward impingement of a jet of the cloudy liquid upon the surface of a column of the clarified liquid. A column of foam several metres high may be produced; it moves countercurrent to the jet, but is otherwise unobstructed. A wetting agent may be added previously, and the process may be applied to the separation of different solids; *e.g.*, in a gas-purification process a mixture of regenerated ferric hydroxide and sulphur may be treated so that the former will sink and the latter float.

B. M. VENABLES.

Filter. J. E. OTIS, JUN., ASSR. to ALEMITE CORP. (U.S.P. 1,746,274, 11.2.30. Appl., 30.11.25).—A strainer suitable for connecting to the outlet of a tank is described. When the former is removed for cleaning, the supply of liquid is automatically cut off.

B. M. VENABLES.

Filter leaf. E. J. SWEETLAND and J. V. ZENTHOEFFER, ASSRS. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,746,409, 11.2.30. Appl., 6.12.26).—Sectors for rotating-leaf filters, especially suitable for use with metallic cloth backed by heavier wire gauze, are constructed of cast metal (*e.g.*, aluminium) with a cored central outlet, suitable for bolting to the rotating hub, with ribs in the drainage space and with twin flanges round the radial and circumferential edges, all cast in one piece. The flanges are drilled for securing the filter medium by rivets or other means, and the radial flanges of adjacent segments abut together and embrace spokes attached to the hub; the ends of the spokes are provided with nuts and bridge washers to clamp the sectors.

B. M. VENABLES.

Hydraulic filter press. W. H. MATHERS, ASSR. to R. DUDGEON, INC. (U.S.P. 1,749,080, 4.3.30. Appl., 25.3.27).—The press is tightened by a hydraulic ram and the preliminary closing is by a rapid-acting screw.

B. M. VENABLES.

Apparatus for producing [edge-]filter members. W. L. KRAEMER and A. C. PURDY, ASSRS. to METAL EDGE FILTER CORP. (U.S.P. 1,745,426, 4.2.30. Appl., 10.6.27).—An apparatus is described suitable for the rapid manufacture of washers with slight projections on the surface, for use as filter packs from a long metal strip. The steps comprise cleaning, drying, applying printers' ink or other tenuous film to the unraised portions of the strip, plating the bare parts and thus producing the projections, cleaning off the ink, and punching out the washers.

B. M. VENABLES.

Dialysing filter-presses. FILTRES PHILIPPE, and E. HEIBIG (B.P. 329,235, 14.8.29).—The construction is similar to that of an ordinary filter-press with additional frames substituted for the usual plates, and with the ports arranged to give series flow of two fluids, each one through every alternate frame, the general flow being countercurrent on opposite sides of dialysing diaphragms in the place of the usual filter cloths. Lattice work may be inserted in the frames to cause the liquid to travel in a longer course within the frames.

B. M. VENABLES.

Purification [of vapours in distillation] apparatus. V. SEEHOLZER (Swiss P. 124,999, 16.3.37).—At least one removable horizontal plate is arranged in the path of the vapours through the condenser to serve as a condenser for the less volatile portion of the vapours.

A. R. POWELL.

Fractionating system. Method of fractionation. M. B. COOKE, ASSR. to ATLANTIC REFINING CO. (U.S.P. 1,748,411 and 1,748,508, 25.2.30. Appl., [A] 24.7.25, [B] 21.3.29. Renewed [A] 20.2.29).—(A, B) In a fractionating column the decks are provided with bubbling caps, and with downflow pipes at alternate ends of a diameter, the downflowing liquid being kept away from the rising vapour. Above the caps is a mass of inert ceramic or other filling which catches any entrained liquid, the dried vapour then passing through a space for vapour only before reaching the next bubbler. The filling is supported on a perforated platform, which may be formed from the tops of the bubble caps themselves, suitably shaped and registering.

B. M. VENABLES.

Method of fractionation. Fractionating column. R. B. CHILLAS, JUN., and A. G. PETERKIN, JUN., ASSRS. to ATLANTIC REFINING CO. (U.S.P. 1,748,595 and 1,748,704, 25.2.30. Appl., 24.11.25).—In the spaces between the decks of a bubbling column are placed moisture eliminators, permitting increased output owing to the fact that entrainment produced in the bubblers may be ignored and the velocity of the vapours raised to 2–6 ft./sec. The eliminators must not occupy much of the cross-section of the tower, and they return the liquid to the tray below in which the spray was produced; they may consist of parallel V-shaped sheets.

B. M. VENABLES.

Dephlegmator or fractionating column. L. C. HUFF, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,741,519, 31.12.29. Appl., 10.12.26).—The decks of a fractionating column are provided with the usual downflow pipes maintaining a layer of liquid; the upflows are in the form of ejectors, the rush of vapour through which entrains liquid, thereby lifting it in the form of a spray or film and returning it to the same tray.

B. M. VENABLES.

Fractional condensation of composite vapours. A. LOSEY, P. GOTTLIEB, and L. L. HAUPT (B.P. 329,418, 4.3.29. U.S., 31.1.29).—Elaborate systems involving expansion engines and the use of several different pressures are described.

B. M. VENABLES.

Congeeing of liquid or semi-liquid substances. C. W. VOGT (B.P. 329,129, 23.4.29).—The liquid is sprayed on to a refrigerated surface and continuously removed therefrom. The atmosphere is also chilled by a refrigerating surface, not in contact with the spray, to such an extent that partial congelation takes place before the spray reaches the first-mentioned surface. Materials such as ice-cream are preferably pasteurised and homogenised before spraying, and the mist produced should be so fine that the frozen product is aerated by the atmosphere.

B. M. VENABLES.

Electric hygrometer. W. LUBACH (U.S.P. 1,749,826, 11.3.30. Appl., 7.6.27. Ger., 8.6.26).—A pair of closely-spaced, convoluted conductors, preferably of nichrome, inserted in an electrical circuit including a source of

constant potential and an indicating instrument, *e.g.*, a galvanometer or cathode-ray oscillograph, are bridged by non-conducting material impervious to moisture, but upon which moisture can condense, *e.g.*, glass-wool. A current will then flow between the conductors when moisture is deposited on the material, the magnitude of the current being a measurement of the humidity of the atmosphere surrounding the conductors.

J. S. G. THOMAS.

Refractometers for liquids. C. ZEISS (B.P. 314,994, 1.7.29. Ger., 6.7.28).—A refractometer is provided with a knurled ring operating a deflection device by which the boundary line between the light and dark part of the field may be adjusted to a mark; when this is effected the scale showing refractive index (or concentration in the case of sugar solutions) appears superposed on a number of curved isothermal lines, so that the result can be read at once according to the known temperature.

B. M. VENABLES.

Instrument for measuring fluids for turbidity, colour, and other characteristics of fluids. W. G. EXTON (B.P. 328,556, 24.1.29).—A cylindrical casing has at one end a source of light and at the others an eye-piece; intermediately are transverse plates with central apertures carrying, in order, a target, a test-tube containing the sample of liquid, a lens, a calibrated wedge of which alternative types are provided either absorbing or dispersing the light till the target disappears, and a set of colour screens. To prevent the final observation being made in darkness an annular space is left round all the plates, and the casing is made reflecting so that diffused light always reaches the eye-piece. Alternatively, the sample itself may be contained in a wedge-shaped container and adjusted till it extinguishes the target. When desired, a comparison field may be simultaneously viewed by means of a Fresnel rhomboid prism.

B. M. VENABLES.

Air filtering, heating, humidifying, and like apparatus. T. ANDREW (B.P. 328,948, 6.2.29).—The air is passed from the inside outwards through a number of tubular fabric filters, which are supported in a horizontal position by means of spring tension applied to the closed end. The humidifying etc. may be done in a subsequent chamber.

B. M. VENABLES.

Air filter. H. E. BIRKHOLZ, Assr. to AMER. AIR FILTER Co., INC. (U.S.P. 1,747,694, 18.2.30. Appl., 30.3.25).—The apparatus consists of a slowly rotating, drum-shaped screen formed from a spongy metallic mass through which the air passes inwards and leaves by one end which is open. The screen dips into a bath of oil, clean oil being supplied to the lower part of the interior by sprays so arranged that the screen is washed by a flow of oil in a direction opposite to that of the air.

B. M. VENABLES.

Air filters. C. G. VOKES (B.P. 329,099, 28.3.29).—The filter medium is composed of several layers of wire gauze of the same or different mesh, with or without felt in between; the laminated sheet thus produced is pleated without substantial separation of the layers.

B. M. VENABLES.

Air filter. W. M. REED and C. P. HEGAN (U.S.P. 1,746,283, 11.2.30. Appl., 3.3.26).—A method

of fastening filter cells to supporting frames by means of latches is described.

B. M. VENABLES.

Apparatus for extracting smoke and other solid particles from air. T. D. AUSTIN and C. DEWHURST (B.P. 329,501, 14.5.29).—The apparatus is of the dry centrifugal type, separate fans being used to draw in the air and to centrifuge it.

B. M. VENABLES.

Air separator. T. J. STURTEVANT, Assr. to STURTEVANT MILL Co. (U.S.P. 1,746,686, 11.2.30. Appl., 23.6.28).—The separation is effected in two upward, outward (through the blades of a fan), downward, inward circulations one above the other. The material is fed from a hopper to a distributing plate situated in the upward current of the upper circulation, and the oversize which is not carried up falls on to a similar distributor in the lower circuit, three products being thus obtained.

B. M. VENABLES.

Gas and air cleaner. A. F. JENKINS (U.S.P. 1,746,774, 11.2.30. Appl., 10.9.27).—The gas etc. is admitted downwards through an axial tube nearly to the bottom of a cylindrical container, and on its return flow upward passes through a number of filter elements which may be composed of (lower) convex plates having small perforations surmounted by concave plates with larger perforations. Application to air for paint-spray apparatus is indicated.

B. M. VENABLES.

Treating [cleaning and cooling] gases. A. G. MCKEE (U.S.P. 1,748,723, 25.2.30. Appl., 1.7.25).—A tower is provided with a number of separately driven, superposed, spraying devices each of which can be stopped and repaired without affecting the operation of the rest of the tower, access being afforded by work-passages. The earlier stages are supplied with hot water, which removes the solid matter, and is then passed to a settler where the bulk of the mud settles out. The water is returned to the same stages of the tower, though still containing sufficient solids to be a legal nuisance if it were discarded, but yet quite clean enough to use again; the elevated temperature promotes both the entrainment of the dust and the settlement in the thickener. All losses of water from this circuit are made up from the later cooling circuit in the upper stages, in which another supply of water is circulated through the sprays and a cooler, losses being made up with fresh water, the consumption of which is much reduced when compared with that from methods previously used.

B. M. VENABLES.

Apparatus for removing oil from gases. FREINS JOURDAIN MONNERET Soc. ANON. (B.P. 306,899, 18.2.29. Fr., 27.2.28).—The gases are passed in a zigzag path through a chamber composed of heat-radiating (external) and baffling (internal) sections; other baffles are also formed round the central pipe admitting the gas.

B. M. VENABLES.

[Spectroscopes for] analysing gases or vapours. V. HENRI (B.P. 329,111, 9.4.29).—Two forms of spectro-scope, suitable for the quantitative analysis of gases by ultra-violet light, are described. In one form the light rays from an electric lamp fitted with a quartz bulb are collected by a lens, passed in turn through a chamber for the gas, formed out of the tube of the

instrument and a pair of non-absorptive windows, then through a slit diaphragm, two prisms each of which can be rotated, to a fluorescent screen or photographic plate the angle of which can be varied and for the former a magnifying eyepiece may be provided. The other form makes use of a concave diffraction grating instead of the prisms. The determination of the concentration may be effected either by actual measurement of the energy absorbed by the bands, *e.g.*, by thermocouple, or, usually more conveniently, by comparison with a standardised gas. A convenient method of adjustment is to alter the pressure of the sample.

B. M. VENABLES.

Fluid composition for treating brake and clutch linings. LIQUID VENEER CORP., ASSCS. OF E. F. GINGRAS (B.P. 307,342, 14.2.29. U.S., 5.3.28).—Worn parts of a lining are built up by a suspension of particles of friction material in a vehicle comprising a gum in a suitable solvent.

B. M. VENABLES.

Production of high vacua. KODAK, LTD., ASSCS. OF K. C. D. HICKMAN (B.P. 329,918, 22.8.29. U.S., 19.2.29).—A vacuum pump of the vapour-jet type is described suitable for use with an organic liquid having a vapour pressure at 0°, which is less than the pressures used in high-vacuum work, and which is not decomposed by prolonged boiling, *e.g.*, *n*-dibutyl phthalate or other aromatic ester. The advantage over mercury is that the condensing vapour wets the surface of the apparatus, does not form globules, and uncondensed vapour is less likely to diffuse into the vessel being evacuated.

B. M. VENABLES.

[Pyrometer] device for measuring the temperature of hot fluids. R. HASE (U.S.P. 1,761,392, 3.6.30. Appl., 15.12.24. Ger., 28.1.24).—See B.P. 231,398; B., 1925, 482.

Liquid-circulating devices for absorption refrigerating apparatus. Refrigerating apparatus. ELECTROLUX, LTD., ASSCS. OF PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 309,545 and 312,359, [A] 5.4.29, [B] 16.5.29. Ger., [A] 12.4.28, [B] 25.5.28).

Apparatus for washing minerals by means of liquid streams. A. FRANCE (U.S.P. 1,759,940, 27.5.30. Appl., 30.12.26. Belg., 30.12.25).—See B.P. 285,987; B., 1928, 320.

[Dual] filter. J. ZWICKY (U.S.P. 1,759,927, 27.5.30. Appl., 26.9.25).—See B.P. 258,905; B., 1927, 176.

Separation of liquids of different specific gravities. W. A. WHITE, ASSR. TO WHITE OIL SEPARATORS, LTD. (U.S.P. 1,761,505, 3.6.30. Appl., 27.1.28. U.K., 7.6.27).—See B.P. 285,707; B., 1928, 507.

Method for atomising and drying liquids. A. NYROP (U.S.P. 1,762,026, 3.6.30. Appl., 3.8.26. Denm., 10.5.26).—See B.P. 275,404; B., 1927, 801.

Refractometer for liquids. F. PFEIFFER, ASSR. TO C. ZEISS (U.S.P. 1,760,209, 27.5.30. Appl., 29.6.29. Ger., 6.7.28).—See B.P. 314,994; preceding.

Re-gasification of liquefied gases. C. W. P. HEYLANDT (U.S.P. 1,747,366, 18.2.30. Appl., 16.2.28. Ger., 29.3.27).—See B.P. 287,909; B., 1929, 501.

[Device for] controlling coal-dust furnaces [of locomotives etc.]. R. ROOSEN (B.P. 317,372, 7.8.29. Ger., 14.8.28).

Suspended roofs for furnaces and the like. A. REPPMAN (B.P. 330,326, 18.3.29).

Furnace walls. H. W. SPENCER, and LIPTAK FIRE-BRICK ARCH CO. (B.P. 330,351, 16.4.29).

Apparatus [valve devices] for aerating liquids under pressure. E. RYLES and J. SOPWITH (B.P. 328,924, 5.2.29).

Carbonic anhydride (CO₂) [refrigerating] compressors. W. F. JENNINGS (B.P. 330,007, 5.3.29).

Fine powders (B.P. 327,979).—See II. **Heat-insulating material** (G.P. 461,889).—See IX. **Heating of liquids** (G.P. 461,567). **Deoxidising system** (U.S.P. 1,745,875).—See XI. **Rubber-coated articles** (B.P. 328,686). **Porous body** (B.P. 306,129). **Brake liners** (B.P. 329,021).—See XIV. **Pasteuriser for liquids** (B.P. 329,733).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coal properties and its applications. T. KIDOKORO and G. SHIRANE (J. Fuel Soc. Japan, 1930, 9, 43—49).—A method for the identification of the geological rank of coals, lignites, etc. by their treatment with oxidising agents followed by precipitation of the humic acids with caustic soda is described. Experiments on the oxidation and spontaneous combustion of coals, with special reference to their rank, as determined above, are also discussed.

C. B. MARSON.

"Ball coals" (Mugelkohlen). S. VON FINÁLY (Brennstoff-Chem., 1930, 11, 188—189).—"Mugelkohlen" are round or elliptical masses of coal of varying size up to 40 cm. in diam., found in the coal seams in the neighbourhood of Pécs, Hungary. They are readily separated from the surrounding coal, and frequently, but not always, possess a more or less complete outer shell, 0.5—1.0 cm. thick. Slight but definite differences exist between the compositions (as shown by the ultimate analyses based on the ash-free, dry basis) of the "ball coal," the surrounding shell, and the coal-seam itself. In the two samples investigated, however, the ash of the "ball coal" (about 4%) was much less than that of the surrounding seam (about 19%). These coal masses have most probably been formed by the "rolling together" of particles of the original peaty material by the action of wind and water (cf. Gothan, Sitzungsber. Königl. Preuss. Akad. Wiss., 1910, 1, 136). A. B. MANNING.

Peat. III. Dry substance of peat. G. STADNIKOV (Kolloidchem. Beih., 1930, 31, 59—88; cf. B., 1930, 495).—Determinations of the dry residue of peat by drying in air at 105° give erroneous results, since simultaneous oxidation occurs and it is not possible to determine the end of the drying process. The weight is altered also by the decomposition of organic constituents at this temperature. The residue may be determined by heating at 80° in a stream of nitrogen, but a vacuum desiccator provided with electric heating is recommended. The weight of dry residue obtained from peat varies much with its nature, but lies between 9 and 15%. The ash content is obtained by ignition, and is found to

increase with the depth of layer from which the peat is obtained. Both the ash content and its composition vary greatly with the kind of peat; analysis have been made, and the principal constituents found were calcium, iron, aluminium, potassium, silica, sulphate, and phosphate. The composition of the ash is closely connected with the suitability of the peat as a combustible, especially in determining the porosity of the burning material, and it is said that the iron content catalyses the oxidation of the organic matter. The elementary composition of the organic constituents does not vary appreciably with the depth of layer from which the peat is taken. The nitrogen content of peat is considerably higher than that of other fuels; it often reaches 2.5% and rarely falls below 1.5%. The products of distillation of peat at different temperatures have been investigated, and the yields of coke, tar, gas, water, ammonia, acetic acid, and methyl alcohol at 525°, 710°, and 850° are tabulated.

E. S. HEDGES.

Further X-ray studies of carbonaceous and bituminous materials. C. MAHADEVAN (Indian J. Physics, 1930, 4, 457—466; cf. B., 1929, 965).—The X-ray diffraction patterns of fusain, graphitic anthracite, fibrous and non-fibrous peat, and elaterite have been examined. That of fusain shows a number of rings, much like durain (cf. *loc. cit.*), but with the interspaces fairly clear. There is also evidence of the fibre pattern in the diagram. Graphitic anthracite shows well-defined and fairly sharp halos for both the carbon and the ash, indicating that neither is in the colloidal state. In both fusain and anthracite the carbon seems to be in a free state. The prominent ash rings in both cases have been ascribed to silica and alumina. Fibrous peat gives a fibre pattern, whilst ordinary peat gives the diffuse and ill-defined diagram of a colloid. From the parallelism of the spacings in the peats and in the coals it is suggested that the difference between the two may be mainly one of structure rather than of composition. Elaterite gives a number of rings with spacings showing close identity with those of the higher members of the paraffin series, so it is concluded that elaterite is a mixture of the higher member of this series.

J. W. SMITH.

Flash point of Polish coal dust. W. SWIENTO-SLAWSKI and M. GROCHOWSKI (Przemysl Chem., 1930, 14, 246—249).—The flash point of durain and vitrain dust lies between 330° and 360° for grains of mean diam. 1.5–2 mm., and between 190° and 220° for grains of less than 1 mm. diam. The flash point of fusain dust is 190–270°, both for grains of more and of less than 1 mm. diam.

R. TRUSZKOWSKI.

Determination of soot. J. HIRSCH (Z. Hyg., 1929, 110, 399—412; Chem. Zentr., 1929, ii, 2800).—The soot is collected in a Dubsy combustion tube which is partly filled with alternate layers of copper oxide (as wire) and quartz splinters. Air is sucked through by an oil pump and measured. The soot is then heated in an oxygen stream and the carbon dioxide formed absorbed in 0.1N-barium hydroxide and determined by titration. The maximum error of the method is 4.85%. The small soot particles can be caught finally by means of a filter.

L. S. THEOBALD.

Humic acids. M. SAMEC [with B. PIRKMAIER] (Kolloid-Z., 1930, 51, 96—100).—The humic acid content of a number of coals has been determined by measurements of solubility in sodium hydroxide. Ammonium humate and hymatomelanate are retained by a collodion membrane and give a measurable osmotic pressure. Using different coals, the values 1445, 1235, and 1345 were obtained for the mol. wt. of ammonium humate and the values 855, 761, and 739 for ammonium hymatomelanate. The properties of these compounds are compared with those of ammonium lignate.

E. S. HEDGES.

Softening of ash of solid fuel. H. A. J. PIETERS (Chem. Weekblad, 1930, 27, 331—334).—A modified apparatus for determining the softening point of ash is described, and a large number of results are recorded. Three classes are distinguished, with softening temperatures respectively below 1250°, and above 1250°, the third class showing softening but not melting above 1250°. The relationship between softening temperature and chemical composition is discussed.

S. I. LEVY.

History and development of submerged combustion. C. F. HAMMOND (J. Inst. Fuel, 1930, 3, 303—320).—The methods of submerged combustion are described and details are given of the Hammond system and its industrial applications, e.g., evaporation of chemical solutions, heating of linseed oil, dyeing and washing processes, white-metal melting.

H. E. BLAYDEN.

Effect of addition of dolomite on carbonisation and combustion of bituminous coal. (Sulphur distribution.) I. TRIFONOV and E. RASCHEVA-TRIFONOVA (Brennstoff-Chem., 1930, 11, 185—188; cf. B., 1930, 592).—The addition of 10% of dolomite to coal has little effect on the yields of coke, tar, and gas obtained by carbonisation at 950°, and on the distribution of the sulphur between the coke and volatile products. The added dolomite, however, greatly increases the proportion of sulphur retained by the ash when the coke is burned, the effect indeed being greater than would be expected from the composition of the dolomite. This is attributed to the protective action of the calcium carbonate on the magnesium sulphate formed. In the combustion of coke produced at a normal rate of carbonisation, the addition of dolomite causes less sulphur to be retained in the ash than does that of calcium carbonate, whereas the reverse is true if a rapid rate of carbonisation is used, the proportion of sulphur retained depending on the relative rates of evolution of hydrogen sulphide and decomposition of the carbonates. Addition of dolomite produces a marked increase in the strength of the coke, an increase which, in general, cannot be brought about by the addition of an equivalent mixture of calcium and magnesium carbonates. By the direct combustion of a mixture of coal and dolomite almost the same proportion of the sulphur is retained in the ash as by carbonisation followed by combustion of the coke.

A. B. MANNING.

Electric welding in carbonising plants and gas-works. W. KÜRSCHNER (Brennstoff-Chem., 1929, 10, 468—469).—Electric welding is recommended for the

construction and repair of gas holders, boilers, pipes, etc. The process is rapid and repairs can often be done without interrupting the working of the plant. Electrically welded joints are less liable to leakage or to attack by corrosive fluids than riveted joints; they are, moreover, stronger and more economical in material.

A. B. MANNING.

Corrosion at riveted and welded joints in fuel-technology practice. O. GROSS (Brennstoff-Chem., 1930, 11, 133—134. Cf. Kürschner, preceding abstract).—A number of cases are quoted, e.g., sulphuric acid conduits and containers, pipes conveying steam containing ammonia and small quantities of sulphurous acid, copper centrifuges for draining acid ammonium sulphate, etc., in which much less corrosion occurs at riveted or flanged joints than at welded joints. A. B. MANNING.

Nomography in gas analysis. W. J. G. DAVEY (Gas World, 1930, 92, 694—695).—The application of nomographs to the determination of methane and hydrogen in coal gas when the percentages of the other constituents and the calorific value are known is described.

C. W. GIBBY.

Coal-tar oil for heating industrial furnaces. H. G. LUHN (Brennstoff-Chem., 1930, 11, 192—193).—The objections which have been raised against the use of coal-tar oils in oil-fired furnaces are attributed principally to unsuitable choice of plant or faulty operation. The oil should always be preheated, not only to prevent separation of naphthalene or anthracene, but also to lower the viscosity and improve the atomisation at the burner. Coal-tar oil is lower in price than gas oil, and, owing to its higher carbon and lower hydrogen content, its use diminishes the heat lost in the flue gases. By preheating both the oil and the air rapid and efficient combustion is attained with a minimum excess air.

A. B. MANNING.

Gum formation in cracked petrol. M. NAPHTALI (Chem.-Ztg., 1930, 54, 371).—The work of various investigators on the determination of gum, "preformed" and "potential," in cracked petrols and the comparative effect of inhibitors on its formation is summarised. Distillates cracked in the liquid phase contain much more resin than those cracked in the vapour phase.

C. IRWIN.

Refining of benzol. Action of chlorine on crude benzol. T. I. RABEK (Brennstoff-Chem., 1930, 11, 189—192).—Bromine value determinations show that the unsaturated constituents of crude benzol occur principally in the lowest- and highest-boiling fractions. The intermediate fractions contain relatively small amounts of impurities. The sulphuric acid reaction is given not only by the unsaturated hydrocarbons, but also by some sulphur compounds of unknown constitution, and is unsuitable for the evaluation of benzol as a motor fuel. When the crude benzol is treated with chlorine the unsaturated constituents are attacked first and then the sulphur compounds, whilst the benzene itself remains unchanged. It is possible by such treatment to free the benzol completely from thiophen and its homologues. From the amounts of chlorine directly added, substituted, and unabsorbed, respectively, the unsaturation of the crude benzol has been determined

and shown to correspond exactly with that deduced from the bromine values.

A. B. MANNING.

Effect of refining agents and fused caustic alkali [alkali hydroxide] on [pure organic] sulphur compounds in naphtha solution. M. A. YOUTZ and P. P. PERKINS (Ind. Eng. Chem., 1930, 22, 610—611).—Treatment of solutions of *n*-, *sec*-, and *iso*-butyl sulphides in naphtha with sulphuric acid (cf. B., 1928, 78) causes almost complete removal of the sulphides. When aluminium chloride is used as the refining agent it is found that the *n*-sulphide is removed much less completely. Treatment of solutions of 13 sulphides with a mixture of sodium and potassium hydroxides at 343° results in the removal of most of the sulphide with heptyl and allyl sulphides, and ethyl and *iso*amyl disulphides; the lower alkyl and phenyl sulphides are not appreciably affected.

H. BURTON.

Fuel-efficiency tests on batch oil stills. H. KREISINGER, W. R. ARGYLE, and W. E. RICE (U.S. Bur. Mines, Bull. 302, 1929, 94 pp.).—In order to promote the more efficient use of fuels in oil refining, 103 tests were made on batch oil stills fired with coal on chain-grate and underfeed stokers, with fuel oil, and with producer gas, various oil stocks being distilled. Comparable data are given for the three processes: (1) topping crude oil, (2) coking crude oil, and (3) re-running wax distillate. All distillations except the coking of the crude oil were made by the fire and steam method. Complete physical characteristics and chemical analyses of the fuels used are given, and the results are tabulated as weight of fuel fired per barrel of oil charged to each still, duration of the run, percentage of heat in the fuel lost in the flue gases, and the efficiency. The steam used in the stills was not considered in computing the heat accounts, and the amount of steam used was not measured except in a few tests on the re-running of the wax distillate for paraffin distillate. Samples of the flue gases were collected continuously and analysed every 30 min., their average carbon dioxide content for each test being recorded. Heat losses in unburnt carbonaceous matter in the ash and refuse, and losses due to radiation and convection are taken into account. The results obtained for each still or furnace are represented graphically. The first tests show how the efficiency might be improved by changes in the method of firing or operation, which are embodied in further tests. Data thus obtained point to further improvements that can be made by modifying the design or construction of the furnace. Such alterations are made step by step and their effect is determined by further tests. The development of the furnaces is traced by a series of drawings augmented by a discussion in the text. Temperature curves in connexion with heat distribution within the still during operation are recorded.

H. S. GARLICK.

Heavy distillates, fuel oils, asphalts, and residues. F. H. GARNER (J. Inst. Petroleum Tech., 1930, 16, 281—283).—A series of abstracts of recent publications on these subjects is presented.

H. S. GARLICK.

Special [petroleum] products. J. A. CARPENTER (J. Inst. Petroleum Tech., 1930, 16, 284—312).—A

review of recent literature and patents concerning paraffin wax: its crystallisation, structure, chemical constituents, physical properties, and behaviour of wax-oil mixtures; the use of petroleum products in insecticides and larvicides; naphthenic acids; oxidation and oxidised products of petroleum; asphalt, pitch, and bituminous emulsions. H. S. GARLICK.

Analysis and testing [of petroleum products]. C. I. KELLY (J. Inst. Petroleum Tech., 1930, 16, 325—333).—The new and modified methods of the Inst. Petroleum Tech., the B.E.S.A., the Bureau of Mines, and the A.S.T.M., for testing petroleum and its products, are reviewed. Methods of testing gasoline for knocking and gum content are also included.

H. S. GARLICK.

Synthetic fuels. A. W. NASH (J. Inst. Petroleum Tech., 1930, 16, 313—324).—The progress of research during the last 2 years concerning (a) catalytic berginisation of coal and heavy tarry materials, and (b) production of liquid hydrocarbons from gases, e.g., methane, ethane, propane, etc., and their polymerisation to heavier oils is reviewed, and extensive references are made to the patent literature covering these processes.

H. S. GARLICK.

Lubricants. R. W. L. CLARKE (J. Inst. Petroleum Tech., 1930, 16, 255—265).—Progress made in this subject in the latter part of 1927 and during 1928—9 is reviewed. The chief improvements appear to be in the manufacturing and refining branches of the mineral oil industry. The problems of the cause of oiliness and the correlation of laboratory with practical tests have received attention.

H. S. GARLICK.

Thermal decomposition of [lubricating] oils. J. DAMIAN and G. DIXMIER (Chim. et Ind., 1930, 23, 834—839).—The changes in viscosity, mol. wt., saponif. value, and iodine value of castor oil and two mineral lubricating oils, on being heated at 140° in contact with air, have been determined. The oils were heated in copper boats which were kept in oscillation; the bottom of each boat was so shaped that in flowing from one end to the other the oil passed over a raised central portion, and was thereby exposed to the air in the form of a thin film. The viscosity, measured at 35° and at 100°, and the mol. wt. of each oil increased with the time of heating, whilst the iodine value fell. The rise in mol. wt. corresponded with the formation of a dark-coloured sludge, which, in the case of the mineral oils, was insoluble in the original oil, but was partially soluble therein in the case of the castor oil. The changes were similar to those occurring in the engine, but took place much more rapidly in the laboratory test.

A. B. MANNING.

Graphite lubrication. Colloidal graphite. R. CORDEBAS (Chim. et Ind., 1930, 23, 1092—1098).—Commercial graphite from Madagascar contains about 15% of siliceous impurities which render it quite unsuitable for lubrication purposes. The refined product should be wholly crystalline, powdered to pass 200-mesh, and free from amorphous graphite. It is used in the dry state, particularly at high temperatures, or incorporated in oil etc. At low pressures or high speeds a colloidal solution of graphite in water is an effective

lubricant, but it is only at low speeds and high pressures that the benefit of the use of graphite is realised. The object of graphite lubrication is to obtain a layer of graphite adsorbed in the metallic surfaces 1 μ in thickness. This layer when formed will persist if oil alone is subsequently used. Colloidal graphite can be obtained by grinding, or by the action of potassium bichromate and sulphuric acid on graphite, but these methods are expensive. A stabiliser such as soap or gum arabic should be added to the colloidal oil solution. It is also possible to apply graphite by the use of a special antifriction metal containing it. Powdered graphite may be added to the amount of 1½% to oil and kept in suspension by rapid circulation until graphitisation has occurred. This treatment should be repeated every two months. Graphite may also be incorporated in grease. C. IRWIN.

Heavy oils and lubricants. W. LEE (J. Inst. Petroleum Tech., 1930, 16, 266—279).—A progress report covering the years 1928—1929 is presented, together with a review of the literature and patents dealing with the subject. The production of heavy lubricants by polymerisation and the recovery of used oils is referred to, and attempts which have been made to correlate laboratory tests with results obtained in practice are noted.

H. S. GARLICK.

Sulphur chloride from gas-purifier refuse. IPATIEV and VASILEVSKI.—See VII. **Hot-patching of retorts.** PARDOE.—See VIII.

PATENTS.

Coke ovens. DR. C. OTTO & Co., G.M.B.H. (B.P. 315,668, 15.7.29. Ger., 14.7.28).—The lower ends of the vertical flues of a horizontal coke oven are divided by a perforated partition into passages for gas and air. The partitions are built of removable superimposed bricks formed with slanting channels through them, and designed to ensure that each brick will be permeable to gas and air and thus bring about any desired degree of pre-combustion.

A. B. MANNING.

Apparatus for baking carbonaceous fuel briquettes. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 304,284, 14.1.29. U.S., 18.1.28).—A multiple-chamber baking oven comprises a battery of horizontal ovens vertically disposed one above the other, each provided with one or more fuel containers, removable from one end thereof, for charging and discharging purposes. Between the ovens are horizontal, transverse passageways through which the heating gases are passed from suitably arranged combustion chambers; the passageways are provided with baffles in order to give a turbulent flow to the gases. The temperature of the heating gases can be adjusted by returning a controlled amount of the flue gases to the combustion chambers. Means are provided for admitting air to the baking ovens and for leading the mixture of air and volatile products from the ovens to the combustion chambers. A condensing system is also provided for the recovery of condensable products evolved from the briquettes during the baking, the non-condensable products being led to the combustion chambers. The apparatus is adapted to carry out the

process in two stages as described in B.P. 320,814 (B., 1930, 7).

A. B. MANNING.

Destructive hydrogenation of carbonaceous bodies. HOLZVERKOHLUNGS-IND. A.-G., and J. VARGA (B.P. 313,505, 12.6.29. Ger., 12.6.28).—Coal, tars, oils, etc. are treated with an excess of hydrogen at a high temperature and under pressure in the presence of catalysts containing molybdenum and/or tungsten, and in the presence of hydrogen sulphide, or a material liberating hydrogen sulphide under the conditions of the reaction, *e.g.*, elemental sulphur. An auxiliary catalyst or activator may also be used, *e.g.*, boric acid. The presence of the hydrogen sulphide brings about a marked decrease in the tar acid content of the product and an increase in the quality and yield of light oils.

A. B. MANNING.

Manufacture of finely-divided active carbon. I. G. FARBENIND. A.-G. (B.P. 304,791, 25.1.29. Ger., 26.1.28).—Carbon monoxide is passed at atmospheric or higher pressures over a catalyst comprising a metal of group VIII, *e.g.*, iron, nickel, or cobalt, in a finely-divided form, in admixture with a difficultly reducible oxide, *e.g.*, magnesia or alumina. The reaction is carried out preferably at 300–350°. A hydrocarbon, *e.g.*, methane, may be mixed with the carbon monoxide. The catalyst is dissolved out from the finely-divided carbon formed and is recovered.

A. B. MANNING.

Treatment of carbon black and other fine powderous materials. J. E. POLLAK. From W. B. WIEGAND and L. J. VENUTO (B.P. 327,979, 14.1.29).—Finely-powdered materials are converted into readily frangible, non-dusting agglomerates by treatment with two non-miscible liquids, both non-solvents for the material, one of which has a greater wetting power for the solid than the other. The agglomerates so formed are separated by filtration or decantation, and are then dried. Suitable liquids for use with carbon black are water and petrol. The agglomerates are denser than the original carbon and the industrially essential physical properties, *e.g.*, easy and uniform dispersion in oils, rubber, etc., are unaffected.

A. B. MANNING.

Coolers or condensers for fuel gases. D. M. HENSHAW and W. C. HOLMES & Co., LTD. (B.P. 328,421, 12.4.29).—Below the condenser of a plant for the production of fuel gas are two chambers, one for accommodating mainly the condensed tar, and the other for cooling the aqueous distillate. A pipe from the condenser delivers the total condensate to the first chamber, whence the aqueous condensate passes through an overflow into the second chamber. The latter is provided with banks of tubes through which cold water is circulated. The condenser and the cooling chamber for the aqueous condensate are preferably separated from the tar-collecting chamber by heat-insulating partitions.

A. B. MANNING.

Production of water-gas or coal- and water-gas. BAMAG-MEGUIN A.-G., and O. HELLER (B.P. 304,139, 14.1.29. Ger., 12.1.28).—Finely-pulverised fuel and a mixture of gas and highly superheated steam are admitted to a reaction chamber, and the resulting gas is passed through a dry ash collector and a heat exchanger, after which part of the gas is passed through

a wet dust filter, which serves as a steam generator, back to the steam superheater and the reaction chamber, while the surplus gas is passed through a second dust filter to a holder. The superheater is heated intermittently by the combustion of gas therein. The process may be made continuous by duplicating the superheater and reaction chamber, and arranging for each set to be operated alternately as described above.

A. B. MANNING.

Gas purification. D. L. JACOBSON, ASSR. to KOPPERS Co. (U.S.P. 1,741,113, 24.12.29. Appl., 11.1.27).—Gases are freed from hydrogen sulphide by washing with an alkaline solution containing in suspension a compound of a metal, the sulphide of which is insoluble therein, and containing also a compound of manganese. *E.g.*, a liquid containing 1–3% of sodium carbonate, 2% of ferric oxide in suspension, and 0.5% of manganese chloride is suitable. The presence of the manganese diminishes the formation of sodium thiosulphate. The spent solution is revived by aeration, and free sulphur is thereby recovered.

A. B. MANNING.

Destructive hydrogenation of bars, oils, suspensions of carbonaceous materials in oils, etc. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,719, 21.2.29).—Heat exchangers used in connexion with high-pressure apparatus are built of concentric tubes, the outer one being thick enough to withstand the pressures, and the inner one thin-walled to allow of heat transference. The relatively cold material passes through the annular space to a reaction vessel in a countercurrent direction to hot material which is leaving the reaction vessel by means of the inner tube.

T. A. SMITH.

Hydrogenation and distillation of (A) hydrocarbon oils and other carbonaceous material, (B) petroleum oils. STANDARD OIL DEVELOPMENT Co., Assees. of E. M. CLARK (B.P. 304,796–7, 26.1.29. U.S., 26.1.28).—(A) Preheated crude petroleum or reduced crude is mixed with heavy residuum from the stills and passed into a hydrogenation zone where the oil is held in contact with hydrogen or hydrogen-containing gases under a pressure exceeding 50 atm. (preferably about 200 atm.) and at a temperature of 400–510°, in the presence of a catalyst, preferably finely-pulverised metal oxides, *e.g.*, 90% of chromium oxide and 10% of molybdenum oxide. Gases and vapours from the primary reactors are conducted into secondary reactors operating at 427–510° and above 25 atm. in the presence of the same catalyst, the requisite temperature being obtained by forcing in a supply of highly heated hydrogen or gas containing it. Vapour from the secondary reactors is cooled in preheating incoming crude oil and hydrogen in heat exchangers, and the liquid products are condensed in suitable condensers. The residual hydrogen is washed with alkali and water to remove hydrogen sulphide and with oil to remove hydrocarbons such as methane. The condensers may be of the simple type, or adapted to give any degree of reactification. The condensed hydrogenated oil is conveyed to a battery of stills, where it is separated into gasoline, naphtha, kerosene, gas-oil, and lubricating oil fractions. (B) The oil to be treated is preheated, and

a light fraction distilled from the oil in the first of a series of distillation zones, the residual oil being submitted to the above-mentioned process of destructive hydrogenation.

H. S. GARLICK.

Recovery of motor fuel from crude oil, shale, lignite, coal, and other organic material. E. and E. L. SCHULTZ (B.P. 328,295, 22.1.29).—The crude oil or other material is vaporised in a retort or still, provided with the usual pressure- and temperature-recording instruments, up to a temperature of 270°, and the vapours are led off from the top to a pot or converter. The converter is fitted with heating means and a vertical shaft carrying agitating gear. The whole of the metal portions of the converter are formed preferably of a nickel-zinc-lead alloy or of steel. In the converter the vapours are subjected to the action of lime, zinc chloride, or zinc oxide, with or without sodium or magnesium chloride. The process consists in starting at a low pressure to recover the light oil, the temperature in the converter being regulated relatively to that in the still or retort, and then gradually raising the dual temperatures to treat the heavier oil, and thus obtain a high percentage of motor fuel fraction, the pressure being regulated by an expansion or needle valve as the temperature rises. The vapours in the converter are subjected to the action of the chemicals and pass through the valve to a dephlegmator and condenser from which the condensate is recovered, and, if required, treated with chlorine or alkali and fractionated with fuller's earth. The residue is then returned to be re-treated in a fresh batch of material.

H. S. GARLICK.

Cracking of petroleum oils. STANDARD OIL DEVELOPMENT Co., Assees. of N. E. LOOMIS (B.P. 304,798, 26.1.29. U.S., 27.1.28).—Heavy oil is heated to cracking temperature in a tube furnace and passed to a vertical reaction chamber fitted with an expansion valve. The oil leaving this chamber is diluted with a little fresh cracking stock or other oil before passing through the expansion valve. The vapours pass through a tar trap to a dephlegmator column which is fitted at top and bottom with coils, in which the cracking stock is heated by the cooling vapours. Condensate from the tower is added to the cracking stock and vapours from the tower are passed to a condenser. Coke deposition at the expansion valve is thereby reduced.

T. A. SMITH.

Conversion of petroleum. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,741,509, 31.12.29. Appl., 21.10.21. Renewed 1.10.28).—Oil is continuously passed under superatmospheric pressure through a heating zone, where it is raised to cracking temperature, to an enlarged reaction zone where conversion occurs; the vapours are led to a dephlegmator and condenser from which the resulting condensate is collected. The reflux from the dephlegmator is separated into light and heavy fractions, the light fraction being returned directly to the heating zone for re-treatment, and the heavier passed to the reaction zone.

H. S. GARLICK.

Treatment of petroleum products. F. B. MUHLENBERG (U.S.P. 1,750,420, 11.3.30. Appl., 15.12.25).—Gasoline, kerosene, head oil, etc. containing sulphur

impurities are treated with an ammoniacal solution of copper hydroxide by spraying such a solution into the oil at atmospheric temperature, continuously or periodically removing the solution and impurities from the bottom of the oil and returning it to the sprayer, and repeating the operations until the oil is sufficiently purified.

H. S. GARLICK.

Apparatus for treating crude petroleum. G. A. STAFFORD and C. D. CORK (U.S.P. 1,749,235, 4.3.30. Appl., 16.2.25).—Crude oil is stored in closed storage tanks fitted with drain valves for the removal of water. Gas given off from the oil or displaced from the storage tank is taken to another storage tank through a condenser. Liquid products condensed from the gas may be removed, and oil is displaced from the storage tank by returning the gas which is forced through the oil. Any light constituents are absorbed and the gravity of the oil is lowered. In addition, gas may be taken from a gas-well and pumped through the oil with the object of lowering the gravity of the oil.

T. A. SMITH.

[Mineral] oil cracking process. J. B. JAQUA (U.S.P. 1,752,004, 25.3.30. Appl., 26.11.26).—Oil at a temperature slightly below cracking temperature is brought to reaction temperature by injecting small streams of a stable oil, heated externally to a temperature above that desired to effect an endothermic reaction of the new mineral oil, into an advancing stream of the oil. The additions are proportioned and so spaced, relatively to the dimensions and rate of advance of the treated oil, that a desired reacting temperature or temperatures is maintained for a predetermined period.

H. S. GARLICK.

Treatment of emulsions of mineral oils. W. B. LERCH, Assr. to J. S. DEWAR (U.S.P. 1,747,119, 11.2.30. Appl., 31.5.28).—An improved demulsifying agent for resolving stable hydrocarbon emulsions consists of sulphonated, highly unsaturated hydrocarbons of the polyolefine, cyclopentene, and cyclohexene series, obtained by the addition of sulphuric acid to materials such as Blau or Pintsch gas tars or other residual tars containing an appreciable alumina content in their ash.

H. S. GARLICK.

Cracking of [hydrocarbon] oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,741,510, 31.12.29. Appl., 26.6.26).—Oil is raised to cracking temperature while it is flowing through a heating coil, and the evolved vapours are subjected to reflux condensation. A number of progressively lighter fractions of the condensate are collected and the lightest fraction is returned to the inlet of the heating coil, while progressively heavier fractions are separately returned to spaced points in the length of the heating coil, thereby passing the heavier fractions through a smaller portion of the heating coil than the lighter fractions.

H. S. GARLICK.

Hydrocarbon oil conversion. C. J. PRATT, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,741,535, 31.12.29. Appl., 8.7.26).—Charging stock is subjected to non-cracking conditions of temperature and the vapours are separated into lighter and heavier portions. The separated fractions are then simultaneously subjected to independently controlled temperature and pressure conditions so that the lighter fractions are

treated at higher temperatures and pressures than the relatively heavier ones. The vapours from the fractions are dephlegmated in the zone where fractionation of the charging stock takes place.

H. S. GARLICK.

Cracking and distillation of heavy hydrocarbon oils. MIDLAND COAL PRODUCTS, LTD., and J. E. TRUZZELL (B.P. 328,639, 19.12.28).—Heavy hydrocarbon oil is mixed with up to 40% by wt. of bituminous coal slack and, while being agitated, is submitted to a distilling and cracking process under a pressure not above 150 lb./in.² and a temperature not above 200°, this being sufficient to volatilise light hydrocarbons without distilling the coal. The residue is submitted to low-temperature carbonisation, giving a product suitable for fuel in Diesel engines and a semi-coke.

H. S. GARLICK.

Distillation of heavy liquid hydrocarbons. J. H. DE LIGNAC (B.P. 328,875, 22.7.29).—Heavy oil, mixed with water, is distilled in a vessel containing in its lower portion a catalyst supported on grids. The catalyst consists of separate layers of nickel (2 pts.), copper (1 pt.), and iron (1 pt.). The vapours produced pass through similar beds of catalyst supported in the upper part of the chamber and through a further mass of catalyst in the vapour pipe before being condensed.

T. A. SMITH.

Decolorising and colour-stabilising hydrocarbon oil. V. VORHEES, Assr. to STANDARD OIL Co. (U.S.P. 1,747,806, 18.2.30. Appl., 31.10.27).—Pressure distillate, which has been treated in the vapour phase with clay, is treated with 1% by vol. of sulphuric acid (10–70% H₂SO₄). The product does not develop colour on storing.

T. A. SMITH.

Production of stable hydrocarbon oils. B. T. BROOKS, Assr. to GRAY PROCESS CORP. (U.S.P. 1,748,507, 25.2.30. Appl., 10.10.25).—0.5–1.5 G. of ammonia are added per gal. of gasoline in order to stabilise the colour and prevent sedimentation.

H. S. GARLICK.

Manufacture of hydrocarbons of low b.p. from those of higher b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,649, 1.2.29).—Tars, mineral oils, etc. are cracked in the presence of solid catalysts which maintain their shape unchanged under the conditions of working and which are dispersed in the liquid materials to be treated. Suitable catalysts are, e.g., bauxite, silica gel, etc., and those containing molybdenum or chromium. A part of the liquid material, together with the catalyst dispersed therein, is drawn off, without interruption of the cracking process, from the reaction chamber and, if desired, returned after partial or complete separation from the catalyst, which is regenerated outside the reaction chamber and then reintroduced either alone or together with fresh initial material.

H. S. GARLICK.

Conversion of hydrocarbons of high b.p. into those of lower b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,959, 22.2.29).—Tars, mineral oils, etc. are subjected to consecutive restricted crackings in such manner that each time fractions having a comparatively narrow b.p. range not exceeding 200° are treated, and at least one stage of the process is carried

out in the presence of catalysts immune from sulphur-poisoning and under pressure of at least 50 atm.

H. S. GARLICK.

Manufacture of low-boiling hydrocarbon products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,618, 13.10.28).—Carbonaceous materials which are liquid at ordinary temperatures (e.g., producer tar, tar from mineral coal) or middle oils obtained therefrom in the liquid state, are treated with a stream of hydrogen or hydrogen-containing gas at an elevated pressure and temperature, and in the presence of a catalyst (containing molybdenum or chromium) immune to sulphur-poisoning, under such conditions that products richer in hydrogen than the initial materials are obtained; these are then subjected to a high-temperature cracking process in the presence of gases having a reducing action, in order to obtain products of low b.p. [Stat. ref.]

H. S. GARLICK.

Manufacture of hydrocarbons and oxygen derivatives of hydrocarbons, in particular those of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,586, 26.11.28).—Carbonaceous materials are treated with hydrogen at elevated temperatures and at pressures above 20 atm. in the presence of catalysts which are immune to sulphur-poisoning, and before or during the reaction a medium which is liquid under the conditions of working and which does not take part in the reaction, e.g., high-boiling organic solvents, in particular, oils of aromatic character, is added for the purpose of preventing the deposition of high-molecular substances on the catalyst. The medium is continuously removed from the reaction chamber, the lighter-boiling fractions are removed, and the residue is returned to the process.

H. S. GARLICK.

Separation of hydrocarbons. W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,746,197, 4.2.30. Appl., 16.8.22).—Mixtures of gasoline and kerosene are fractionated under a pressure of 15 lb., a temperature difference of approx. 80° being maintained between the top and bottom of the fractionating column used.

T. A. SMITH.

[Production of gasoline from heavier hydrocarbons by] pressure distillation. R. E. HUMPHREYS, Assr. to STANDARD OIL Co. (U.S.P. 1,745,931, 4.2.30. Appl., 9.4.21).—Gasoline is produced by distilling naphtha residues at about 340° and under 325 lb./in.²

T. A. SMITH.

Production of gasoline from cracked distillate. G. P. KOCH, Assr. to SHELL OIL Co. (U.S.P. 1,742,263, 7.1.30. Appl., 29.3.26).—Cracked distillate is treated with concentrated nitric acid in amount substantially equal in wt. to the sulphur content of the oil (as determined by previous analysis), without materially affecting the unsaturated hydrocarbons; it is then washed with water and further washed with caustic soda and sulphuric acid in amount considerably less than if the nitric acid had not been used. After a final water wash the gasoline and unsaturated hydrocarbons are distilled off from the distillate.

H. S. GARLICK.

Recovery of gasoline from natural gas. F. E. HOSMER (B.P. 311,693, 14.1.29. U.S., 14.5.28).—"Wet"

natural or casinghead gas enters the bottom of a tower and flows upwardly past a number of bubble-plates before coming in contact with a refrigerating coil. The liquefied constituents fall back through the bubble-plates and are partially evaporated by the incoming gas, which is thereby cooled and saturated with the previously condensed constituents. Continuing upwardly, the now lean gas leaves the system after passing through a second series of bubble-plates over which a stream of brine is pumped from a reservoir at the base of the tower. The brine stream is cooled by the lean cold gas and picks up the condensed constituents of the gas, washing them downwardly past the lower bubble-plates, where some of the gaseous constituents are evaporated, into the reservoir at the base of the tower, which is provided with a trapped line through which the separated and "weathered" gasoline is removed. H. S. GARLICK.

Apparatus for salvaging used cleaners' gasoline. W. S. BAYLIS, Assr. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,751,613, 25.3.30. Appl., 21.12.25).—The used solvent passes through a trap to remove any large particles and is pumped through a primary filter consisting of a shell provided with layers of filtering material separated by perforated plates. From this filter the solvent passes to a washer where it is agitated with an absorptive material, *e.g.*, fuller's earth. After settling, the solvent is drawn off to a secondary filter capable of removing very fine particles and is then passed to storage. Arrangements for cleaning the primary filter by backwash are described. H. S. GARLICK.

Manufacture of lubricating oils. G. H. TABER, JUN., Assr. to SINCLAIR REFINING Co. (U.S.P. 1,746,916, 11.2.30. Appl., 15.12.28).—Vapours from a lubricating oil still are passed to a tower down which alkali solution is sprayed. Vapours which are not condensed in the tower are passed through a cooling coil. Condensate and alkali from the treating tower are received in a tank where they may be separated, or recirculated if desired. The apparatus is arranged to work under ordinary or reduced pressure. T. A. SMITH.

Recovery of entrained [petroleum lubricating] oils from filter cakes. G. F. OLSEN (U.S.P. 1,745,837, 4.2.30. Appl., 22.1.27).—Entrained oil is recovered from a hot filter-press cake which is simultaneously cooled by displacing and dissolving the entrained oil with a petroleum solvent intermediate in gravity and viscosity between kerosene and lubricating oil of 75 sec. viscosity (Saybolt). The solution is reduced by partial distillation to the consistency of a lubricating oil, the recovered distillate being utilised in a succeeding operation. H. S. GARLICK.

Purification of used lubricating oils. J. G. FORD, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,746,641, 11.2.30. Appl., 6.8.27).—Used transformer etc. oils are treated with 25–50% by vol. of liquid sulphur dioxide. After separation the residual sulphur dioxide is removed by treatment with alkali or a stream of air or nitrogen. It is then treated with 5% by wt. of infusorial earth at 100° and filtered. T. A. SMITH.

Refining of used [lubricating] oil. J. C. PATRICK (U.S.P. 1,750,350, 11.3.30. Appl., 28.10.27).—Free water is allowed to settle from used crank-case oil which is then

heated to 49° and agitated at that temperature with 2% by vol. of sulphuric acid (*d* 1.83). After settling, the oil is removed from the resultant sludge and heated under vacuum to 232° to remove diluents. The hot oil is treated with 15–18% of fuller's earth to which has been added an anhydrous oxide in order to clarify, decolorise, and neutralise the oil, and the resultant purified oil is drawn off. H. S. GARLICK.

Reclaiming used lubricating oils. H. W. CHETWIN and W. MANN (B.P. 328,558, 25.1.29).—Used lubricating oils (flash point under 177°), if emulsified, are brought to the b.p. with about 20% of a 10% alum solution before proceeding with the same treatment as with non-emulsified oil, which consists in heating it for 4–6 hrs. at 82–88°, allowing to settle for 10 hrs. without further heating, centrifuging or filtering the oil, passing it through a continuously working steam-heated still wherein light fractions are volatilised, clarifying by means of an acid, washing with alkali, and finally filtering. The still consists of one or more pipes each having downwardly extending baffle plates subdividing the interior, with an uptake pipe in the roof communicating with each subdivision and uniting in a common condenser pipe leading to one or more water-cooled receivers. H. S. GARLICK.

Apparatus for treating used lubricating oil. R. M. DILWORTH and F. G. NIECE, Assrs. to ELECTRO-MOTIVE Co. (U.S.P. 1,749,571, 4.3.30. Appl., 4.11.27).—A vertical still is connected to a heating coil, a charging tank, and a condenser. The arrangement is such that oil pumped from the charging tank through the coil to the still returns to the charging tank when the oil in the still reaches a determined level. Steam is passed into the oil stream before it enters the heating coil. Vapours from the still are taken to a condenser. The oil is circulated until all the volatile impurities have been removed. A small draw-off line is so arranged that any water condensed in the still is drawn into the oil stream to the heating coil and not into the storage tank. After sufficient treatment the oil is filtered before re-use. T. A. SMITH.

Reclaiming [mineral insulating] oil. D. C. COX, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,752,238, 25.3.30. Appl., 18.3.25).—Sludged transformer oil is passed through a series of tanks, the oil flowing from the bottom of one into the top of the next. Each tank contains a charge of fuller's earth supported on a perforated plate with a suitable filter-cloth interposed between the earth and the plate, and a second fabric is placed on top of the earth. Means are provided for separating any one or more tanks from the series for cleaning and recharging without interfering with the rest. To the cleaned oil is added an approximately equal volume of new oil before returning it for use in a transformer. H. S. GARLICK.

Apparatus for refining [mineral] oil. G. H. TABER, JUN., Assr. to SINCLAIR REFINING Co. (U.S.P. 1,746,915, 11.2.30. Appl., 15.12.28).—Lubricating oils are produced in a plant consisting of a series of stills each provided with its own fractionating tower, treating tower connected to the fractionating tower, condenser, and cooler for receiving the condensate from the fractionating tower. Caustic alkali solution is supplied to at least one of the

treating towers of the series, the condensate from the associated cooler being delivered to a succeeding treating tower of the series.

H. S. GARLICK.

Purification of hydrocarbon distillates. R. C. OSTERSTROM (B.P. 328,309, 9.10.28).—Cracked hydrocarbon oils containing unsaturated compounds are treated with an absorbent capable of polymerising gum-forming and colour-imparting constituents normally present in such oils by introducing the absorbent into a stream of oil or oil vapours which carry the absorbent with them in their subsequent continuous flow before or after vaporising the oils. The effective treatment takes place while the oil is in the vapour phase, the heat generated by the reaction causing an increase of pressure and temperature sufficient to cause polymerisation, but insufficient to cause cracking. The mixture is discharged into an enlarged separating or vaporising and fractionating zone the upper part of which is supplied with means for applying heat in order to facilitate the separation of the light oils from the heavier polymerised oils and absorbent brought about by the release of pressure.

H. S. GARLICK.

Refining of benzol. SOC. DU GAZ DE PARIS (B.P. 307,935 and Addn. B.P. 330,045, [A] 15.3.29, [B] 2.4.29. Fr., [A] 17.3.28, [B] 29.11.28).—(A) The vapours of crude benzol, b.p. 75—180°, are passed through porous masses, impregnated with sulphuric acid (*d* 1.83 or less) and used in the proportion of 1—2% of the benzol treated, placed in a suitable heat-insulated apparatus capable of being heated, if necessary. The refined benzol is washed free from acid, and the porous mass as it becomes inactive is replaced by fresh acidified material. (B) The fraction of crude benzol distilling over the range 65—120° is caused to flow through a mass of refractory material maintained at 90—95° countercurrent to a stream of dilute sulphuric acid, and is washed with water and alkali and finally rectified in a reflux condenser.

H. S. GARLICK.

Vacuum distillation and rectification of paraffin distillate. N. E. LOOMIS and W. K. LEWIS, ASSRS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,746,198, 4.2.30. Appl., 2.6.24).—Paraffin-base crude oil is heated to not above 340° under an absolute pressure not exceeding 30 mm. Hg, and the vapours are led to a group of fractionating zones where liquid and vapour phases exist in partial equilibrium, the vapours being subsequently led away and those of the paraffin distillate condensed, cooled to a point at which the paraffin precipitates in crystalline form, and filtered to produce a residue of paraffin wax and a filtrate of petroleum lubricating oil.

H. S. GARLICK.

Purification of wax distillates. E. PETTY, ASSR. to DE LAVAL SEPARATOR Co. (U.S.P. 1,750,646, 18.3.30. Appl., 24.8.21).—Purified paraffin and wax-free oils of different gravities are produced by separately distilling from a petroleum crude oil a number of wax distillates and subjecting each fraction separately to the following treatment: dilution with a light-petroleum distillate, agitation of the diluted stock with acid, removal of the acid sludge, agitation of the stock with alkali, and centrifuging the resultant emulsion to separate the soap and alkaline solution. The diluted stock is then chilled

sufficiently to precipitate the wax and centrifuged, thereby separating the wax from the oil. The wax-free oil is then further treated with a clarifying and decolorising agent, the last traces of which are finally removed by centrifuging, and the diluent is eliminated from the oil by distillation.

H. S. GARLICK.

Manufacture of motor fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,587, 22.12.28).—The stability to compression of motor fuels consisting substantially of saturated hydrocarbons and containing, if desired, iron carbonyl, lead tetraethyl, alcohols, amines, or benzol, is increased by the addition of up to 30% of liquids consisting only of polymerides of the lower olefines. Examples are diisobutylene, alone or with diisoamylene; mixed polymerides of isobutylene and trimethylamine.

H. S. GARLICK.

Motor fuels and their manufacture. CARBIDE & CARBON CHEM. CORP. (B.P. 303,505, 13.12.28. U.S., 6.1.28).—Alkylbenzenes, the alkyl groups of which contain two or more carbon atoms, are added to motor fuels in order to produce an antidetonating fuel for use in high-compression engines.

A. B. MANNING.

Purification [desulphurisation] of [combustible] gases. E. A. PRUDHOMME, ASSR. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,760,522, 27.5.30. Appl., 7.4.26. Fr., 4.3.26).—See B.P. 267,138; B., 1928, 220.

Treatment of [asphalt] dispersions. F. R. MOSER (U.S.P. 1,759,913, 27.5.30. Appl., 27.7.25. Holl., 2.1.25).—See B.P. 245,418; B., 1927, 101.

Treatment of hydrocarbon oils. J. B. WEAVER, ASSR. to GYRO PROCESS Co. (Re-issue 17,681, 27.5.30. of U.S.P. 1,601,786, 5.10.26).—See B., 1927, 6.

Protecting the interiors of oil-cracking retorts. A. C. HOLZAPFEL (U.S.P. 1,761,343, 3.6.30. Appl., 30.3.27).—See B.P. 291,585; B., 1928, 560.

Burners for gaseous, liquid, or powdered solid fuel. P. J. J. ANDRIEUX, ASSEC. of SOC. GÉN. DES CHAUX ET CEMENTS (B.P. 310,881, 29.4.29. Fr., 2.5.28).

Miners' safety lamps. G. MÜLLER (B.P. 330,469, 5.9.29. Ger., 6.7.29. Addn. to B.P. 318,481).

Grates more especially for gas producers. A. L. GALUSHA (B.P. 315,887, 20.7.29. U.S., 20.7.28).

[Valve for] retort-house gas governors. METERS, LTD., and W. T. GLOVER (B.P. 330,189, 23.8.29).

Retorts for continuous working (B.P. 317,066). **Bubble tower** (U.S.P. 1,748,855). **Pulveriser** (U.S.P. 1,747,922). **Separation of solids by flotation** (B.P. 329,013). **Fractionation** (U.S.P. 1,748,411, 1,748,508, 1,748,595, and 1,748,704). **Removing oil from gases** (B.P. 306,899).—See I. **Purified ethylene** (U.S.P. 1,741,559). **Cuprene from acetylene** (B.P. 303,797). **Humous colloids** (B.P. 312,233). **Dispersing etc. agents** (B.P. 300,574).—See III. **Neutral ammonium sulphate** (B.P. 310,536). **Thiocyanates from gas liquors etc.** (U.S.P. 1,751,274). **Phosphorus-free carbon monoxide** (G.P. 462,521).—See VII. **Refractories** (B.P. 304,729).—See VIII. **Construction of roads etc.** (B.P. 329,569).—See IX. **Purification of hot gases** (B.P. 329,962).—See XI. **Lyophile**

products (B.P. 329,305). **Emulsions** (B.P. 329,266).—See XII. **Lacquers etc.** (B.P. 329,694).—See XIII. **Paraffin emulsions** (G.P. 446,598).—See XX.

III.—ORGANIC INTERMEDIATES.

Petroleum products. CARPENTER.—See II.

PATENTS.

Purification of ethylene [for anaesthesia]. H. H. DAWSON, Assr. to OHIO CHEM. MANUF. CO. (U.S.P. 1,741,559, 31.12.29. Appl., 7.1.25).—Relatively warm ethylene is expanded from a pressure of about 1500 lb./in.², whereby the resulting cold causes condensation of moisture and other impurities, which are removed; the cold gas is then passed in heat-exchange relation with warmer compressed ethylene, treated with sulphuric acid of sufficient concentration to remove olefinic impurities without reaction with the ethylene itself, neutralised (preferably with caustic potash solution), compressed, and dried. The product is again treated with sulphuric acid and neutralised, and the gas finally passed in heat-exchange relation with cold ethylene and collected at a pressure less than that from which it was initially expanded.

H. S. GARLICK.

Manufacture of acetic anhydride. O. Y. IMRAY. From A.-G. F. STICKSTOFFDÜNGER (B.P. 328,572, 29.1.29).—Concentrated acetic acid is treated with carbonyl chloride in presence of about 10% of aluminium chloride, magnesium acetate, or an oxide, chloride, or acetate of a metal of group II or III.

C. HOLLINS.

Preparation of highly adsorbent cuprene from acetylene. N.V. "ELECTRO" ZUUR-EN WATERSTOFF-FABR. (B.P. 303,797, 8.1.29. Holl., 9.1.28).—Acetylene is passed at 200—400° over copper, nickel, iron, or oxides of these, to which less than 1% of magnesium powder has been added.

C. HOLLINS.

Manufacture of 1:3-[γ]-butyleneglycol. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,083, 9.3.29).—Aqueous aldol is hydrogenated in presence of a copper catalyst, e.g., reduced copper on silicic acid or on vitreous alumina, reduced copper silicate, copper and nickel on silica gel, etc., at 40—90° and 50—200 atm.

C. HOLLINS.

Manufacture of aliphatic or cyclic primary amines. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,381, 11.3.29).—Unsaturated aldehydes or ketones are passed in vapour phase with ammonia and hydrogen over catalysts as in B.P. 265,960 (B., 1928, 360). Saturated amines are produced; e.g., crotonaldehyde gives *n*-butylamine; acetaldehyde, *n*-propylamine; and cinnamaldehyde, γ -phenyl-*n*-propylamine (b.p. 102—104°/118 mm.).

C. HOLLINS.

Production of humous colloids. R. EBERHARD (B.P. 312,233, 16.5.29. Ger., 22.5.28).—Humous substances (powdered peat) are boiled with aqueous solutions of neutral or weakly basic organic compounds containing nitrogen or nitrogen and sulphur, other than aliphatic amides; e.g., hexamethylenetetramine, carbamide, formanilide, formaldoxime, acetaldehyde-ammonia, piperazine, etc.

C. HOLLINS.

Manufacture of esters of polymerised carbohydrates. I. G. FARBERIND. A.-G. (B.P. 305,661, 29.1.29.

Ger., 10.2.28).—A polymerised carbohydrate (cellulose, starch, agar-agar) is allowed to swell in caustic alkali solution of at least 30% concentration, and then treated preferably in suspension in a solvent (benzene, chlorobenzene, etc.) with a halide of a higher fatty acid or cyclic carboxylic acid (lauric acid, coconut oil acids, naphthenic acid, phenylacetic acid), without any cooling; if desired, heat may be applied. The resulting esters are soluble in organic solvents. Mixed esters may be prepared.

C. HOLLINS.

Catalytic molecular association [esterification] of organic compounds. SELDEN CO., Assees. of A. O. JAEGER (B.P. 308,582, 26.2.29. U.S., 23.3.28).—Base-exchange substances (artificial zeolites) containing esterification catalysts, such as zirconia, titania, alumina, or thoria, are used to catalyse the production of esters from vapours of alcohols and organic acids at 280—350°.

C. HOLLINS.

Manufacture of dispersing, emulsifying, and stabilising agents, and of dispersions and emulsions. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 300,574, 11.10.28. Holl., 15.11.27).—The aromatic constituents extracted from mineral oil by liquid sulphur dioxide, furfuraldehyde, aniline, methyl sulphate, or similar solvents are sulphonated to give products similar to those of B.P. 291,393 (B., 1929, 805).

C. HOLLINS.

Manufacture of wetting, cleansing, dispersing agents, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,675, 31.1.29).—Salts, amides, ester-amides, or esters of high-molecular organic acids, which are wetting agents etc. are improved by incorporation with wetting agents of the sulphonic acid type. Examples are: β -diethylaminoethylamide of oleic acid and sodium propylnaphthalenesulphonate; stearamide and sulphonated oleic acid; oleic β -hydroxyethylamide, or anilide, or cyclohexylamide and diisopropylnaphthalenesulphonic acid.

C. HOLLINS.

Sulphonation of aromatic hydroxy-derivatives. MAJOR & CO., LTD., and H. H. HINCHCLIFFE (B.P. 328,220, 19.1.29).—Phenol, *o*- and *p*-cresols, α - and β -naphthols are sulphonated in the form of their arylsulphonates with concentrated sulphuric acid at 40—70° or oleum at 15—20°. On diluting the reaction mixture the arylsulphonyl group is removed and the phenol-sulphonic acid is obtained in solution. The process is ineffective with nitrophenols.

C. HOLLINS.

Manufacture of *m*-2-xylidine. IMPERIAL CHEM. INDUSTRIES, LTD., and L. J. ALLCHIN (B.P. 328,418, 11.4.29).—After removal of the bulk of *m*- and *p*-xylidines by known methods, the crude oil is dissolved in hydrochloric acid and zinc chloride is added to the hot solution. The double salt of *m*-2-xylidine separates on cooling.

C. HOLLINS.

Manufacture of aromatic amines from mixtures thereof. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 328,210, 18.1.29).—Dry hydrogen chloride is led into the mixture of aromatic amines, preferably in an organic solvent, and successive fractions of hydrochloride are removed. Crude xylidine in benzene gives a 90% recovery of *m*-xylidine hydrochloride, and the *p*- and *o*-salts separate in that order by application of more

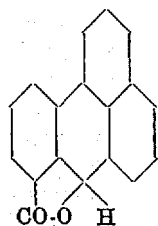
hydrogen chloride. The method is also used for the separation of *m*-4- from *m*-2-xylydine, *o*-4- from *o*-3-xylydine, *p*- from *o*-toluidine, 5- from 8-chloro- α -naphthylamine, aniline from toluidines, etc. C. HOLLINS.

Manufacture of hydroxysulphamic acids and salts thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,032, 13.1.28).—An aminonaphthol, aminoarylpirazolone, acetoacetic or 2 : 3-hydroxynaphthoic aminoarylamide is treated with chlorosulphonic acid or ester, sulphur trioxide in tetrachloroethane, or pyridinesulphuric anhydride, preferably in presence of pyridine or other acid-binding agent. The *N*-sulphonic acids of 2 : 7-aminonaphthol, 1-*m*-aminophenyl-3-methylpirazolone, acetoacetic *p*-aminoanilide, and 2 : 3-hydroxynaphthoic *p*-aminoanilide are described. C. HOLLINS.

Production of nitrosodiaz solutions. I. G. FARBENIND. A.-G. (B.P. 307,890, 15.3.29. Ger., 15.3.28).—A solid, stabilised diazo compound made from an aminodiphenylamine is mixed with sodium nitrite and, if necessary, an acid or acid salt. On dissolution in water the nitrosodiazodiphenylamine is formed at once. Examples are diphenylamine-*p*-diazonium hydrogen sulphate or zinc chloride double salt; 4'-methoxydiphenylamine-4-diazonium hydrogen sulphate or benzene-sulphonate. C. HOLLINS.

Preparation of *N*-substituted cyanoformarylamides. DEUTS. GOLD- U. SILBER-SCHIEDEANSTALT, VORM. ROESSLER (B.P. 306,450, 5.2.29. Ger., 20.2.28).—Hydrogen cyanide is added to a pyridine solution of a "urea chloride," $\text{ArNR} \cdot \text{COCl}$, at 50–80°, to give cyanoformarylamides ("urea cyanides"). Examples are products from hydrogen cyanide and the carbamyl chlorides prepared by the phosgenation of ethylaniline (m.p. 51°, b.p. 156–158°/16 mm.), methylaniline (m.p. 64–65°, b.p. 154–156°), phenylglycine nitrile (m.p. 143°), *p*-bromomethylaniline (m.p. 95°), tetrahydroquinoline (m.p. 74°), diphenylamine (m.p. 132°), ethyl- α -naphthylamine (m.p. 64°), ethyl- β -naphthylamine (m.p. 104°), and tetrahydro- β -naphthaquinoline (m.p. 134°). C. HOLLINS.

Manufacture of products of the anthracene series [intermediates and vat dyes]. I. G. FARBENIND. A.-G. (B.P. 303,375, 31.12.28. Ger., 30.12.27).—*o*- α -Naphthoylbenzoic acid is converted by sodium aluminium chloride at 100° into a compound (probably having annexed formula), m.p. 177°, which yields a violet vat dye when fused with alkali at 230–260°. C. HOLLINS.



Manufacture of derivatives of the anthracene series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,128, 26.9. and 1.10.28. Cf. B.P. 324,661; B., 1930, 452).— α -Naphthaquinone derivatives hydrogenated in the benzene nucleus are condensed with α -butadiene or its homologues to give hydrogenated anthracenes, which may be converted by the method of B.P. 320,375 (B., 1930, 233) into anthraquinones. The hydrogenated naphthaquinone from benzoquinone and α -butadiene is heated with α -butadiene or its β - γ -dimethyl derivative at 60–100° under pressure;

the conversion of the product from α - γ -butadiene into 9 : 10-dihydroxy-1 : 4 : 5 : 8-tetrahydroanthracene is described. C. HOLLINS.

Manufacture of leuco-indigo. IMPERIAL CHEM. INDUSTRIES, LTD., and K. H. SAUNDERS (B.P. 328,104, 8.4.29).—An alkaline indigo paste is passed continuously with hydrogen under pressure over a nickel catalyst on a rigid support at 120–140°.

C. HOLLINS.

Production of primary [aliphatic and cyclic] amines. W. REPPE, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,762,742, 10.6.30. Appl., 11.2.27. Ger., 9.2.26).—See B.P. 265,960; B., 1928, 360.

Introduction of an aldehydic group into heterocyclic nitrogen compounds. G. KALISCHER, H. SCHEYER, and K. KELLER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,763,557, 10.6.26. Appl., 30.1.28. Ger., 2.2.27).—See B.P. 311,208; B., 1929, 747.

Manufacture of condensation products from hydroxybenzenes and hydroaromatic ring-ketones. E. KORTEN, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,760,758, 27.5.30. Appl., 5.1.28. Ger., 20.1.27).—See B.P. 310,825; B., 1929, 747.

Preparation of trihalogenobenzene sulphochloride. R. HERZ, E. RUNNE, and E. ALBRECHT, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,763,556, 10.6.30. Appl., 15.3.28. Ger., 18.3.27).—See B.P. 287,178; B., 1929, 235.

Preparation of chlorine-substitution products of 4-nitro-1 : 3-dimethylbenzene [chlorinated 4-nitro-*m*-xylene]. E. HOFFA and E. THOMA, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,762,018, 3.6.30. Appl., 21.9.27. Ger., 11.10.26).—See B.P. 298,761; B., 1928, 225.

Preparation of condensation products of the naphthastyryl series. G. KRÄNZLEIN, M. HEYSE, and P. OCHWAT, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,762,021, 3.6.30. Appl., 21.11.27. Ger., 30.11.26).—See B.P. 289,692; B., 1928, 475.

Preparation of arylides of 2-hydroxy-3-naphthoic acid. E. B. HIGGINS, ASSR. to BRIT. SYNTHETICS, LTD. (U.S.P. 1,762,474, 10.6.30. Appl., 26.11.26. U.K., 28.12.25).—See B.P. 262,958; B., 1927, 102.

Separation of hydrocarbons (U.S.P. 1,746,197). **Refining of benzene** (B.P. 307,935 and 330,045).—See II. **Table salt** (B.P. 312,088).—See VII. **Emulsion** (B.P. 329,266).—See XII.

IV.—DYESTUFFS.

PATENTS.

Preparations useful for production of dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 328,383, 12.3.29).—Dyeing preparations of the Rapid Fast series containing nitrosamine alkali salts and diacetoacetic tolidide etc. are rendered more stable to long storage by addition of feebly acid substances (sodium hydrogen carbonate) and a water-binding salt (sodium acetate, anhydrous sodium sulphate).

C. HOLLINS.

Manufacture of vat dye preparations. J. R. GEIGY A.-G. (B.P. 308,730, 26.3.29. Ger., 27.3.28).—

In solid preparations containing vat dye, hyposulphite, and alkali, the alkali is added in the form of its compound with a polyhydric alcohol, or an aliphatic or aromatic hydroxy-acid, *e.g.*, the sodium compound of glycol, of sodium glycollate, or of sodium salicylate. Alkali carbohydrate compounds are not included.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,967, 12.1.29).—The acridone from 2:4-dibromo-1-aminoanthraquinone and anthranilic acid is halogenated in the benzene nucleus by treatment with halogenating agents in the usual way; or 2:4-dibromo-1-aminoanthraquinone is condensed with 3:5-dichloro-anthranilic acid and the product cyclised. Blue-green vat dyes of good tinctorial power are obtained.

C. HOLLINS.

Manufacture of vat dyes of the indigo series. I. G. FARBENIND. A.-G. (B.P. 305,592, 7.2.29. Ger., 7.2.28).—5:5'-Dihalogeno-4:4':7:7'-tetramethyl-indigos, obtained from *p*-3-xylydine (with halogenation of the tetramethylindigo) or its 6-halogeno-derivatives containing hydrogen or a carboxyl group in position 2, give pure blue shades fast to washing, boiling, and chlorine.

C. HOLLINS.

Manufacture of polymethine dyes. R. KUHN and A. WINTERSTEIN (B.P. 328,357, 13.2.29).—Formic acid or a formate is condensed with a cyclic ammonium organic salt derived from a heterocyclic compound containing a reactive methyl group, or with the corresponding methylene (ψ -) base. Examples are: formic acid with β -naphthaquinaldine methiodide, 2:3:3-trimethylindoline methiodide, 2-methylbenzthiazoline methiodide, etc., or the corresponding ψ -bases. The method is applicable where other processes fail.

C. HOLLINS.

Preparation of 2:3-benzocarbazole-1:4-quinones [vat dyes of the α -naphthaquinone series]. H. T. BUCHERER, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,763,216, 10.6.30. Appl., 26.6.28. Ger., 3.6.26).—See B.P. 317,928; B., 1929, 890.

Indigoid vat dyes. K. THIESS, T. MEISSNER, and C. J. MÜLLER, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,760,797, 27.5.30. Appl., 17.12.27. Ger., 24.12.26).—See B.P. 282,805; B., 1928, 849.

Manufacture of azo dyes insoluble in water. L. LASKA and A. ZITSCHER, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,762,022, 3.6.30. Appl., 29.1.29. Ger., 1.2.28).—See B.P. 323,937; B., 1929, 366.

Anthracene dyes (B.P. 303,375).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Examination of the fine structure of vegetable fibres by means of concentrated sulphuric acid. A. P. SAKOSTSCHIKOV (Textilber., 1930, 11, 441—443).—The fine structure of vegetable fibres such as linen, hemp, ramie, and cotton is conveniently revealed by covering them with a few drops of sulphuric acid (*d* 1.83) on a glass microscope slide and then arresting the dissolution of the fibre by adding glycerin; greater clarity is obtained by then adding a few drops of a

solution of zinc iodochloride and washing the stained fibres with lactic acid. After such treatment the fibres appear to consist of small units bounded by parallel planes at right angles to the fibre axis. Numerous photomicrographs are given.

A. J. HALL.

Comparison of various brands of cellulose acetate silks. H. STADLINGER (Textilber., 1930, 11, 450—458).—A comprehensive comparison is made of the physical and dyeing properties of Setilose (Fabr. de Soie Artificielle de Tubize), Celanese, Fr. Rhodiaseta (Soc. pour la Fabr. de la Soie Rhodiaseta, Rousillon), G. Rhodiaseta (Deutsche Acetat-Kunstseide A.-G. "Rhodiaseta," Freiburg), Aceta (Aceta Ges.m.b.H., Berlin-Rummelsberg), Apex, and "Drya" (N.V. Nederlandsche Kunstzijdefabriek) silks. The acetyl contents (%) of the silks are: Setilose (53.2), Celanese (53.8), Fr. Rhodiaseta (54.4), G. Rhodiaseta (54.3), and Aceta (54.9), and the viscosities of their 2% solutions in acetone are 5.70, 5.80, 5.55, 5.75, and 8.85, respectively. The dry and wet tensile strengths, and also the dry tensile strength after immersion for 0.5 hr. in boiling water (g. per 1 denier at 20° and R.H. 60%), are, respectively: Setilose (1.22, 0.64, 1.08), Celanese (1.23, 0.64, 1.08), Fr. Rhodiaseta (1.25, 0.76, 1.12), G. Rhodiaseta (1.33, 0.78, 1.22), and Aceta (1.34, 0.90, 1.36). Under similar conditions the extensibilities were 28.2, 31.7, and 20.5%; 26.0, 32.2, and 19.4%; 27.7, 38.5, and 21.5%; 24.7, 32.8, and 19.9%; 26.4, 36.4, and 19.9%, respectively. Celanese and Fr. Rhodiaseta silks lost much lustre and became woolly, whereas Setilose and G. Rhodiaseta silks lost but little lustre and became only slightly woolly, and Aceta silk was almost unaffected when boiled in water for 0.5 hr. Setilose silk had the highest affinity for Cellit (cellulose acetate) dyes, Celanese having the next highest affinity, and the other silks considerably less affinity; Celanese showed an abnormally high affinity for Cellit Fast Blue A when dyed with a mixture of dyes containing this dye. Setilose and Aceta silks have the softest handle, and G. Rhodiaseta the harshest. The fluorescence of the silks in ultra-violet light was affected by means of lubricating oils added to the silk, but after extraction with ether Setilose and Aceta silks showed the strongest fluorescence (violet). The cross-sections of Apex, Enka-Drya, and La Soie de Clairoux are similar in that they are ribbon-like and free from indentations; those of Celanese, Aceta, Setilose, and Fr. and G. Rhodiaseta are circular and slightly indented.

A. J. HALL.

Properties of cellophan and transparite. F. LENZE and L. METZ (Kunststoffe, 1929, 19, 217—219, 247—250, 271—277; Chem. Zentr., 1930, i, 773).—Chemically, the differences are small, values being: glycerol 13—14, water 7—8, ash 0.5, cellulose 77—78%, and acid coefficient 7—8. Physical properties were determined.

A. A. ELDRIDGE.

Testing paper for bursting strength. B. SCHULZE (Papier-Fabr., 1930, 28, 267—271, 377—382).—The Schopper-Dalén bursting-strength tester is discussed, and experiments carried out with the instrument are described. It is found that a rate of application of load equal to 0.2 kg./sec. is most suitable for testing, and

that the arithmetic mean of ten tests gives accurate results. The effect of increasing the surface under test is to reduce the bursting pressure and increase the height of the diaphragm at the point of rupture. A testing area of 10 sq. cm. is recommended. The correlation of bursting strength with tensile strength has been investigated; it is considered impracticable to formulate any definite relationship between the two. The elongation under tensile strain may be approximated from the curvature of the sample at bursting point, and curves are given which show the relationship for different testing areas.

T. T. POTTS.

Xylose from cellulosic waste. HALL and others.—See XVII.

PATENTS.

Manufacture of yarns. C. LIÉNARD-FIÉVET (B.P. 304,606, 21.1.29. Fr., 21.1.28).—Short, waste fibres of wool, which have been lubricated with sulphonated castor oil, are bound together into a solid yarn by incorporation with vegetable fibres which have been given a cork-screw form by the lanification treatment described in B.P. 304,602 (B., 1930, 709.)

F. R. ENNOS.

[Manufacture of] cloth-like fabric. C. F. BURGESS LABS., INC., Assees. of M. J. SHOEMAKER (B.P. 317,330, 12.8.29. U.S., 13.8.28).—A water-absorbent sheet of unwoven fibres of cellulose material (wood pulp) is held together by discontinuous films of water-absorbent material (regenerated cellulose), preferably in the form of parallel strips at right angles to one another on the two sides of the sheet, the fibres of which are securely anchored to the adjacent strips.

F. R. ENNOS.

Utilisation of waste wool. S. R. and E. R. TROTMAN, and WOLSEY, LTD. (B.P. 329,766, 4.4.29).—Waste wool is heated under pressure with an aqueous solution of a weakly alkaline salt (borax, sodium phosphate) to extract the protein in a form which is soluble in water, but which has undergone as little degradation as possible.

F. R. ENNOS.

Manufacture of artificial silk etc. from viscose. DEUTS. ZELLSTOFF-TEXTILWERKE GES.M.B.H., Assees. of K. LEUCHS (G.P. 461,749, 13.1.21).—The precipitation bath comprises a sulphuric acid solution of sodium and aluminium sulphates, preferably in equimolecular ratio, the content of $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ being greater than 15%.

A. R. POWELL.

Spinning of tubular bodies from cellulose solutions such as viscose. KALLE & Co. A.-G. (B.P. 306,851, 25.2.29. Ger., 23.2.28).—Viscose is spun through an annular nozzle open at both ends, and the tubular body formed is distended by introducing the precipitant in a strong jet into its interior by means of a pipe projecting into the nozzle. A core consisting of a bar carrying one or more rings and provided at its end with a triangular member, the width of which can be adjusted as desired, is caused to descend gradually into the tubular body, and precipitation is completed by passing the precipitant through the supporting rings.

F. R. ENNOS.

Manufacture of non-fibrous cellulose flour. E. R. DARLING, ASSR. to CORNSTALK PRODUCTS Co., INC.

(U.S.P. 1,751,267, 18.3.30. Appl., 7.6.28).—Cottonseed hulls are heated in an oxidising solution for a predetermined time and at a predetermined temperature to oxidise the constituents other than cellulose, which are removed by washing with water and heating under pressure with caustic soda and sodium sulphite; the product is finally washed, bleached, again washed, and dried.

F. R. ENNOS.

Manufacture of cellulose acetate. U.S. INDUSTRIAL ALCOHOL Co. (B.P. 329,718, 22.1.29. U.S., 24.2.28).—After acetylating cellulose in the presence of liquid sulphur dioxide as in B.P. 306,531 (B., 1930, 609), the ester produced is partially hydrolysed in the same medium to the required solubility by the addition of water, with or without acetic acid or alcohol. By abrupt liberation of the sulphur dioxide as gas the product is discharged from the reaction vessel in a light or fluffy form.

F. R. ENNOS.

Utilisation of cellulose ester waste. R. SPELLING (B.P. 311,368, 2.3.29. Ger., 10.5.28).—After removal of their chemical coating, worn-out films etc. are dissolved in a suitable solvent and spun into thread, new spinning solution being added, if necessary, to bring the prepared solution to the desired viscosity. [Stat. ref.]

F. R. ENNOS.

Apparatus and process for separating or extracting cellulose or paper pulp. E. SPENCER (U.S.P. 1,761,543—4, 3.6.30. Appl., 10.2.28. Brit. India, 28.5.27).—See B.P. 283,910; B., 1929, 13.

Cementitious product [from cellulosic material]. R. A. MARR, ASSR. to RAMAR SYND., INC. (U.S.P. 1,762,481, 10.6.30. Appl., 12.1.24. Renewed 31.7.26).—See B.P. 266,168; B., 1927, 295.

Manufacture of cellulose derivatives. W. H. GLOVER and C. DIAMOND, ASSR. to COURTAULDS, LTD. (U.S.P. 1,763,428, 10.6.30. Appl., 3.2.27. U.K., 10.4.26).—See B.P. 268,552; B., 1927, 473.

[Abrasive-covered] rollers for drawing, spinning, or otherwise treating fibrous materials. D. MCL. PROCTOR, and CARBORUNDUM Co., LTD. (B.P. 330,323, 16.3.29. Addn. to B.P. 275,762).

Materials in sheet form (B.P. 329,691). **Centrifugal separator** (U.S.P. 1,749,291).—See I. **Esters of carbohydrates** (B.P. 305,661).—See III. **Laminated glass** (B.P. 306,397).—See VIII. **Insulating materials** (B.P. 308,224).—See XI. **Products from wool fat** (B.P. 305,597). **Emulsions** (B.P. 329,266).—See XII. **Impregnation of paper etc. with varnishes etc.** (B.P. 329,066).—See XIII. **Rubbered fabrics** (U.S.P. 1,749,743). **[Filaments from] chlorinated rubber** (B.P. 328,818).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

X-Ray study of mercerisation. G. VON SUSICH and W. W. WOLFF (Z. physikal. Chem., 1930, B 8, 221—230: cf. A., 1929, 1354).—The process of mercerisation has been followed by means of X-ray photographs taken during the course of the reaction, and hydrated soda-pulp has been detected as an intermediate compound. Diagrams of untreated ramie, hydrated and non-hydrated soda-pulp, and mercerised cellulose are given.

The X-ray diagram of the intermediate compound is identical with that obtained under similar conditions by Katz and Mark (A., 1925, ii, 660). Concentration and temperature conditions for mercerisation by lithium, sodium, or potassium hydroxides are tabulated.

F. L. USHER.

Submerged combustion. HAMMOND.—See II. **Comparison of acetate silks.** STADLINGER.—See V.

PATENTS.

Dyeing of esters or ethers of cellulose or of its transformation products. I. G. FARBENIND. A.-G. (B.P. 328,308, 25.1.29. Addn. to B.P. 304,739; B., 1930, 506).—Cellulose acetate etc. is dyed with 4-amino-1:8-naphthalimides carrying as substituent in the imide group a hydroaromatic residue, *e.g.*, the cyclohexyl derivative, m.p. 240—242°.

C. HOLLINS.

Dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 328,706, 13.2.29).—Level shades on viscose are obtained by the use of disazo dyes from tetrazotised 1:5-naphthylenediamine coupled with (a) a phenol, naphthol, a phenolcarboxylic acid, a naphtholsulphonic acid, or an aminonaphtholsulphonic acid, and (b) another coupling component, J-acid and its derivatives being excluded in both cases. Preferably there is present one sulphonic group for each naphthalene residue. Examples are 1:5-naphthylenediamine with 1:8-aminonaphthol-2:4-disulphonic acid and β -naphthol (blue), with salicylic acid and methyl β -naphthylamine-7-sulphonic acid (red), or with N.W.-acid and γ -acid (violet).

C. HOLLINS.

Dyeing of chrome leather with basic dyes. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 304,608, 21.1.29. Ger., 19.1.28).—Chrome leather is dyed with basic dyes with the aid of highly sulphonated neutral fats, fatty acids, resin acids, naphthenic acids, petroleum distillates, brown-coal tar, or sulphonated condensation products, such as are described in B.P. 269,917, 275,267, 288,126, 308,280, 313,861, and 289,841 (B., 1927, 650; 1929, 292, 529, 727, 708; 1930, 276).

C. HOLLINS.

Production of a wool finish on cotton goods. R. G. KNOWLAND and G. T. LOVERIDGE, ASSRS. to NASHUA MANUFG. CO. (U.S.P. 1,751,089, 18.3.30. Appl., 11.5.26. Renewed 13.8.29).—Cotton goods are treated with caustic alkali of mercerising strength until the fibre has become permanently swollen, the material being allowed to shrink freely during the process. After removal of a considerable part of the alkali by washing, the goods are dried by ironing while still retaining a part of the caustic soda, which is finally washed out with water.

F. R. ENNOS.

Conversion of textile vegetable fibres into threads having the texture of wool. F. G. MAILLARD (B.P. 329,792, 24.4.29).—Fibres of jute, hemp, etc., after mechanical treatment in a greasy bath, are spun into thread, doubled, and treated with a solution of alkaline carbonate at 60° followed by a solution of sodium sulphuricinoleate and alkaline carbonate at ordinary temperature in order to separate the ligneous constituents, which are then hydrolysed by means of caustic soda. The threads are finally bleached, finished,

and rendered impermeable by electrolytic treatment in a bath of aluminium sulphoacetate.

F. R. ENNOS.

Treatment of vegetable fibres to effect their lanification. C. LIÉNARD-FIÉVET (B.P. 304,602, 21.1.29. Fr., 20.1.28).—Vegetable fibres, which have been freed from pectic substances by steeping in a 1% sodium carbonate solution and then in an alkaline solution of vegetable oil ("Tetrapol"), are crimped or waved by treatment with a solution of sodium carbonate and Monopol soap. After washing, the fibres are neutralised, bleached by the action of sulphur dioxide and trioxide, and finally dried by hot air.

F. R. ENNOS.

Production of lustre effect on textile fabrics [by calendering]. K. WESSEL, ASSEE. of A. BOSSHARD (B.P. 328,175, 8.8.29. Ger., 11.3.29).—Fabric is dried by passing over drying cylinders, the last two of which are electrically insulated and driven at a slightly slower rate than the remaining ones, so that the fabric becomes charged electrostatically by friction, and are then calendered between two jute rollers each heated to about 180° by contact with a gas-heated steel roller.

A. J. HALL.

Dressing of textile materials. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 320,018, 30.9.29. Ger., 29.9.28).—The material is treated with an aqueous solution or emulsion of a drying oil, *e.g.*, linseed oil, wood oil, preferably boiled or mixed with a siccativ, or of a varnish made therefrom.

F. R. ENNOS.

Waterproofing of sailcloth and similar textiles. H. LANDMARK and W. NAGEL (Nor.P. 42,619, 4.6.25).—The material is impregnated with an emulsion of a drying or semi-drying oil (linseed oil, whale oil, etc.) in a solution containing chromium salts and glue, preferably together with asphalt, paraffin, pigments, cellulose, wood-pulp, or similar fillers or colouring materials.

A. R. POWELL.

Increasing the lustre of goods made of animal hair or wool. E. BÖHM (U.S.P. 1,760,738, 27.5.30. Appl., 18.9.28. Austr., 1.12.26).—See B.P. 281,646; B., 1928, 520.

Dyeing and like machines. C. S. BEDFORD (B.P. 330,377, 8.5.29).

Salvaging cleaners' gasoline (U.S.P. 1,751,613).—See II. **Wetting agents etc.** (B.P. 300,574 and 328,675).—See III. **Dyeing rubber** (U.S.P. 1,742,757). **Colouring rubber** (B.P. 329,761).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of water by distillation with tetrachloroethane. W. LEPPER (Z. Unters. Lebensm., 1930, 59, 79—81).—The use of tetrachloroethane as the distillation medium in the apparatus of Pritzker and Jungkuntz (cf. A., 1929, 1029) is unsatisfactory owing to its corrosiveness, its sensitivity towards alkalis, and its tendency to split off hydrogen chloride in the presence of water. When the method was applied to the determination of the water content of sucrose, decomposition occurred resulting in very high values. For flour and starch-containing materials a liquid having

a b.p. lower than that of tetrachloroethane is essential, and the apparatus of Spiehl and Striemann (cf. *Z. angew. Chem.*, 1927, **40**, 464), using benzene, toluene, and xylene mixtures, is recommended.

H. J. DOWDEN.

Synthetic preparation of ammonia under hyper-pressures and production of the necessary hydrogen. G. CLAUDE (*Z. angew. Chem.* 1930, **43**, 417—423).—A historical account of the author's work. He early reached the conclusion that hydrogen for ammonia synthesis could never be sufficiently freed from carbon monoxide by liquefaction of the latter at -190° , its b.p. A liquefaction method of separation, using lower temperatures, was gradually developed which yielded hydrogen of 94% purity, but the process was tedious. The author's earlier experiments on ammonia synthesis were conducted with electrolytic hydrogen which was in France too expensive for commercial use, but in 1917 the dissolution process for the removal of carbon monoxide from water-gas made it possible to obtain hydrogen containing less than 0.2% CO. The complication of the dissolution process caused the author to return to liquefaction, and he solved the principal difficulty, viz., that of finding a lubricant capable of use below -200° , by employing liquid nitrogen. This yielded a hydrogen of 99% purity. The final success was the removal of the remaining 1% by catalytic reaction ($\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$) at 350° . In 1922 the Claude process was first successfully applied to coke-oven gas. In this case ethylene is obtained as a by-product and serves for the manufacture of ethyl alcohol, and the traces of carbon monoxide left in the hydrogen can be used for the synthesis of methyl alcohol. The advantages of the use of hyper-pressures are discussed. The thermal conditions are, however, entirely different from those of the Haber process. Whilst in the latter electric heating is often necessary, in the Claude process there is an excess of heat which must be removed. This is effected by cooling the catalyst with the reaction gases without the use of a heat exchanger. The total energy consumption of the Claude process as realised in existing plants is below 2.5 kw.-hrs. per kg. of anhydrous ammonia. C. IRWIN.

Types of common salt and its standardisation.

I. K. EROSHKIN (*Ann. Inst. Anal. Phys. Chem.*, 1930, **4**, 291—305).—The necessity of the adoption of standards for the common salt industry is urged.

R. TRUSZKOWSKI.

Mechanism of the azide-iodine test for thiosulphate and its quantitative application. II. BAINES (*Phot. J.*, 1930, **70**, 235—236).—The azide test (cf. Jelley and Clark, B., 1930, 55) depends on the formation; during the reaction between thiosulphate and iodine in very dilute solution, of a substance, other than tetrathionate, which is capable of giving a positive reaction with sodium azide. Attempts to identify this substance as an unstable sulphur compound, such as sulphylic acid, were not successful. A method is outlined for the application of this test to the determination of traces of thiosulphate.

J. W. GLASSETT.

Water solubility of the phosphoric acid of monocalcium phosphate. W. STOLLENWERK (*Z. Pflanz. Dting.*, 1930, **17A**, 42—47).—In concentrated solutions

monocalcium phosphate, both pure and in the form of superphosphate, undergoes decomposition with the formation of free phosphoric acid. The extent of the decomposition decreases with increasing dilution. (Cf. Schneider, Diss. Giessen, 1929.) A. G. POLLARD.

Rapid opening-up of calcium cyanamide. W. LEPPER (*Z. anal. Chem.*, 1930, **80**, 331—334).—The crude cyanamide (5 g.) is mixed in a 500-c.c. flask with copper sulphate (1 g.) and powdered potassium sulphate (10—20 g.), and 50 c.c. of concentrated sulphuric acid are added. The flask is heated for 15 min. by a small flame until the initial reaction is over, and the liquid then boiled for a further 15 min. The contents of the flask are then made up to 500 c.c. and an aliquot part of the solution is distilled with excess of sodium hydroxide in the usual way. Concordant results cannot be assured if less than 5 g. of the material be taken for analysis.

H. F. HARWOOD.

Volumetric determination of citric acid in calcium citrate. G. ROMEO (*Riv. Ital. Prof.*, 1929, **11**, xxiii—xxvi; *Chem. Zentr.*, 1930, **i**, 265).—The calcium citrate is converted into the potassium salt and treated with an excess of standard silver nitrate solution, the excess being titrated with thiocyanate. Oxalic, tartaric, hydrochloric, and phosphoric acids, but not calcium sulphate nor pectic substances, interfere.

A. A. ELDRIDGE.

Antimony pentasulphide. E. SCHÜRMANN and W. BÖHM (*Kautschuk*, 1930, **6**, 70—73, 91—94, 136—141).—After a review of earlier work on the methods of formation and the composition of antimony pentasulphide, an account is given of selected methods for the complete analysis of this raw material for the rubber industry, followed by a record of experiments attempting the production of pure pentasulphide in various ways, wet and dry. Commercial samples always contain more or less free sulphur, after the removal of which the sulphur content of the residue falls below that for the composition Sb_2S_5 , and indicates a mixture of pentasulphide and tri-sulphides. Pure pentasulphide probably has never yet been obtained. It is now found that by passing hydrogen sulphide into a carefully prepared solution of antimony pentachloride containing 12—15% of hydrogen chloride and conducting all operations, including drying, as expeditiously as possible, a product is obtained which, after the removal of the small proportion of free sulphur by purified carbon disulphide, is free from any reducing action on ammoniacal silver solution, and represents substantially pure antimony pentasulphide.

D. F. TWISS.

Manufacture of sulphur chloride from pyrites, alkaline-earth sulphates, and refuse from gas purifiers. V. N. IPATIEV and V. V. VASILEVSKI (*J. Appl. Chem.*, Russia, 1929, **2**, 689—701).—When chlorine is passed through refuse from gas purifiers a mixture of sulphur mono- and di-chlorides (e.g., 58:42) is obtained; iron catalyses the formation of the latter, whilst decrease in the rate of passage of the gas or distillation in presence of excess of sulphur favours that of the former. Gaseous cyano-compounds are also formed. In the similar preparation (e.g., 89:11) from pyrites the maximum yield is obtained at 200° (based

on the amount of pyrites reacting) or 300° (on the amount of pyrites present). At 500° activated carbon does not react with barium sulphate in absence of chlorine, and chlorine has no appreciable action on calcium sulphate in absence of carbon; by joint action on either sulphate, the metal chlorides are formed in yields 91% and 81% of the theoretical, respectively. Increase in the amount of carbon diminishes the yield of barium chloride to 76–85%, whilst 27–30% of the sulphur is converted into the monochloride. CHEMICAL ABSTRACTS.

Welded joints and corrosion. KÜRSCHNER; GROSS.—See II.

PATENTS.

Manufacture of phosphoric acid and hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,711, 15.2.29).—The formation of red phosphorus in a coherent form during the production of phosphorus acids from liquid phosphorus and water at elevated temperatures and pressures is prevented, *e.g.*, by bringing the phosphorus introduced into the pressure-tight reaction vessel into immediate intimate contact with the water. Thus phosphorus and water may be continuously or periodically introduced in small quantities into the vessel heated at such a temperature that interaction occurs in a short period of time. Alternatively, larger quantities of phosphorus and water may be introduced and finely dispersed and intermixed by stirring; or the phosphorus may be quickly heated to a temperature (300 – 400°) at which it reacts rapidly with water. It is also advantageous to supply the phosphorus in a state of fine subdivision, *e.g.*, as a fine spray. S. K. TWEEDY.

Preparation of substitutes for table salt suitable for flavouring purposes. CHEM. PHARM. A.-G. (B.P. 312,088, 17.5.29. Ger., 19.5.28).—The flavour of sodium formate, lactate, acetate, or other organic salts or of potassium chloride is improved by addition of glutamic acid, sodium hydrogen glutamate, or mixtures of amino-acids obtained by hydrolysis of albumins, so that the p_H value is moved towards the acid side (about +5). The products have a purely saline taste. C. HOLLINS.

Production of alkali [sodium carbonate] from natural brine. R. CUMMINGS, Assr. to R. D. PIKE (U.S.P. 1,751,132, 18.3.30. Appl., 14.12.27).—The brine from Green River, Wyoming, is concentrated in salting-out evaporators until about 86% of the water has evaporated and about 44% of the total Na_2O is precipitated and removed as sodium carbonate monohydrate; the mother-liquor, after cooling to 30 – 35° , is carbonated to precipitate most of the remaining sodium carbonate as the bicarbonate which, as well as the monohydrate, is calcined to yield soda ash. The final liquor is treated for the recovery of sodium chloride, bromine, etc. L. A. COLES.

Working natural sodium sulphate deposits. G. F. ANDERSEN, Assr. to WELTHA PROCESS CORP. (U.S.P. 1,751,901, 25.3.30. Appl., 12.11.28).—In lakes containing water saturated with sodium sulphate, silt-proof walls are built enclosing a portion of the lake; from this portion the silt is removed and the clear solution allowed to flow into it in such a manner as not to disturb the bottom. In due course long columnar-crystals of

pure sodium sulphate grow up from the bottom, and may readily be collected free from entrained silt.

A. R. POWELL.

Precipitation of potassium cobaltinitrite. "SACHTLEBEN" A.-G. F. BERGBAU U. CHEM. IND., and H. NITZE, Assees. of H. NITZE (G.P. 461,898, 5.5.27).—The precipitation is effected in a solution containing alkali hydrogen sulphate and a smaller proportion of acetic acid than that usually employed. A. R. POWELL.

Recovery of neutral ammonium sulphate from the mixture of crystals and liquor obtained in an ammonia sulphate saturator. C. STILL (B.P. 310,536, 25.4.29. Ger., 28.4.28).—An enclosed rotary plane or drum filter is described, adapted so that gases or air containing ammonia can be led over and/or through the layer of salt deposited on the surface of the filter for the purpose of neutralising the acid mother-liquor adhering to the filtered salt. S. K. TWEEDY.

Mixtures of nitrate of ammonia and nitrate of lime. APPAREILS ET EVAPORATEURS KESTNER (B.P. 316,664, 11.7.29. Fr., 2.8.28).—Dry, non-deliquescent mixtures of the compounds are prepared by concentrating the calcium nitrate to about 82% before mixing it with the ammonium nitrate. L. A. COLES.

Manufacture of alkali, alkaline-earth, and ammonium nitrates from chlorides. E. E. NAEF (F.P. 638,551, 9.12.26).—The chlorides are heated at 50 – 80° with brown, fuming nitric acid preferably in the presence of manganese dioxide or sulphuric acid.

A. R. POWELL.

Obtaining alkaline-earth thiocyanates [from gas-purification liquors etc.]. R. R. FULTON, Assr. to KOPPERS Co. (U.S.P. 1,751,274, 18.3.30. Appl., 23.12.26).—The liquor is treated with sufficient calcium chloride to precipitate thiocyanates, thiosulphates, carbonates, and ferrocyanides as their calcium salts and the mixture is heated at 110 – 120° to decompose the calcium thiosulphate into calcium sulphite and sulphur; after removal of the precipitated material, the solution is concentrated and cooled to remove sodium chloride, treated with clarifying agents (lime, bleaching powder, etc.) to remove colloidal and coloured impurities, and finally concentrated to crystallise calcium thiocyanate. L. A. COLES.

Production of hypochlorite compositions. MATHIESON ALKALI WORKS, Assees. of (A–C) R. B. MACMULLIN and (A, C) M. C. TAYLOR (B.P. 317,437, 319,727, and 329,896, 12.7.29. U.S., [A] 16.8.28, [B] 27.9.28, and [C] 29.12.28).—(A) A new triple salt, $\text{Ca}(\text{OCl})_2 \cdot \text{NaOCl} \cdot \text{NaCl} \cdot 12\text{H}_2\text{O}$, crystallising in hexagonal prisms and stable below 22° , is obtained by chlorinating a mixture of 37 pts. of calcium hydroxide, 40 pts. of sodium hydroxide, and 180 pts. of water at 0 – 16° , or by adding sodium chloride to a saturated solution of calcium hypochlorite below 16° . Agitation of the triple salt with a chlorinated milk of lime slurry in suitable proportions at 10° followed by heating at 16° causes the formation of a rigid mass of interlocking crystals of calcium hypochlorite, $\text{Ca}(\text{OCl})_2 \cdot 2\text{H}_2\text{O}$, and sodium chloride, which can be dried *in vacuo* to give a product containing 65–70% of calcium hypochlorite. (B) The triple salt is obtained by chlorinating a mixture

of sodium and calcium hydroxides in a saturated solution of sodium chloride at 10°, separating the precipitated sodium chloride, adding more of the hydroxides, and again chlorinating at 0°. (c) The chlorinated lime slurry to which the sodium chloride is to be added is produced by a two-stage chlorination at (a) above and (b) below 10°, so that calcium hypochlorite dihydrate does not separate. A. R. POWELL.

Simultaneous production of precipitated dicalcium phosphate and nitrates. F. C. and F. PALAZZO (B.P. 316,605, 14.2.29. It., 1.8.28).—Finely-divided raw phosphate mixed to a paste with water is stirred at the ordinary temperature with nitric acid of 30–50% concentration; after removal of insoluble sludge, the solution is treated with sodium or potassium hydroxide or ammonia to precipitate dicalcium phosphate, which is removed, washed, and dried at 50° under reduced pressure. The solution is evaporated to yield a mixture of sodium or ammonium and calcium nitrates, or, where potassium hydroxide has been used for neutralisation, it is concentrated so that, on cooling, the potassium nitrate crystallises out and a residue of calcium nitrate is obtained on further evaporation. The liquors obtained by washing the insoluble sludge and the dicalcium phosphate are used for the production of nitric acid for use in the initial stage of the process. L. A. COLES.

Separation of the constituents of mineral silicates. S. W. SCOFIELD, Assec. of J. B. LA RUE (U.S.P. 1,751,663, 25.3.30. Appl., 7.2.24).—Potash feldspar ground to 35-mesh is calcined for 1–2 hrs. at 800–1050°, then ground to pass 200-mesh, and digested for 1–2 hrs. in an autoclave at 275–325° with 2.5–4 times its weight of a 45% solution of potassium hydroxide. The mixture, after cooling, is treated with 1.4 times its volume of water and carbon dioxide is passed through the liquor to convert all the potassium into potassium hydrogen carbonate and to precipitate aluminium hydroxide and gelatinous silica. The filtered solution is evaporated to recover part of the potassium as carbonate and the mother-liquor is diluted and treated with slaked lime to regenerate potassium hydroxide for use again in the process. A. R. POWELL.

Treatment of greensand or glauconite for rendering it an efficient water-softening agent. F. K. LINDSAY, Assr. to ARIZONA MINERALS CORP. (U.S.P. 1,750,847, 18.3.30. Appl., 19.2.25. Renewed 8.1.30).—Greensand etc. is stirred into a solution containing sodium silicate and a soluble salt (e.g., sodium aluminate) so that the particles are held in suspension in the slowly gelling mass; when gelation is complete the product is drained for 12 hrs. at the ordinary temperature, and then dried in a current of air at about 60°. L. A. COLES.

Manufacture of decomposition products from iron sulphates. I. G. FARBERIND. A.-G. (B.P. 303,808, 9.1.29. Ger., 9.1.28).—The dehydration and decomposition is effected in the presence of, e.g., 10% of a solid decomposition product obtained by heating ferric sulphate, e.g., ferric oxide, which product usually acts as a catalyst and also diminishes the plasticity of the mass so that the heating can be effected in a rotary

furnace. Small quantities of alkali or alkaline-earth oxide, hydroxide, or carbonate may also be added, e.g., when adhering sulphuric acid is present. Should no catalytic action occur a catalyst which promotes the oxidation of sulphur dioxide to sulphur trioxide may be added. S. K. TWEEDY.

Separation of iron from solutions containing cobalt and rich in iron, such as those obtained by lixiviating roasted pyrites. H. E. POTTS. From ORKLA GRUBE-AKTIEBOLAG (B.P. 329,543, 18.6.29).—The ferrous iron, prior to precipitation, is oxidised to ferric iron by treatment with an alkali (sodium) chlorate. L. A. COLES.

Production of pure aluminium compounds suitable for the production of aluminium. W. BACHMANN (B.P. 307,345, 25.2.29. Austr., 5.3.28).—Clay is digested with nitric acid to obtain a neutral solution of aluminium nitrate which, after filtration, is treated with sodium fluoride to produce cryolite or aluminium fluoride. Either of these may be converted into sodium aluminate by digestion with sodium hydroxide, and aluminium hydroxide may be recovered from the aluminate by known means. Alternatively, the fluoride may be heated with silica to produce alumina and silicon fluoride; the latter is then caused to react with water and the hydrofluosilicic acid produced is mixed with the sodium nitrate solution previously obtained to regenerate nitric acid. The precipitated sodium fluosilicate is heated to produce sodium fluoride and silicon fluoride for use again in the process. A. R. POWELL.

Conversion of cobalt and nickel carbonates into hydrated sesquioxides. I. G. FARBERIND. A.-G., Assecs. of F. MEIDERT (G.P. 462,350, 22.4.27).—The carbonate is suspended in water and the suspension treated with an alkali hypochlorite solution containing no free alkali and at least 150 g. of active chlorine per litre. Alternatively, chlorine may be passed into the suspension and alkali hydroxide added from time to time in such a way that an excess is never present. A. R. POWELL.

Manufacture of a readily soluble complex silver compound [thiosulphate]. REMEDIUM CHEMISCHES INSTITUT G.M.B.H. (G.P. 462,782, 14.11.24).—Silver nitrate (2 mols.) is added to a solution containing sodium chloride or bromide (2 mols.) and sodium thiosulphate (3 mols.) Addition of alcohol precipitates the compound $\text{Ag}_2\text{S}_2\text{O}_3 \cdot 2\text{Na}_2\text{S}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$. A. R. POWELL.

Intumescent compositions. A. E. WHITE. From UPSON Co. (B.P. 329,474, 16.4.29).—A mixture of "light magnesium carbonate," sodium silicate solution ($d < 1.51$), and sand, slag, shale, limestone, etc., ground so that 75% passes 30-mesh, is moulded, heated, e.g., by being passed through steam-heated platens, and treated with hot air at about 210°. L. A. COLES.

[Apparatus for absorption of oxides of nitrogen for] production of nitric acid. HERCULES POWDER Co., Assecs. of J. H. SHAPLEIGH (B.P. 329,427, 9.3.29. U.S., 28.1.29).—High, relatively narrow towers constructed, e.g., of chrome iron are packed with hollow glass cylinders each containing an irregular lump of quartz etc., are cooled by an external stream of water, and are provided

at the top with a water inlet and a spent-gas outlet and at the bottom with a gas inlet and an acid outlet.

L. A. COLES.

Purification of gases [e.g., nitrogen and hydrogen for ammonia synthesis]. SOC. D'ELECTROCHIM., D'ELECTROMÉTALL., ET DES ACIÉRIES ELECTR. D'UGINE (F.P. 638,520, 6.12.26).—The compressed gases are bubbled through molten sodium or, preferably, molten sodium-potassium alloy.

A. R. POWELL.

Separation of phosphorus vapours from carbon monoxide. W. KYBER (G.P. 462,521, 19.7.25).—The gases evolved from the furnace in which phosphates are reduced with carbon are passed through a tower packed with porous earthenware and coke, through which water is allowed to trickle and in which the phosphorus condenses, leaving a gas of high calorific value for industrial use. The phosphorus is recovered from the tower packing by burning it in a current of air and collecting the phosphorus pentoxide in an electrical fume-precipitating plant.

A. R. POWELL.

Production of chromates with simultaneous recovery of alumina. H. SPECKETER and G. HENSCHEL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,760,788, 27.5.30. Appl., 23.6.27. Ger., 1.7.26).—See B.P. 273,666; B., 1928, 191.

Manganese-compound recovery. N. A. LAURY, Assr. to J. C. WIARDA & Co. (U.S.P. 1,761,133, 3.6.30. Appl., 22.7.25).—See B.P. 284,098; B., 1928, 235.

Purification of pearl essence. J. PAISSEAU (U.S.P. 1,760,771, 27.5.30. Appl., 9.2.25. Fr., 3.10.24).—See B.P. 240,858; B., 1926, 665.

Absorption apparatus (U.S.P. 1,747,687).—See I. **Welding flux** (U.S.P. 1,750,301).—See X. **Lithopone and zinc sulphide** (G.P. 462,372).—See XIII. **Fertilisers** (B.P. 312,169, 329,326, 329,641, and 329,883). **Dusting powders** (U.S.P. 1,749,575). **Manure from final liquors of potash industry** (B.P. 329,495).—See XVI.

VIII.—GLASS; CERAMICS.

System lime-alumina-silica. E. BERL and F. LÖBLEIN (Arch. Wärmewirtsch., 1929, 10, 339–342; Chem. Zentr., 1930, i, 574).—The behaviour of a series of mixtures heated at and above 1600° was investigated. Mixtures of composition approaching that of aluminium silicate are regarded as refractory. Determinations of resistance to pressure were made. The softening temperature falls with increasing content of lime. The thermal conductivity is but slightly dependent on the chemical composition and changes little with rise of temperature.

A. A. ELDRIDGE.

Constitution of silicate fusions. H. SALMANG (Glastech. Ber., 1929, 7, 277–279; Chem. Zentr., 1930, i, 423).—The scorifiability, viscosity, etc. of refractory materials are influenced by oxides in the fused mass in the following order: calcium, lead, potassium, sodium, magnesium, aluminium, zinc, silicon, boron.

A. A. ELDRIDGE.

Manufacture of ceramic structures. L. MELLERSH-JACKSON. From W. TAYLOR (B.P. 329,011, 5.2.29).—In the drying and firing of clay structures, e.g., water closets, special supports are used which are shaped to

correspond with the desired final shape of that portion of the piece in contact with the support. To facilitate uniform drying, the support may vary in thickness and may be so arranged that thin portions of the piece are in contact with thicker portions of the support.

F. SALT.

Hot-patching [of high-temperature retorts]. H. PARDOE (Trans. Ceram. Soc., 1930, 29, 87–92).—The methods and materials for closing leaks caused by thermal expansion in high-temperature retorts are outlined. The spraying of material of a fluxing nature on to the refractory walls is effective as a preventative. Hot-patching may be carried out by the "paddle" or "peel" method or by the aid of air-guns or detonator guns. A device capable of firing on to a definite target from outside the structure is briefly described. A suitable material for hot-patching contains 3 pts. of silica sand (200-mesh), 2 pts. of precalcined bauxite grog (10-mesh), 3 pts. of powdered plate glass (100-mesh), 2 pts. of powdered vegetable glue, and 1½ pts. of lime. This mixture has m.p. 1550°.

F. SALT.

Behaviour of soluble salts during the burning of clays: the problem of efflorescence. IV. Conditions for the formation of sulphate efflorescences in ceramic materials. A. SIMON and K. SCHMIDER (Z. angew. Chem., 1930, 43, 380–382; cf. B., 1930, 326).—Clay material containing quartz, or quartz material, was admixed with varying quantities of lime (containing magnesia) and heated at 1000° for 3 hrs. and then maintained at this temperature for 5 hrs. in air containing 0.5% or 2% SO₂. For the same lime content the amount of sulphate formed rises with increasing sulphur dioxide concentration, the amount of sulphur dioxide absorbed being directly proportional to its concentration. Similarly, for the same sulphur dioxide concentration the quantity of this gas absorbed and of sulphate formed increases with the rise in lime content, but at a much lower rate than the latter. The temperature interval 300–600° is most favourable for the absorption of sulphur dioxide, maximum absorption probably occurring between 400° and 500°. In the firing of ceramic materials, therefore, the interval 300–600° should be passed as quickly as possible in order to avoid absorption of the sulphur dioxide produced by the combustion of the sulphur of the coal. No absorption occurs at 750° and above; the sulphate detected in the experiments carried out at 1000° is formed during the heating up to this temperature, and although the dissociation pressure of calcium sulphate in presence of silica is already exceeded at 750°, greater energies than are here present are necessary before decomposition occurs. The drying and preheating of ceramic material, therefore, should be completed as far as possible in absence of sulphur dioxide, whilst for sharp firing coal containing sulphur may be used without adverse effect.

S. K. TWEEDY.

Microstructure of some West of England siliceous bricks. J. M. COON (Trans. Ceram. Soc., 1930, 29, 125–137).—Five kinds of bricks made, by similar manufacturing processes, from mixtures of material resulting from kaolinisation of granite were studied. The bricks were fired to a maximum temperature of 1300°. Data are presented on the inversion of quartz to tridymite and

crystalite, the formation of sillimanite or mullite by dissolution of silica in the glassy melt, and on slag formation on exposed surfaces. A number of photomicrographs are reproduced and described. F. SALT.

Decoration of glazed tiles. J. W. THOMPSON (Trans. Ceram. Soc., 1930, 29, 99—111).—Saturated solutions of salts commonly used in the manufacture of pottery were applied with a brush to tiles coated with a soft, lead majolica glaze. After crystallisation had taken place, the tiles were fired to about cone 08A. The effects of using unsaturated solutions, of adding colourless salts to simple solutions, and of mixing simple solutions were also studied. The results are tabulated. F. SALT.

PATENTS.

Production of glasses with changing colour effects. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H., Assees. of F. WEIDERT (B.P. 329,946, 29.10.29. Ger., 15.12.28).—Praseodymium and neodymium oxides in the ratio of from 3 : 4 to 5 : 3 are added to the glass frit to produce glasses which exhibit colours varying from pale ruby-red to greyish-blue and brownish-green, according to the incidence of the light. A. R. POWELL.

Production of heat- and chemical-resisting glass. C. G. BROOKS (B.P. 328,432, 22.4.29).—A borosilicate glass suitable for cooking or chemical ware is made by melting a batch containing <70% SiO_2 , >6% Al_2O_3 , and a moderate amount of boric oxide, together with at least three oxides each of mono- and bi-valent metals, the proportions being determined to give a very low thermal expansion. A preferred composition is: SiO_2 68, Al_2O_3 7.5, B_2O_3 10, Na_2O 3, K_2O 4, Li_2O 1.5, MgO 1, ZnO 2.5, BaO 1.5, CaO 1%. M. PARKIN.

Manufacture of [eye-protecting] glass. H. SEABROOK. From DEUTS. SPIEGELGLAS-A.-G. (B.P. 328,520, 2.8.29).—A glass, absorbing strongly in the infra-red and ultra-violet, besides considerably reducing the visible transmission, is produced by adding cobalt and iron oxides (at least 0.3% on the weight of finished glass) to a batch for producing a colourless glass, and melting the batch under reducing conditions (e.g., with carbon or stannous chloride). Addition of arsenic oxide or antimony oxide increases the absorption of the heat rays. Suitable proportions are Fe_3O_4 1—5%, CoO 0.3—1%, As_2O_3 or Sb_2O_3 0.5—1.5% on the weight of glass. M. PARKIN.

Laminated sheet glass. BRIT. CELANESE, LTD. (B.P. 306,397, 6.2.29. U.S., 18.2.28).—Splinterless glass permeable to ultra-violet rays is produced from pure quartz glass, or glass having a high quartz content, and cellulose acetate, preferably by using the ester as a powder (cf. B.P. 282,723; B., 1928, 853) mixed with sufficient plasticiser to produce the sheet *in situ*, the whole being pressed under 300—500 lb./in.² at 50—80° (preferably 70°) until consolidated. Other cellulose esters or ethers may be substituted for the acetate. M. PARKIN.

Manufacture of yellow glazes. DEUTS. TON & STEINZEUG-WERKE A.-G., Assees. of P. SCHNEIDER (G.P. 462,284, 3.9.27).—Stoneware is coated with any ordinary loam glaze containing more than 30% of calcium carbonate and fired in a salt-glaze furnace. A. R. POWELL.

[Refractory floating rings for use in] glass manufacture. R. E. THRELFALL (B.P. 329,468, 15.4.29).—Clay rings which float readily on glasses of relatively low density are prepared by enclosing a core of suitable material (asbestos, papier maché, cardboard) in the clay before firing, or by building the rings up from hollow sections which are joined together by clay or refractory cement. L. A. COLES.

Manufacture of refractory insulating material. METALLGES. A.-G. (B.P. 304,729, 24.1.29. Ger., 25.1.28).—Natural minerals rich in magnesium orthosilicate, or hydrosilicates, such as serpentine, capable of being converted into orthosilicates by preliminary heat treatment at 800—1000°, are crushed, graded, and used to prepare insulating materials, suitable for metallurgical furnaces, coke ovens, etc., that retain both their insulating and mechanical properties at high temperatures. The material together with binders (sawdust, cork waste, etc.) and magnesium nitrate or carbonate, aluminium nitrate, or other gas-producing substances which do not sensibly reduce the refractoriness of the product are moulded and fired at a temperature insufficient to cause fusion. M. PARKIN.

Manufacture of refractory materials. L. MELLSHER-JACKSON. From HARTFORD-EMPIRE CO. (B.P. 329,803, 27.4.29).—Grog for the manufacture of refractory materials is prepared by compressing the raw materials, e.g., fireclay, with a moisture content of about 12%, into compact blocks under 200 lb./in.², firing the blocks under the usual conditions, and breaking them down to the desired grade for incorporation in fireclay mixtures for the manufacture of bricks for glass-melting tanks. A. R. POWELL.

Porcelain articles and their manufacture. A. S. WATTS (B.P. 330,397, 1.6.29).—See U.S.P. 1,728,382; B., 1928, 919.

Artificial plagioclase compounds. F. SINGER (U.S.P. 1,759,919, 27.5.30. Appl., 9.12.27. Ger., 17.12.26).—See B.P. 282,402; B., 1928, 817.

Methods and apparatus for manufacture of tubes made of a fusible material which can be drawn out, in particular glass tubes. P. A. FAVRE (B.P. 304,664, 22.1.29. Belg., 23.1.28).

Mechanism for feeding molten glass. L. MELLSHER-JACKSON. From HARTFORD-EMPIRE CO. (B.P. 330,366, 27.4.29).

[Vacuum apparatus for manufacture of splinterless] sheet glass. J. JEFFRAY (B.P. 327,949, 12.11.28).

Apparatus for coating glass sheets. R. W. MUNRO, LTD., and J. F. WILSON (B.P. 330,290, 5.3.29).

IX.—BUILDING MATERIALS.

Significance of fineness and chemical composition in the water requirement of cement. H. KÜHL (Tonind.-Ztg., 1929, 53, 1509—1510; Chem. Zentr., 1930, i, 882).—The effect of fine grinding in diminishing the setting time was examined quantitatively. A. A. ELDRIDGE.

Petroleum products. CARPENTER.—See II.

PATENTS.

Rotary kiln for burning cement. KLÖCKNER-WERKE A.-G. ABT. MANNSTAEDTWERKE (G.P. 461,431, 7.1.25).—The furnace head is joined firmly to the cooler so that both rotate simultaneously, and compressed air is blown through the cooler to assist the chimney draught. A. R. POWELL.

Manufacture of a heat-insulating material from gypsum. J. and H. SCHEIDEMANDEL (G.P. 461,889, 9.6.25).—Hot milk of lime is added to hot sulphuric acid, whereby a vigorous action ensues and the water is removed by the heat evolved, leaving a fine porous mass of gypsum of $d < 0.35$. A. R. POWELL.

Production of moulded articles. BELL'S UNITED ASBESTOS Co., LTD., J. A. CANN, and E. R. HARRAP (B.P. 329,330, 16.11.28).—A mixture of basic and acid compounds which react to form difficultly soluble compounds is moulded under the influence of heat and pressure; e.g., Portland cement, calcined magnesite, or lime may be mixed with citric, tartaric, oxalic, tannic, lactic, or boric acid. Citric or oxalic acid is particularly suitable for Portland cement, and boric or oxalic acid is preferred for magnesite. (Cf. B.P. 326,825; B., 1930, 511.) C. A. KING.

Manufacture of moulded or rolled articles. W. H. BOORNE (B.P. 329,382, 19.2.29).—Tiles, slabs, etc. may be moulded by hot-pressing a mixture composed of an acid binder, e.g., resin (or pitch) and clay, a basic compound, e.g., lime, a filler, e.g., sawdust or sand, and 2–8% of water. C. A. KING.

Manufacture of blocks, bricks, slabs, etc. for the construction of roads, buildings, etc. A. F. BERRY (B.P. 329,291, 9.2.29).—A mixture composed of a hard base material, comminuted cement, a metal or alloy powder, and a chemical which will react with the other constituents with the generation of gas to cause the brick to be porous is moulded and allowed to harden. Such a mixture may contain broken vitreous clayware, Portland cement treated with a 10% solution of sodium thiosulphate, and a ferrosilicon alloy (Stalloy). C. A. KING.

Manufacture of materials for use in the construction of roads, roofs, etc. SOUTH METROPOLITAN GAS Co., H. PICKARD, and C. M. C. HUGHES (B.P. 329,569, 23.7.29).—A binder (15–18%), made by mixing coal with coal tar at 250–300°, so that the mixture has m.p. 115–122° and a penetration of 1–5, is mixed with stone dust (26–30%) and hard chippings (55–58%). The warmed mass may be pressed into blocks or spread over a road foundation. C. A. KING.

Preservation of wood. GRUBENHOLZIMPRÄGNIERUNG. GES.M.B.H. (B.P. 314,380, 18.6.29. Ger., 26.6.28).—Wood is saturated with a solution under pressure, the solution is drained, and the container evacuated so that only the cell walls are impregnated. A second suitable solution is then forced into the cells and an insoluble protective compound is formed by interaction between the two solutions. The liquids are employed in the form of their aqueous solutions; for the first, zinc, mercury, or copper salts may be used: for the second, alkali fluorides, arsenates, or arsenites. C. A. KING.

Immunising agents for wood, grain, or the like. I. G. FARBENIND. A.-G. (B.P. 304,741, 25.1.29. Ger., 25.1.28).—An *o*-dihydroxybenzene is treated with a solution of arsenious oxide in hydrochloric acid to give products of the type $C_6H_4 \begin{smallmatrix} O \\ \diagup \diagdown \\ O \end{smallmatrix} AsCl$. These and the corresponding hydroxides are used, alone or with other fungicides, for protecting wood, grain, etc. against plant pests. C. HOLLINS.

Grinding of cement materials etc. C. PONTOPPIDAN, ASST. to F. L. SMITH & Co. (U.S.P. 1,762,241, 10.6.30. Appl., 30.12.27. U.K., 8.10.27).—See B.P. 283,091; B., 1928, 194.

Building slabs for walls, floors, pavings, etc. R. G. ROSS and F. A. WILLIAMS (B.P. 330,418, 25.6.29).

[Bull-nose] tiles. J. B. BLAKEY (B.P. 330,383, 16.5.29).

Roof coverings. S. FACDOUELLE (B.P. 330,340, 5.4.29).

Manufacture of [felt-base] floor coverings [from waste]. J. LUNAN (B.P. 330,384, 16.5.29).

Building block (B.P. 329,063).—See I. **Insulating masses** (G.P. 461,493).—See XI. **Paint for cement** (U.S.P. 1,749,923). **Impregnation of wood etc. with varnishes etc.** (B.P. 329,066).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Nitrogen in technical iron. W. KÖSTER (Arch. Eisenhüttenw., 1929–1930, 3, 637–638; Stahl u. Eisen, 1930, 50, 629–631).—The solid solubility of nitrogen, as iron nitride, in α -iron is 0.001% N at 20°, 0.005% N at 200°, 0.01% N at 300°, and 0.02% N at 400°. Relatively slow cooling of mild steel containing nitrogen from below the A1 point does not result in the attainment of a state of equilibrium at room temperature as, by ageing at temperatures up to 100°, considerable changes occur in the magnetic and mechanical properties. After 12 hrs. at 65° the coercivity begins to rise, and at 100° it increases by 3.2 gauss for every 0.01% N precipitated over a period of 14 days. The conductivity also increases proportionally with the amount of nitride precipitated. The reaction can readily be followed in micro-sections etched with Fry's copper reagent, precipitation of nitride being accompanied by the appearance of minute black dots and needles in the ferrite crystals. These needles become coarser and more numerous with rise of ageing temperature, reaching a maximum at 110°, and then decreasing in size and number as the temperature is raised to 250°. The separation of nitride causes a slight increase in the density and torsion value, a slight decrease in the tensile strength, and a considerable increase in the rate of dissolution in acids. The differences in the properties of Thomas steel and open-hearth steel are ascribed to their different nitrogen contents. On ageing a nitrogenous mild steel which has been quenched from just below the A1 point, maxima occur in the hardness curve at 150° and at 300° showing that, although the separation of excess nitride from its supersaturated solid solution takes place independently of the precipitation of carbide

to p_H 2.5 is added; this is followed by addition of calcium chloride, filtration, and electrolysis in a cement cell having graphite anodes and a rotating-disc cathode.

CHEMICAL ABSTRACTS.

Production of electrolytic iron printing plates.

C. T. THOMAS and W. BLUM (Amer. Electrochem. Soc., May, 1930. Advance copy. 13 pp.).—An investigation has been made of the best conditions for the electro-deposition of iron from a bath of the Fischer–Langbein type to form a backing for nickel electrotypes. From a bath which was 3*N* with respect to ferrous chloride and 6*N* to calcium chloride the most satisfactory deposits were obtained at about 90°, using a cathodic current density of 5–7 amp./dm.², provided that the bath was moderately agitated. Mechanical agitation produced by motion of the cathodes themselves is preferred. Air agitation leads to somewhat brittle deposits and lowers the current efficiency. The calcium chloride concentration can be reduced to 2–4*N*, but the acidity of the bath is important and should correspond to a free hydrochloric acid concentration of about 0.01*N*. Hourly additions of acid should be made to make up for loss due to chemical action on the anodes. The anodes may be of Arinco iron, but since the impurities lead to roughening of the cathode deposits when the thickness of the latter exceeds about 1 mm., it was found necessary to enclose each anode in a rectangular alundum pot. The possibility of avoiding this need for using a diaphragm is being examined. The electrolyte is contained in a rubber-lined steel tank with a thin inner lining of cypress wood to protect the rubber against impact or abrasion. Methods of analysis for control of the bath composition are given. The deposited iron has a tensile strength of about 4000 kg./cm.² and an elongation of about 20%. The finished plates are chromium-plated.

H. J. T. ELLINGHAM.

Hydrometallurgy of copper at the Bagdad property.

C. T. BAROCH (Amer. Electrochem. Soc., May, 1930. Advance copy. 25 pp.).—A pilot plant has been operated at Bagdad, Arizona, for the extraction of copper from low-grade sulphide ores by electrolysis of the liquors obtained by the acid leaching of roasted concentrates. A three-stage roasting process has been developed which renders iron practically insoluble, whilst about 90% of the copper can be extracted. The important point is that between the first and second stages the temperature must not be raised above 455° until all sulphide sulphur has been oxidised. The calcine carries about 30% of acid-soluble copper, including about 8% which is water-soluble. A flow sheet of the leaching cycle is given. The filtered leach liquor is mixed with spent electrolyte and the resulting solution, which contains about 50 g. of copper and 25 g. of free sulphuric acid per litre, is electrolysed at 2.1 amp./dm.². The cells are of redwood lined with lead, each containing nine copper starting sheets and 10 cast antimonial lead anodes. The cells are arranged in groups of 16, each group comprising 4 cascade units. Cathodes are drawn after 14 days. The copper content of the electrolyte is reduced to about 20 g./litre. A portion of the spent electrolyte is diverted from the cycle to prevent undue accumulation of impurities, and after plating out the copper in special stripping cells it is

discarded. The cathode deposited in the main cells is 99.98% pure, and the energy consumption is 2.56 kw.-hr./kg. of copper. The concentration of precious metals in the ore is too small to warrant recovery.

H. J. T. ELLINGHAM.

Electrolytic zinc plant of the Evans–Walloway Co. at East St. Louis, Ill. U. C. TAINTON and D. BOSQUI (Amer. Electrochem. Soc., May, 1930. Advance copy. 10 pp.).—A plant for the electrolytic extraction of zinc from Joplin ores, which were formerly treated entirely by the retort process, is now operated at this company's works at Illinois. Flotation concentrate containing 19–58% Zn is dried and roasted, the roaster gases which contain at least 5% SO₂ passing to the neighbouring sulphuric acid plant of the Monsanto Chemical Co. The calcine is separated into a magnetic (ferrite) portion and a non-magnetic (oxide) portion. The ferrite portion is first agitated with the spent electrolyte, and, after adding manganese dioxide to oxidise all dissolved iron to the ferric state, the oxide portion is added until the acid is neutralised and all the iron precipitated. The residue is shipped to the lead smelter. The liquor is filtered, treated with zinc dust to precipitate impurities, and refiltered. The precipitate is agitated with acid, giving a solution from which cadmium is electrodeposited and a residue which goes to the copper smelter. The purified leach liquor passes to the zinc-extraction room, which contains 190 cells in series, each containing 12 aluminium cathodes and 24 grid-shaped anodes composed of lead alloyed with 1% of silver. The cathodic current density is 100 amp./ft.² (10.7 amp./dm.²). Cathodes are stripped every 12–16 hrs. and the zinc is melted and cast: it is guaranteed 99.99% pure. Manganese dioxide is formed at the anodes and settles on the bottom of the cells, whence it is periodically removed, washed, dried, and marketed. Steam-generated power supplied from a neighbouring station is used. The daily capacity of the plant is 50 tons of zinc and 450 kg. of cadmium.

H. J. T. ELLINGHAM.

Germanium in relation to electrolytic zinc production. U. C. TAINTON and E. T. CLAYTON (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.).—In the electrolysis of zinc sulphate solutions derived from the leaching of roasted Joplin concentrate (cf. preceding abstract) violent fluctuations of current efficiency occurred, zinc deposits showed a great tendency to redissolve, and sometimes no zinc was deposited at all. The ordinary impurities such as antimony, cobalt, nickel, and arsenic, which are known to produce such effects, had been removed, so that the influence of some other element in the solution was suspected. This proved to be germanium, which is found to cause serious loss in efficiency at concentrations as low as 1 mg./litre. Experiments showed that treatment of the solution with precipitated ferric hydroxide removed the germanium. Normally the ores contain sufficient iron to prevent this element entering the solution, but some of the Joplin ores are very low in iron and apparently high in germanium. The trouble was remedied by starting with a mixture of concentrates containing a sufficient total proportion of iron to effect complete removal of germanium. H. J. T. ELLINGHAM.

Electrodeposition of zinc on aluminium and its alloys. H. C. COCKS (J. Electroplaters' Dep. Tech. Soc., 1930, 5, 83—90).—An account is given of the influence of p_H , addenda, and current density on the nature of the deposits from zinc sulphate and cyanide baths. With sulphate baths gum arabic (1 g. per litre), and sulphuric acid (to p_H 4) as addenda were preferred, with 10—30 amp. per sq. ft. In cyanide baths the concentrations of cyanide and hydroxyl ions should be low. Sulphate baths are preferred, except when irregularly shaped articles are being plated.

CHEMICAL ABSTRACTS.

Addition agents in bismuth electrolytes. E. F. KERN and T. R. JONES (Amer. Electrochem. Soc., May, 1930. Advance copy. 19 pp.).—The suitability of fluosilicate, fluoborate, cresolsulphonate, perchlorate, fluoride, sulphate, and chloride electrolytes in the electrolytic refining of bismuth has been examined. The most satisfactory was a chloride electrolyte containing 70 g. of bismuth, 50 g. of hydrochloric acid, and either 100 g. of sodium or calcium chloride or 65 g. of magnesium chloride per litre. The solubility of bismuth chloride in the hydrochloric acid solution is notably raised by the presence of these high concentrations of alkali or alkaline-earth chlorides. The character of the cathode deposit is improved by the addition of 0.1 g. of resorcinol, pyrogallol, or benzoic acid per litre. The current density employed was 1 amp./dm.² If the anodes contain lead, sulphuric acid may be added to the electrolyte (except when it contains calcium chloride), and as much as 20 g./litre may be added without altering the nature of the cathode deposit.

H. J. T. ELLINGHAM.

Oven-furnace installation. WALTER.—See I. **Submerged combustion.** HAMMOND. **Electric welding.** KÜRSCHNER. **Corrosion at welded joints.** GROSS.—See II. **Hot-patching of retorts.** PARDOE.—See VIII. **Rubber linings.** COURBIER and BOUSSAND.—See XIV.

PATENTS.

Moulds for the manufacture of castings from metals or alloys. J. Y. JOHNSON. From I. G. FARBININD. A.-G. (B.P. 330,018, 11.3.29).—The moulds are made from, or lined with, sheets made from a pure metal powder compressed and sintered to a porous mass permeable to gases. The metal powder is preferably obtained by the thermal decomposition of metal carbonyls.

A. R. POWELL.

Production of cast iron containing little gas or oxides. C. BRACKELSBURG (B.P. 306,141, 12.2.29. Ger., 16.2.28).—The charge is smelted in a rotary furnace fired by a coal-dust flame produced directly in the furnace to avoid the formation of pointed flames which cause local overheating.

A. R. POWELL.

Compound for filling recesses in metal castings and the like. Z. M. MARR (U.S.P. 1,749,541, 4.3.30. Appl., 16.4.28).—The material comprises a molten mixture of 7.5 pts. by vol. of sulphur, 1 pt. of graphite, and 1 pt. of antimony.

A. R. POWELL.

Preparation of [an iron] aluminothermic mixture. R. W. HYDE (U.S.P. 1,751,550, 25.3.30. Appl.,

29.9.28).—A mixture of iron oxide or iron scale is mixed with a solid carbonaceous material, *e.g.*, coal or coke dust or sawdust, moistened with water, and blast-roasted in a thin layer to convert the greater part of the iron into granular, porous magnetite. The mass is then crushed through 14—20-mesh and mixed with the requisite amount of granular aluminium of the same degree of fineness. The mass reacts rapidly with the evolution of intense heat when fired with the usual primer.

A. R. POWELL.

[Iron] thermit mixture. J. H. DEPPERER (U.S.P. 1,750,162, 11.3.30. Appl., 8.9.28).—A mixture of iron oxide and aluminium with 15% of ground slag from a previous thermit operation is claimed.

A. R. POWELL.

Welding flux [for iron]. R. R. FULTON, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,750,301, 11.3.30. Appl., 16.9.26).—The flux comprises a mixture of 10 pts. of an alloy of 92% Cu, 7% Si, and 1% Fe, 50 pts. of ammonium borate, 6 pts. of manganese, and 24 pts. of 50% ferrosilicon, all ground to pass 20-mesh.

A. R. POWELL.

Soldering [and brazing] flux. C. D. LAWTON, ASSR. to FULTON SYLPHON CO. (U.S.P. 1,748,180, 25.2.30. Appl., 3.6.29).—Aniline hydrochloride is employed, either as such or in solution (*e.g.*, in isopropyl alcohol). It is adapted for use with soft solder.

S. K. TWEEDY.

Increasing the elongation limit and tensile strength of low-carbon steels. VEREIN. STAHLWERKE A.-G. (B.P. 300,553, 12.11.28. Ger., 14.11.27).—The steel is annealed above 900° and cooled slowly, it is then reheated at a temperature below the *A₁* point, *e.g.*, at 650°, quenched, and aged for some weeks at the ordinary temperature or for a short time at not above 250°. [Stat. ref.]

A. R. POWELL.

Decarburisation of ferrochromium containing carbon. F. KRUPP A.-G., Assees. of B. STRAUSS and P. KLINGER (G.P. 461,945, 12.11.26).—Finely-powdered ferrochromium is treated at 1200° with a current of atomic hydrogen prepared by passing hydrogen through a high-tension arc or by subjecting it to a glow discharge under reduced pressure.

A. R. POWELL.

[Copper-chromium] alloy steels. VEREIN. STAHLWERKE A.-G. (B.P. 307,492, 27.2.29. Ger., 10.3.28).—A structural steel contains less than 0.2% C, less than 2% (Cr + Cu), and up to 1% Ni, W, Mo, Ti, and/or V. The preferred copper and chromium contents are 0.5—1.5% and 0.25—0.5%, respectively.

A. R. POWELL.

Manufacture of alloy [tungsten] steels. A. D. SHANKLAND, ASSR. to BETHLEHEM STEEL CO. (U.S.P. 1,749,678, 4.3.30. Appl., 19.5.26).—A plastic, impervious mass of partly fused pig iron and steel scrap is produced in an acid open-hearth furnace and on this mass is placed a mixture of scale, containing tungsten trioxide equivalent to 10% W, and ferrosilicon, containing sufficient silicon to reduce the tungsten trioxide. The whole is covered with a charge of scrap steel and the mixture is heated until fusion occurs. The carbon and silicon contents are then adjusted to the desired values by the addition of iron ore, complete deoxidation being finally effected by means of ferromanganese.

A. R. POWELL.

Production of titanium steel. W. and H. MATHEIUS (B.P. 329,705, 25.2.29).—Steels containing not more than 1% C and 0.8% Ti with small additions of silicon, manganese, chromium, and other metals which improve the properties are claimed, a preferred composition being 0.1% C, 0.2% Ti, 0.5% Si, 0.5% Mn, and 0.5% Cr. The steel is produced by deoxidising a bath containing less than 0.1% C by the addition of carbon-free silicides of ferromanganese, ferrochromium, or ferromanganese-chromium having such a carbon content that 0.1% C remains in the bath; the requisite titanium is then added as carbon-free ferrotitanium. A. R. POWELL.

Elimination of silicon in ferrophosphorus. H. E. WHITE, Assr. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,751,783, 25.3.30. Appl., 4.9.25. Renewed 11.1.30).—Phosphate rock is smelted with silicon and scrap iron or with a high-grade ferrosilicon, using a 10% excess of phosphate to ensure absence of silicon in the resulting ferrophosphorus. A. R. POWELL.

Production of a high lustre on articles made of stellite alloys. VEREIN. STAHLWERKE A.-G. (G.P. 461,866, 22.10.27).—The articles are first pickled in a strong acid to remove oxide scale, then in a weak acid (preferably organic) which does not dissolve the alloy nor appreciably corrode it. A. R. POWELL.

Two-step sulphatising roast [for copper-zinc-lead sulphide ores. F. A. BRINKER, Assr. to J. P. RUTH, JUN. (U.S.P. 1,749,125, 4.3.30. Appl., 21.10.26).—The ore is passed downwards through a multiple-hearth roasting furnace through which air is passed in such a quantity and under such conditions counter-current to the ore stream that the temperature in the lowest, hottest hearth never exceeds 550°. In this operation the iron sulphide minerals are oxidised to a highly-active ferric oxide and part of the copper and lead minerals are desulphurised, but the blende remains unattacked. The discharge from this furnace together with the sulphur dioxide gas mixture therefrom and more air are passed downwards through a rotary tube-furnace maintained at 600–700°, whereby the zinc sulphide and the copper and lead compounds present are converted into sulphates by the sulphur trioxide formed by the catalytic oxidation of sulphur dioxide in the presence of active ferric oxide. This procedure avoids the formation of zinc ferrite. A. R. POWELL.

Continuous blasting of granular or briquetted mixtures of ores and metallurgical products in thin layers. VEREIN. STAHLWERKE A.-G. (G.P. 462,411, 14.7.23).—The charge is treated on a water-cooled grate, the blast air being admitted at such a height above the grate that it cannot penetrate downwards; the charge just above the grate becomes only tacky (without melting), thus forming a bed on which the subsequent blast operations are conducted. A. R. POWELL.

Refining of copper. O. NAESER (G.P. 462,264, 4.5.27).—Arsenic, antimony, and tin are removed from copper by melting the metal under a slag comprising alkali or alkaline-earth sulphates and carbonates. This mixture acts more energetically than do the carbonates alone. A. R. POWELL.

Production of composite metals suitable for hot-rolling, by casting-on coatings of copper-zinc alloys. METALLGES. A.-G., H. VON FORSTER, and E. LAY (B.P. 329,572, 30.7.29).—The brass used for the production of composite metals with an iron core by the casting-on process contains 68–72% Cu, 0.05–1% Mn, and a small proportion of iron, but is substantially free from lead (less than 0.01%). The brass is cast at 1000° around the iron core heated at 1200–1250°, whereby appreciable diffusion of the surface layers occurs and a welded article suitable for hot-working is obtained. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 328,138, 21.5.28. Addn. to B.P. 323,353; B., 1930, 198).—The magnesium content is raised to 0.05–2% in the alloy described in the prior patent.

H. ROYAL-DAWSON.

Manufacture of aluminium or aluminium alloy wires. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 306,898, 15.2.29. Ger., 27.2.28).—Wire bars are cooled by blowing air on the surface during or before their passage through the rolls, in which their diameter is reduced to 10–12 mm. In this way the temperature is prevented from rising above 300°. The wire is then drawn down to 6 mm., during which operation the temperature rises to 150°; to prevent recrystallisation the wire is then chilled prior to coiling on reels. A. R. POWELL.

Forming articles from heat-treatable aluminium-base alloys. (A) R. S. ARCHER and (B) W. C. WINTER, Assrs. to ALUMINUM Co. OF AMERICA (U.S.P. 1,751,468 and 1,751,500, 25.3.30. Appl., [A] 24.12.27, [B] 13.12.27).—(A) An aluminium alloy containing 3–5.5% Cu, 0.75% Mn, and 0.75% Si is heated at 515° to obtain a homogeneous solid-solution structure, forged or rolled into shape at 440°, and quenched to retain the greater part of the copper in solid solution. (B) An aluminium alloy containing 3–5% Cu, 0.5% Mg, and 0.6% Mn is heated to cause the alloying elements to enter into solid solution, then die-pressed while still hot, and cooled rapidly in the mould. A. R. POWELL.

Insulating coating on aluminium or aluminium alloys. L. McCULLOCH, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,751,213, 18.3.30. Appl., 19.11.27).—The articles are boiled for 5–30 min. in a suspension of 5 g. each of lime and gypsum in 1 litre of water, whereby a film is produced having the composition: 76.8% Al_2O_3 , 8.1% CaO, and 15.1% SO_3 . After drying, the film may be made non-porous and more adhesive by impregnation with tung oil, linseed oil, or a synthetic or natural resin. A. R. POWELL.

Non-corrodible metal [aluminium bronze]. W. HADDON and J. WINFIELD (B.P. 329,361, 16.1.29).—The alloy contains 88–90% Cu, 7.5–10% Al, 0.25–0.4% Mn, and 1–3.75% Ni; the nickel may be wholly or partly replaced by iron. The preferred composition is 89.3% Cu, 9.1% Al, 1% Ni, and 0.27% Mn, with small quantities of tin and/or lead. A. R. POWELL.

Articles of high resistance to wear, well polishable, and neutral to chemical agents. F. HAUPTMEYER (B.P. 329,953, 27.1.30).—The articles comprise a mixture of up to 50% of a corrosion-resistant nickel-chromium alloy or a rust-resisting nickel-chromium steel

in powder form with sulphur, tar, rubber, resin, or other inert plasticising material.

A. R. POWELL.

Casting of light metals, more particularly magnesium and its alloys, in sand moulds. I. G. FARBENIND. A.-G. (B.P. 319,272, 30.8.29. Ger., 19.9.28).—Green-sand moulds for use in making magnesium castings are heated internally by passing a current of hot air through them until the chills embedded in the mould attain the desired temperature for casting.

A. R. POWELL.

Manufacture of [gold-beryllium] alloys, particularly for production of sheets, membranes, etc. permeable to cathode, Röntgen, and like rays. ROHM & HAAS A.-G. (B.P. 313,527, 12.6.29. Ger., 12.6.28).—The alloy comprises 3–15% Au and 97–85% Be melted together in a vacuum furnace. The gold is preferably introduced as an amalgam.

A. R. POWELL.

Alloys [e.g., barium-nickel alloys for radio-valve filaments]. WESTINGHOUSE ELECTRIC & MANUF. Co., Assees. of P. H. BRACE (B.P. 317,785, 8.8.29. U.S., 22.8.28).—An alloy of nickel with 5–25% Ba is obtained by mixing finely-divided nickel with barium azide, compressing the mixture into bars, and heating the bars in a vacuum furnace, first at 200° to remove the nitrogen, then at a red heat to cause the barium to diffuse into the nickel. The resulting sintered bar is swaged in an inert atmosphere into a rod which can be drawn down to fine wire. The nickel may be wholly or partly replaced by iron or cobalt and the barium by any other alkaline-earth metal, by magnesium, or by an alkali metal.

A. R. POWELL.

Reduction of tin and other metals from their salts and purification of molten baths of metal salts. E. A. ASHCROFT and F. E. ELMORE (B.P. 329,159, 26.2.29).—Lead, stannous, and cuprous chlorides are reduced to metal by passing hydrogen or a mixture of hydrogen and steam through the fused chlorides at 800°, 600°, and 500°, respectively. A bath of molten lead or tin is preferably maintained below the fused chloride in the first two cases. Fused zinc chloride may be purified from copper, lead, and tin by treatment with hydrogen at a temperature below the volatilisation point of the chloride. [Stat. ref.]

A. R. POWELL.

Production of shaped bodies having a predominating percentage of tungsten or molybdenum. F. KRUPP A.-G. (B.P. 310,876, 22.4.29. Ger., 2.5.28).—Shaped bodies containing tungsten or molybdenum carbides and nickel, cobalt, or iron are prepared roughly by casting and the shaping is finished by applying pressure at 2200–2400°, i.e., just below the m.p.

A. R. POWELL.

Treatment of vanadiferous ores. P. ASKENASY and E. ELÖD (G.P. 461,959, 21.4.25).—The finely-ground ore is digested with nitric acid and oxygen under pressure, sulphuric acid also being added when the ore is free from sulphur.

A. R. POWELL.

Annealing of metal bands. ELECTRIC FURNACE CO., LTD., Assees. of HIRSCH KUPFER- U. MESSINGWERKE A.-G. (B.P. 305,951 and 312,988, [A] 11.2.29, [B] 8.3.29. Ger., [A] 11.2.28, [B] 4.6.28).—(A) The material is run continuously through an electric strip-annealing furnace

the maximum temperature of which, controlled by a thermostat, is reached near the inlet end. The pitch of the resistance heating coils is so arranged that from the region of maximum temperature there is a gradual and regulated fall in temperature towards the outlet end of the furnace. (B) The strip hangs freely in the furnace in a catenary curve and is kept in position away from contact with the walls by passing through a clamping device before entering the furnace.

A. R. POWELL.

Production of easily soldered metal coatings. M. SCHLÖTTER (B.P. 329,346, 16.2.29).—Coatings of tin-lead alloys may be obtained from baths containing 100 g. each of lead and tin *p*-benzenedisulphonates, 50 g. of the free acid, 5 g. of gelatin, and 5 g. of phenol in 1 litre of water. The anodes used consist of lead-tin alloys containing the metals in the same relative properties as in the bath. More readily fusible deposits are obtained by adding 20 g. of cadmium perchlorate to the bath and incorporating a corresponding proportion of cadmium in the anodes.

A. R. POWELL.

Production of electrolytic iron from scrap. R. D. PIKE (U.S.P. 1,751,099, 18.3.30. Appl., 20.7.26).—The scrap is dissolved in a mixture of ferric and ferrous chlorides, the solution is purified by treatment with hydrogen sulphide from the action of sulphuric acid on ferrous sulphide, calcium chloride is added occasionally to compensate for losses of chlorine and ferrous sulphate (from the hydrogen sulphide generator) to precipitate calcium, and the solution is electrolysed to obtain pure iron and a ferroso-ferric chloride anolyte for dissolving more scrap.

A. R. POWELL.

Electroplating process [deposition of copper-nickel alloys]. R. B. P. CRAWFORD and R. D. SNYDER (U.S.P. 1,750,092, 11.3.30. Appl., 26.11.21. Renewed 23.1.30).—An alloy resembling monel metal is deposited from baths having the composition (a) 600 g. of nickel sulphate crystals, 20 g. of copper acetate, 40 g. of nickel acetate, and 10 g. of sodium chloride per litre, or (b) 97.5 g. of nickel ammonium sulphate, 3.75 g. of copper oxalate, 7.5 g. of nickel chloride, and 20 g. of boric acid per litre, with enough ammonia to dissolve the copper oxalate. About 0.05% of glue may be added to the baths to improve the lustre of the deposits.

A. R. POWELL.

Chromium[-plating] bath for the production of white, soft, and easily polishable chromium deposits. J. BAUER (B.P. 310,427, 25.4.29. Ger., 25.4.28).—A cerium or yttrium salt is added to the ordinary chromium-plating bath in such quantity that the bath contains 5–15 g. Ce or 2–10 g. Yt per litre.

A. R. POWELL.

Electrodeposition of chromium. ELEKTRO-CHROM GES.M.B.H. (G.P. 461,987, 19.2.26. Addn. to G.P. 398,054).—The electrolyte comprises a solution of ter- and bi-valent chromium compounds obtained by fusion of chromium trioxide with finely-divided carbon, cellulose, paper, leather, or proteins, or with aluminium or magnesium powder.

A. R. POWELL.

Production of protective coatings of chromium. ELEKTRO-CHROM GES.M.B.H. (G.P. 462,275, 30.10.24.

Addn. to G.P. 458,632; B., 1930, 290).—The intermediate layer between the iron or steel and the chromium consists of manganese or tantalum.

A. R. FOWELL.

Electrolytic deposition of heavy metals, [e.g., lead and copper]. M. SCHLÖTTER (B.P. 329,324, 14.2.29. Ger., 9.11.28).—The electrolyte comprises a solution of salts of the metal with one or more inorganic acids and with one or more non-carboxylic organic acids. For the deposition of lead the bath contains 200 g. of lead *p*-benzenedisulphonate and 100 g. of lead perchlorate per litre, and for the deposition of copper it contains 200 g. of copper *p*-benzenedisulphonate, 100 g. of copper sulphate, 5 g. of gelatin, 5 g. of tannin, and 10 g. of *p*-benzenedisulphonic acid per litre.

A. R. POWELL.

Production of firmly adherent electrolytic metal [lead] deposits upon articles plated in the molten bath. H. STEFKE (B.P. 330,040, 26.3.29).—Iron or copper alloys are plated with nickel or copper and then immersed in a bath of molten lead. The lead-coated material is made the anode in an acid plating bath, containing lead fluosilicate, acetate, or phenolsulphonate until the crystal structure of the lead coating is fully developed; the current is then reversed and the coating built up to the desired thickness by electrodeposition of lead.

A. R. POWELL.

Preparation of alkali metals by fusion electrolysis. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (G.P. 461,694, 18.6.25).—The raw material used in the preparation of metallic sodium is the sodium chloride obtained by the evaporation of solutions of sodium hydroxide produced by the electrolysis of brine. The crystals so obtained are washed with brine until free alkali is removed or they are neutralised directly with hydrochloric acid.

A. R. POWELL.

[Multiple-hearth] roasting furnace construction. H. J. HARTLEY, ASSR. to NICHOLS COPPER CO. (U.S.P. 1,760,694, 27.5.30. Appl., 10.11.28).—See B.P. 324,471; B., 1930, 379.

Working up complex ores and metallurgical products. F. JOHANNSEN, ASSR. to F. KRUPP GRÜSON-WERK A.-G. (U.S.P. 1,762,867, 10.6.30. Appl., 21.1.27. Ger., 26.1.26).—See B.P. 265,162; B., 1927, 659.

Iron-nickel alloy. F. STÄBLEIN, ASSR. to F. KRUPP A.-G. (U.S.P. 1,760,326, 27.5.30. Appl., 31.10.28. Ger., 24.12.27).—See B.P. 302,994; B., 1929, 561.

Removing, purifying, and recovering oil from tinplate. J. H. HOLDEN, T. O'BRIEN, J. C. WHETZEL, and R. E. ZIMMERMAN, ASSRS. to AMER. SHEET & TIN PLATE CO. (U.S.P. 1,763,430, 10.6.30. Appl., 15.6.26).—See B.P. 283,614; B., 1928, 235.

Separators for ores etc. (B.P. 329,030). Separation of minerals (U.S.P. 1,749,371). Edge-filter members (U.S.P. 1,745,426).—See I. Separating iron from roasted pyrites (B.P. 329,543). Aluminium compounds for production of aluminium (B.P. 307,345).—See VII. Refractories (B.P. 304,729).—See VIII. Purification of hot gases (B.P. 329,962).—See

XI. Degreasing castings etc. (B.P. 329,156).—See XII. Oligodynamic metals for water sterilisation (B.P. 306,547).—See XXIII.

XI.—ELECTROTECHNICS.

Electrolytes for lead accumulators. N. KAMEYAMA and T. TAKASHIO (J. Soc. Chem. Ind., Japan, 1930, 33, 179B).—Measurements have been made of the *E.M.F.* of lead accumulators in which various sulphates were added to the electrolyte in order to increase the *E.M.F.* without causing excessive corrosive action on the plates. The sulphates of lithium, potassium, sodium, magnesium, and zinc were tried. In most cases the *E.M.F.* of the cell containing *m* mols. of sulphuric acid and *n* mols. of the metallic sulphate was greater than that of the cell containing only *m* mols. of sulphuric acid, but less than that of the cell containing (*m*+*n*) mols. of acid.

O. J. WALKER.

Electric welding in gas-works etc. KÜRSCHNER.—See II. Production of alloy steels. DÖRRENBURG and BROGLIO. Electrolytic iron. PIKE and others. Iron printing plates. THOMAS and BLUM. Hydrometallurgy of copper. BAROCH. Electrolytic zinc. TAINTON and BOSQUI; TAINTON and CLAYTON. Zinc on aluminium. COCKS. Bismuth electrolytes. KERN and JONES.—See X. Solidification of fatty oils. HOCK.—See XII. Rubber for electrotechnical work. HEERING.—See XIV. *p_H* of tan liquors. HEFURN.—See XV.

PATENTS.

[Electric] heat-treating furnace. H. O. BREAKER (B.P. 307,301, 4.3.29. U.S., 3.3.28).—An electric resistance furnace for the treatment of goods which may contain occluded moisture is charged and discharged by means of a drop-bottom which is hydraulically raised and wedged into place; a form of sand or other fluid seal is described which in conjunction with a welded metallic casing forms a substantially gastight furnace. Moisture is removed (to avoid oxidation) as soon as it is evolved, by means of a condenser forming an extension of the casing and surrounding the electric leads, the only communication for gases to the interior of the furnace being passages along or near the conductors.

B. M. VENABLES.

Electrical heating of liquids. I. G. FARBERIND. A.-G., Assees. of F. WINKLER (G.P. 461,567, 12.2.26).—The electric resistor comprises a mass of granular conducting material kept in motion by a current of gas which passes through the liquid in which the resistor is immersed. Heating is rapid and no decomposition of the liquid occurs.

A. R. POWELL.

Purification of hot gases. LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 329,962, 27.2.29).—Hot gas, after passing through a heat-exchange cooler, enters an electrical precipitator having an electric field sufficiently long to precipitate successively spray or mist and dust or fume particles, then traverses an irrigating cooler in which remaining traces of dust are wetted and the gas is cooled, and finally passes through one or more electrical precipitators.

J. S. G. THOMAS.

Manufacture of self-baking electrodes. M. O. SEM and C. W. SÖDERBERG, ASSRS. to NORSKE AKTIESELSKAB FOR ELECTROKEM. IND. OF NORWAY (U.S.P. 1,751,177, 18.3.30. Appl., 6.12.28. Ger., 26.9.28).—Raw electrode paste containing a relatively high proportion of binder, *e.g.*, tar or pitch, is filled into the top end of an enclosing mantle, *e.g.*, of sheet iron, and heated to 100–200°, whereby it is caused to flow and form a solid electrode. Alternatively, the paste may be rammed or pressed after heating. J. S. G. THOMAS.

Deoxidising system [for transformers]. C. A. STYER, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,745,875, 4.2.30. Appl., 5.4.28).—Air above the oil in a transformer casing is maintained substantially free from oxygen by attaching this apparatus to the casing and allowing the air to circulate through it by convection. The apparatus comprises a block of carbon resting on a metallic resistor which is maintained by electric currents at a temperature above the ignition point of the carbon. The heat produced is negligible compared to the losses in the transformer, and when oxygen arrives, due to the breathing of the transformer, the heat in the deoxidiser increases owing to combustion, which thereby increases the convection currents; to that extent the apparatus is self-regulating.

B. M. VENABLES.

(A, E) Asymmetric electric couples. (B–D) Electric-current rectifiers. S. RUBEN, ASSR. to RUBEN RECTIFIER CORP. (U.S.P. 1,751,359, 1,751,360, 1,751,362—3, and 1,751,460, 18.3.30. Appl., [A] 20.8.25, [B] 29.4.26, [C] 17.6.26, [D] 20.12.29, [E] 16.11.25).—(A) An asymmetric rectifying couple comprises an electropositive electrode of magnesium and an electronegative electrode formed of an alloy of copper with an element of the oxygen series of group VI in such proportions that, when current is passed, an inverse current-blocking film is formed and maintained at the junction of the electrode elements. (B) An electropositive metal, *e.g.*, aluminium, is subjected to the action of a solution of sodium sulphide at about 100° and maintained at a positive electrical potential with respect to the solution until the current flowing attains approximately a minimum value, whereby the aluminium becomes coated with a dense homogeneous layer containing sulphide. (C) A rectifier comprising electropositive and electronegative electrodes composed, respectively, of magnesium and of a fused compound of the oxygen family of group VI having a base of copper, and provided with external and internal, independent, electric heating devices is claimed. Other rectifiers described comprise the following electropositive and electronegative electrodes, respectively: in (D) magnesium; a compound of copper and sulphur; and in (E) one of the lighter elements of groups II and III; a compound of an element of the oxygen family of group VI, *e.g.*, sulphur or selenium, with an alloy of copper with zinc, silver, lead, tin, or antimony.

J. S. G. THOMAS.

Manufacture of oxide cathodes for discharge tubes. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 311,705, 25.4.29. Austr., 14.5.28).—An alkaline-earth metal vapour and oxygen simultaneously pro-

duced within the tube, under conditions which prevent direct oxidation of the cathode core-wire, combine to form a deposit of alkaline-earth oxide on the cathode, which is then heated above its working temperature so that the oxide layer adheres firmly.

J. S. G. THOMAS.

Magnetic insulating materials. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (B.P. 308,224, 19.3.29. Fr., 19.3.28).—Homogeneous sheets of textile material, impregnated with a mixture of synthetic or natural resin and filings or fine powder of magnetic material, are piled and consolidated under pressure and heat. [Stat. ref.]

J. S. G. THOMAS.

Electrical insulating materials [comprising rubber]. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HABGOOD, and H. C. CHANNON (B.P. 329,275, 11.2.29).—Unmasticated raw rubber is heated over the range 50–250° in a vacuum or an inert fluid; it is then dried, if necessary, and then treated with a solvent such as benzene or petroleum spirit. The dissolved fraction, after removal of the insoluble residue, is substantially free from non-rubber substances. The heat treatment results in a greater yield of dissolved rubber than is obtainable from unheated, unworked material. The product has superior electrical properties, but it is mechanically weak, and is preferably strengthened by mixing with gutta-percha, balata, or bitumen of high m.p., but low content of mineral matter and of free carbon.

D. F. TWISS.

Electrical insulating materials [comprising rubber]. W. S. SMITH, H. J. GARNETT, H. C. CHANNON, and J. N. DEAN (B.P. 329,981, 28.2.29. Addn. to B.P. 307,390; B., 1929, 400).—Ordinary rubber (20–50%) is mixed with the dirt- and resin-free gutta-percha and/or balata claimed in the prior patent. J. S. G. THOMAS.

Electrical insulating masses for the construction of separation walls. I. G. FARBENIND. A.-G., ASSEES. OF C. THODE and A. BENISCHEK (G.P. 461,493, 7.11.26).—Asbestos, slag wool, or other inorganic fibrous material is mixed with fillers, *e.g.* magnesia or alumina, and nitrocellulose, with or without the addition of a plasticiser, *e.g.*, a phenol ester of phosphoric acid.

A. R. POWELL.

Apparatus for electrometric analysis. M. TRÉNEL (G.P. 462,754, 15.9.25).—The comparison liquid is separated from the solution being titrated by a porous diaphragm saturated with quinhydrone or other reagent capable of evolving hydrogen. In this way continual evolution of hydrogen at the electrodes may be avoided, and no reducing reagent need be added to the electrolyte.

A. R. POWELL.

Magnetic-core induction furnace. P. E. BUNET, ASSR. to SOC. ACIÈRES DE GENNEVILLIERS (U.S.P. 1,760,741, 27.5.30. Appl., 25.5.28. Fr., 2.6.27).—See B.P. 291,455; B., 1929, 823.

[Electric induction] apparatus for treatment of gases at high temperatures. M. FOURMENT (U.S.P. 1,763,229, 10.6.30. Appl., 28.10.25. Fr., 22.12.24).—See F.P. 593,298; B., 1926, 145.

Method of arc-welding. G. A. WHITING and R. A. HOLLAND, ASSRS. to E.M.F. ELECTRIC CO. PROPRIETARY,

LTD. (U.S.P. 1,760,534, 27.5.30. Appl., 23.6.27. Austral., 9.7.26).—See B.P. 296,509; B., 1928, 790.

Glow-discharge lamps. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 314,898, 3.7.29. Ger., 4.7.28).

Manufacture of light-emitting elements for electric lamps. A. SCHMID, and METROPOLE DEVELOPMENTS, LTD. (B.P. 327,951, 12.12.28).

[Zinc container for] electric dry cells. SIEMENS BROTHERS & Co., LTD. FROM GEN. DRY BATTERIES, INC. (B.P. 330,165, 8.7.29).

[Inert-type] galvanic batteries. L. and G. J. A. FULLER (B.P. 330,444, 26.7.29).

X-Ray apparatus [and safety devices therefor]. E. J. WARD-WATKINSON (B.P. 330,318, 14.3.29).

[Semi-portable] X-ray apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of VICTOR X-RAY CORP. (B.P. 329,978, 28.2.29).

[Bulbs for] incandescence electric lamps. J. L. PECH (B.P. 305,227, 1.2.29. Fr., 3.2.28).

Hygrometer (U.S.P. 1,749,826).—See I. **Lubricating oils** (U.S.P. 1,746,641). **Reclaiming insulating oil** (U.S.P. 1,752,238).—See II. **Conversion of fibres into threads resembling wool** (B.P. 329,792).—See VI. **Separation of phosphorus vapours** (G.P. 462,521).—See VII. **Ferrochromium** (G.P. 461,945). **Insulating coating on aluminium alloys** (U.S.P. 1,751,213). **Gold-beryllium alloys** (B.P. 313,527). **Alloys for radio-valve filaments** (B.P. 317,785). **Annealing of metal bands** (B.P. 305,951 and 312,988). **Easily soldered metal coatings** (B.P. 329,346). **Electrolytic iron** (U.S.P. 1,751,099). **Copper-nickel alloys** (U.S.P. 1,750,092). **Chromium-plating bath** (B.P. 310,427). **Chromium coatings** (G.P. 461,987 and 462,275). **Heavy metals** (B.P. 329,324). **Adherent lead deposits** (B.P. 330,040). **Alkali metals** (G.P. 461,694).—See X. **Insulating materials etc.** (B.P. 329,694).—See XIII. **Separation of rubber from latex** (B.P. 307,830 and 309,947). **Electrophoretic deposition of solids from dispersions** (B.P. 305,630 and 307,912). **Preparation of rubber goods** (B.P. 328,980). **Porous body** (B.P. 306,129).—See XIV. **Treatment of seaweed** (F.P. 633,696).—See XIX.

XII.—FATS; OILS; WAXES.

Metallic soaps—their uses, preparation, and properties. W. F. WHITMORE and M. LAURO (Ind. Eng. Chem., 1930, 22, 646—649).—Heavy-metal salts of fatty acids are prepared, usually from an alkali salt of the fatty acid and the metal acetate or nitrate, in aqueous-alcoholic solution at 0° or 60°. The following are described (m.p. in brackets): Oleates: silver, mercury, lead (about 50°), zinc (about 70°), copper (fuses below 100°), and nickel (about 18—20°); stearates: silver, mercury (112.2°), lead (115°), zinc (about 130°), copper (about 125°), and nickel (about 100°); palmitates: silver, mercury (above 105°), lead (113°), zinc (129°), copper (120°), and nickel (about 80°); erucates: silver, mercury (below 50°), lead (about 100°), zinc (94°),

copper (about 100°), and nickel (about 35°); laurates: silver, mercury (106°), zinc (128°), copper (111—113°), and nickel (44°). All the above silver salts have m.p. above 200° (decomp.). A brief *résumé* of the uses of the heavy-metal salts is given. H. BURTON.

Solidification of fatty oils by silent electric discharges ("Voltolfactis"). L. HOCK (Kautschuk, 1930, 6, 142—143).—Removal of unchanged oil from the gelatinous flocks obtained by submitting fatty oils in an atmosphere of hydrogen to the silent discharge yields a bulky, yellowish-white mass resembling white factis. It is possible to vary the character of the product by modifying the treatment. Röntgen diagrams reveal no indication of evidence of fibrous structure, but the weakness of the material prevents considerable extension. Use of hydrogen sulphide instead of hydrogen as atmosphere leads to a sulphurated "voltolfactis" of dark colour and unpleasant odour, and it would be preferable to defer introduction of sulphur as such or as hydrogen sulphide until nearly the end of the electrical treatment. The mol. wt. of "voltoloi" and also its heat of dissolution vary considerably with different solvents. D. F. TWISS.

Determination of unsaturation of fats and fatty acids. II. J. VAN LOON (Chem. Umschau, 1930, 37, 135—137; cf. B., 1929, 567).—Methods for the determination of iodine value are discussed. The Wijs method has been tested under varying conditions on a linseed oil. When a solution containing a small excess (3%) of iodine (no free chlorine) was used, no substitution occurred, constant values being obtained in from 2½ hrs. to 7 days. The iodine value varied slightly with the halogen excess used, not on account of substitution, but because the reaction proceeds to an equilibrium; with 150% excess of halogen the true iodine value (cf. hydrogen value) may be obtained in 2½ hrs. E. LEWKOWITSCH.

Composition of tung oil. J. VAN LOON (Farben-Ztg., 1930, 35, 1767—1769).—On the assumption that elæostearic acid (the glyceride of which constitutes 70—80% of tung oil) is an isomeride of linolenic acid, the values (150—170) quoted in the literature for the iodine value of tung oil are too low. It is shown that, under usual conditions, this oil gives an apparent iodine value only, considerable excess of iodine monochloride and a reaction period of at least 7 days being necessary for the attainment of the "true iodine value." By this method values of 217, 274, and 232 were obtained as the iodine values of tung oil, elæostearic acid, and tung oil total fatty acids, respectively, thus confirming the postulated degree of unsaturation of elæostearic acid. On the basis of these figures and earlier information, the following composition is given for tung oil: glycerin residue 4.7%, unsaponifiable matter 0.5%, volatile matter 3.4%, saturated acids 4.9%, Δ⁹-oleic acid 13.6%, and elæostearic acid 72.8%. S. S. WOOLF.

Production of pure ricinoleic glyceride. P. PANJUTIN and M. RAPOPORT (Chem. Umschau, 1930, 37, 130—135).—Attempts were made to isolate pure ricinoleic glyceride by repeatedly extracting castor oil with light petroleum (containing 10% of aromatic

hydrocarbons) in order to remove other constituents: analyses were made after each extraction of the residual oil, the extract, and the fatty acids from each fraction. Oleic, linoleic, and stearic acids disappeared almost completely after three extractions, and dihydroxystearic acid, occurring mainly as the free acid, was removed by an alkaline wash. The unsaponifiable matter (1.8%) consisted largely of glycerin, which is not removed by the solvent. A ricinoleic glyceride was thus obtained containing only 0.5–1% of other substances. E. LEWKOWITSCH.

Hydrolysis in aqueous dispersions of sulphonated oils. W. SCHINDLER and R. SCHACHERL (Collegium, 1930, 108–116).—When dispersions of various sulphonated oils at different p_H values, respectively, were boiled, sulphuric acid was, in most cases, found to be split off at p_H values < 4.0 and in strongly alkaline solutions. There was practically no hydrolysis between p_H 4.0 and 7.0. The hydrolysis of a highly sulphonated castor oil was small when boiled for 30 min. at p_H 4.2 and diminished to zero at p_H 6.8.

D. WOODROFFE.

Examination of oils in ultra-violet light. J. A. PIERCE (Z. Unters. Lebensm., 1930, 59, 94–99).—The fluorescence phenomena exhibited under ultra-violet radiation by 48 different types of oil, principally crude and refined olive oils, have been studied under standardised conditions. Refined olive oil always gave a blue fluorescence and the crude oil a yellow, thus establishing a means for their differentiation. The filtration of cod-liver oil through fuller's earth produces a change in the fluorescence from yellow to blue. This change cannot be ascribed to the change in the colour of the oil, since by addition of carotene and chlorophyll the yellow fluorescence is not restored, although it is restored when refined oil is irradiated with ultra-violet light. This phenomenon and the observation that irradiation of unrefined olive oil produces an intensification of the yellow fluorescence support van Raalte's theory that fluorescence phenomena are attributable to the presence of vitamins. Bleaching by exposure to sunlight, or by heating for 3 hrs. at 150° , produced no change in the fluorescence, thus proving that the colour of the oil bears no relationship to the fluorescent appearance. H. J. DOWDEN.

Submerged combustion. HAMMOND.—See II. **Sulpholeates in tanning.** GERONAZZO.—See XV. **Xylene value of butter.** HANKE and DEUTSCHMANN. **Soya-bean residues.** MALKOMESIU and SCHRAMM.—See XIX. **Oil from cascara sagrada.** JERMSTAD.—See XX.

PATENTS.

Preparation of lyophile products of fatty substances. H. SANDQVIST and T. O. HULTBERG (B.P. 329,305, 14.2.29).—Relatively pure phytosterol (0.1–10% by wt.) is added to mineral or fatty oils, petroleum jelly, etc. to form lyophile salve bases, compound lubricants, etc.; the phytosterol may be oxidised in the warm by air before or during admixture. E. LEWKOWITSCH.

Preparation of fine granules of soap. C. G. PALMOLIVE—LEET CO. (B.P. 314,432, 13.2.29). U.S.

28.6.28).—Hot, semi-fluid soap is mixed with air under pressure and beaten to a froth in an agitator; the mixture so formed is passed through a shredder and discharged as small granules in a semi-liquid condition into a hot-air drying chamber. E. LEWKOWITSCH.

Manufacture of water-soluble products from commercial fatty acids obtained from raw wool fat. I. G. FARBERIND. A.-G. (B.P. 305,597, 7.2.29. Ger., 7.2.28. Addn. to B.P. 307,776; B., 1929, 442).—The fatty acids are treated with oxygen or air at 100 – 300° (with or without a catalyst such as cobalt resinate) prior to the sulphonation treatment described in the prior patent. E. LEWKOWITSCH.

Degreasing of articles [castings etc.]. CARRIER ENG. CO., LTD., and J. R. ROBERTSON (B.P. 329,156, 10.5.29).—The articles are submitted first to the action of a vapour grease-solvent, e.g., trichloroethylene, which condenses on them and effects removal of soluble matter, and subsequently to sprays of liquid solvent under pressure; a bath open to the air may be used if provided with a suitable canopy or cooled zone to limit the height of the vapour bath. Suitable apparatus, embodying conveyors, stills for regeneration of clean solvent, etc. is described. E. LEWKOWITSCH.

Production of emulsions. A. L. MOND. From T. GOLDSCHMIDT A.-G. (B.P. 329,266, 5.11.28).—The higher fatty acid esters of polyhydric alcohols in which the alcoholic radical still contains unesterified hydroxyl groups, e.g., glyceryl distearate, form stable aqueous emulsions in the presence of caustic alkalis or soaps. By the use of these esters aqueous emulsions of fats, oils, waxes, latex, zinc oxide, etc. are produced, suitable for pharmaceutical preparations, lubricants, wetting agents, wool-oiling, etc. E. LEWKOWITSCH.

Treatment of tung oil and mixtures containing the same. IMPERIAL CHEM. INDUSTRIES, LTD., and H. MORGAN, and A. A. DRUMMOND (B.P. 329,685, 17.12.28).—After heating a solution of raw tung oil in a volatile organic solvent, e.g., cyclohexanol, tetrahydro-naphthalene, for several hours at about 150° , a small amount of a dehydrating agent (e.g., zinc or barium chloride, sulphuric acid, preferably dissolved in a volatile solvent) is added. The product gives a clear dry film on standing at steam temperatures. Linseed oil, resins, etc. may be added before the treatment. E. LEWKOWITSCH.

Pollack for woodwork, linoleum, etc. H. H. BODEN (B.P. 329,909, 9.8.29).—A solution of beeswax in neat's-foot oil with added turpentine oil and/or paraffin oil of desired consistency.

Centrifugal separator (B.P. 329,191, 24.10.28). See VII. **Paint from castor oil** (B.P. 326,453). See XIII. **Adhesives for clatching linoleum** (B.P. 326,906). See XIX. **Vitamin A** (B.P. 326,831). See XX.

PAINTS, PIGMENTS, VARNISHES, RESINS. **Theory of molecular attraction and its application to paints.** D. H. S. FORBES (J. Oil Col. Chem. Assoc. 1930, 13, 110–119).—The theory of molecular attractions in dispersed systems, for which the term "dispersion" is used, is discussed in connection with paint and varnish systems. The theory is applied to the study of the stability of paint and varnish systems. E. LEWKOWITSCH.

Use of ethylene dichloride in lacquer formulation. R. B. FRAZIER and E. W. REID (Ind. Eng. Chem., 1930, 22, 604—608).—Determinations of the solubility of nitrocellulose and cellulose acetate in various three-component solvent mixtures (one component being ethylene dichloride) show that nitrocellulose is practically completely soluble in a mixture of ethylene dichloride (80%) and methyl or ethyl alcohol (20%); a very small amount of an active solvent is added to the mixture to effect complete dissolution. A mixture of the dichloride (70—80%) and alcohol (20—30%) has the highest solvent power for cellulose acetate. The dichloride and the 80:20-mixture are also good solvents for the usual gums and resins. The dichloride is exceptionally stable in lacquers, showing no tendency to hydrolysis.

H. BURTON.

Practical evaluation of nitrocellulose lacquers.

F. ZIMMER (Farben-Ztg., 1930, 35, 1766—1767).—Simple tests on raw materials and finished products, e.g., absence of water or acid, viscosity, sp. gr., general spraying properties, rate of drying of film and its behaviour to finger-nail scratching, are briefly summarised.

S. S. WOOLF.

Formation of synthetic resins. R. H. KIENLE (Ind. Eng. Chem., 1930, 22, 590—594).—Synthetic resins and highly polymerised compounds are classified (cf. B., 1929, 444) as follows: (1) heat-convertible (polyolefines, phenol-aldehydes), (2) heat-non-convertible (styrene, acidic type of phenol-aldehydes), and (3) element-convertible (glycerol polyunsaturated esters). From a survey of the literature and a study of the varying reactivity of alcohols and acids in the formation of polymeric esters and "alkyd" resins (*loc. cit.*), three postulates are advanced: (a) high-molecular compounds are formed only when the interacting molecules are poly-reactive, i.e., the molecules must be able to combine with one another through more than one primary valency linking; (b) the combination of molecules is determined by the probability of contact of mutually reactive, primary valency linkings; and (c) molecular size and shape, and the position of the reactive centres determine the physical properties of the resulting polymeride. A practical application of these postulates is illustrated by a study of the reactions between phenolphthalein, formaldehyde, and glycerol.

H. BURTON.

Rinmann's green and rubber. DITMAR and PREUSSE.—See XIV.

PATENTS.

Coating compositions, plastic compositions, etc. containing cellulose ethers. BRIT. CELANESE, LTD. (B.P. 313,133, 7.6.29. U.S., 7.6.28).—Compositions containing aralkyl ethers of cellulose, e.g., benzyl cellulose, one or more synthetic resins, e.g., resins of the phenol-aldehyde, glyptal, zinc butyl phthalate, polymerised lactic acid, or coumarone type, and, if desired, solvents, softeners, pigments, dyes, etc. are claimed.

S. S. WOOLF.

Coating composition [for painting cement etc.]. J. V. REARDON, ASST. to REARDON Co. (U.S.P. 1,749,923, 11.3.30. Appl., 9.11.26).—A mixture of approx. equal parts of a hydraulic cement and slaked lime, together with relatively small proportions of a hygroscopic salt

(sodium chloride), a sugar, and a water-repelling soap (e.g., calcium or aluminium stearate) is worked up with water to a paste suitable for brushing or spraying.

L. A. COLES.

Manufacture of dope material for producing non-transparent coatings. I. G. FARBENIND. A.-G. (B.P. 302,280, 7.12.28. Ger., 13.12.27).—A cellulose ester, e.g., nitrocellulose, a gelatinising agent, e.g., camphor, a substantial proportion of oil, e.g., castor oil (preferably at least equal to the quantity of cellulose ester), colouring materials, fillers, etc. are thoroughly subdivided and mixed by working together, e.g., on rollers under pressure, in the presence of a volatile solvent, e.g., alcohol, the temperature being such that a large proportion of the solvent is evaporated during the operation, but without bringing the colouring matter into the colloidal condition. The material produced is capable of withstanding pressures of 200 atm. without separation of its constituents, contains a small proportion of low-boiling solvent, and is soluble in such solvents to form a lustrous varnish.

S. S. WOOLF.

Impregnation of articles of wood, tissue, paper, cardboard, etc. with varnishes of synthetic resins. MANUF. DE MACHINES AUXILIAIRES POUR L'ELECTR. ET L'IND., and D. A. L. TEXIER (B.P. 329,066, 6.3.29).—The stages of solvent-extraction and resin-polymerisation in the customary synthetic-resin varnish-impregnation processes are carried out in a gaseous atmosphere of high sp. heat, e.g., in dry steam, capable of absorbing or of carrying along solvents and of being condensed at ordinary temperatures. Suitable apparatus is described and the process is detailed. The use of relatively low temperatures and the complete recovery of solvents and excess synthetic resin components are claimed.

S. S. WOOLF.

Paint, varnish, and enamel, and their production. W. F. BLEECKER and E. A. CLARK (U.S.P. 1,750,087, 11.3.30. Appl., 12.11.23).—Rubber, sulphur, and an oil, preferably lard oil, although vegetable or mineral oil will suffice, are heated together to above 177° to form a composition applicable as a paint or varnish or useful for impregnating porous substances to withstand chemical corrosion. Disintegrated vulcanised rubber can be used to supply both rubber and sulphur, a mixture with one ninth of its weight of oil being well adapted for the purpose. Asphaltum, fillers, pigments, and thinners may be incorporated.

D. F. TWISS.

Production of painting materials from castor oil. J. SCHEIBER (B.P. 306,452, 9.2.29. Ger., 20.2.28).—Ricinoleic acid (castor oil fatty acids) is heated *in vacuo* at about 200° (with or without catalysts such as alumina, silica gel), whereby water is split off; the isomeric linoleic acid with conjugated double linkings which is formed is subsequently esterified with glycerol or other polyhydric alcohol by heating. The esters so produced can be used for varnishes etc. which dry to brilliant films highly indifferent to the action of water.

E. LEWKOWITSCH.

Manufacture of lithopone or zinc sulphide resistant to light. E. MAASS and R. KEMPF (G.P. 462,372, 26.8.24).—The pigment prepared by any of the usual precipitation processes is mixed, while still wet, with a

small quantity of a soluble thiosulphate, polythionate, sulphite, or hyposulphite and an ammonium salt, or with a soluble, readily dissociated metal sulphide, and the mixture is dried. The mass so obtained is ground and leached with water until both wash water and pigment react neutral to phenolphthalein. The pigment is again dried and calcined in the absence of air.

A. R. POWELL.

Manufactures of lacquers, coating materials, insulating and impregnating materials, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 329,694, 23.2.29).—The residues from the distillation of the condensation products of olefines (cf. B.P. 291,137 and 309,199; B., 1928, 560; 1929, 424) are dissolved in volatile organic solvents, with or without the addition of resins, rubber, etc.

E. LEWKOWITSCH.

Production of rapidly hardening, odourless, phenol-aldehyde resins and mixtures of these with fillers. BAKELITE GES.M.B.H. (B.P. 305,237, 2.2.29, Ger., 3.2.28).—Metal oxides, hydroxides, or salts, e.g., lime, magnesia, zinc hydroxide, calcium stearate, in quantity not much in excess of that necessary for complete binding of free phenol and uncombined by-products, are added to fusible, water-insoluble, resinous phenol-aldehyde condensation products. The time of hardening of the resin is shortened and the thermal and electrical properties of the finished product are improved.

S. S. WOOLF.

Moulding mixtures. H. WADE. From BAKELITE CORP. (B.P. 329,628, 18.2.29).—A urea-formaldehyde resin is produced in the presence of fillers including a protein substance of the type of casein and a polyhydric alcohol, e.g., glycol. Cellulose esters, plasticisers, etc. may be added as desired.

E. LEWKOWITSCH.

Manufacture of resinous plastic material [by dry process]. A. V. KELLER (B.P. 328,938, 2.1.29).—Dry urea, thiourea, or derivatives thereof are caused to react with solid anhydrous polymerides of formaldehyde, e.g., paraformaldehyde, so as to obviate the presence of free formaldehyde in the final condensation stage. A neutral salt, e.g., sodium chloride, may be present as catalyst for the initial condensation, and such salts or solid organic acids may be added as accelerators for the final conversion into the insoluble form under heat and pressure.

S. S. WOOLF.

Manufacture of condensation products of urea and aldehyde. K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,762,456, 10.6.30. Appl., 27.9.24. Austr., 25.8.24).—See B.P. 238,904; B., 1925, 891.

Materials in sheet form (B.P. 329,691). **Air cleaner** (U.S.P. 1,746,774).—See I. **Carbon black** (B.P. 327,979).—See II. **Esters of carbohydrates** (B.P. 305,661).—See III. **Dressing of textiles** (B.P. 320,018).—See VI. **Coatings on aluminium alloys** (U.S.P. 1,751,213).—See X. **Foils from rubber-like substances** (B.P. 329,000). **Chlorination of rubber** (B.P. 328,818).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Structure of rubber; swelling experiments in various liquids. P. BARY (Kautschuk, 1930, 6, 121—126).—Rubber probably consists of an intimate mixture

of different polymerides of isoprene, the lowest degree of polymerisation present being dependent on the temperature and pre-history of the sample. The polymerides can be separated into two groups by their relative diffusibility, and unvulcanised rubber consists of a solution of the more easily diffusible polymerides in those of lower diffusibility. Harries' conception of the stability of the rubber molecule is out of accord with recent investigations, and the theory of true solutions is not applicable to jellies such as rubber gives with solvents. If a piece of vulcanised rubber, swollen with a liquid such as benzene, is transferred to the vapour space above the liquid, it shrinks gradually to the volume it would have attained by direct swelling in the vapour. This is not due to greater vapour tension in the jelly than in the pure liquid, but arises owing to liberation of heat by syneresis causing a temperature difference which actuates evaporation of the solvent from the jelly. The swelling of rubber is a two-fold phenomenon comprising entry of the solvent by diffusion and subsequent solvation which accounts for its alteration in character from exothermic initially to endothermic later. Swelling of rubber, whether vulcanised or not, in a liquid such as toluene, is accompanied by a contraction of the system during the earlier stages, corresponding presumably with the exothermic period; the subsequent endothermic course of the swelling is probably associated with the solvation process.

D. F. TWISS.

Viscosity of rubber solutions. C. M. BLOW (Trans. Inst. Rubber Ind., 1930, 5, 417—425).—Solutions of over-masticated rubber show small structural viscosity, but introduction of carbon black into the rubber before dissolving causes an increase in viscosity with development of structure viscosity and thixotropy which become more marked on storage. This effect is attributable to orientation of adsorbed rubber molecules on the carbon particles. The rubber which diffuses out of such mixtures as swell to a jelly has the original viscosity. It is believed that the viscosity of rubber solutions bears no relation to the mol. wt. of the rubber and that mastication does not effect real depolymerisation, changes in physical structure being responsible for the phenomena.

D. F. TWISS.

Viscosity of rubber solutions. C. M. BLOW (Kautschuk, 1930, 6, 126—129; cf. preceding abstract).—In addition to the previous investigation, experiments on the viscosity of rubber after various periods of mastication and subsequent storage are reported. Any explanation of the effect of mastication as solely the result of depolymerisation induced by the action of oxygen would be difficult to reconcile with the above results.

D. F. TWISS.

Sun-cracking of vulcanised rubber. B. B. EVANS (Trans. Inst. Rubber Ind., 1930, 5, 442—450; India-rubber J., 1930, 79, 853—854).—Using mixtures containing (a) rubber (100 vols.), sulphur, and filler (20 vols.); (b) as in (a), but coloured with carbon black; and (c) similar to (a), but with vulcanisation aided by diphenylguanidine, and exposing vulcanised samples, bent double, (i) in the open facing south, (ii) indoors near a south window, and (iii) to a mercury-vapour lamp, it is

found that zinc oxide is of little advantage and mixtures containing it are inferior to those containing clay or magnesium carbonate in resistance to sun-cracking. The addition of 1% of carbon black is found generally to cause increased tendency to sun-cracking, the results of Shepard, Krall, and Morris (B., 1926, 598) being thereby confirmed; the divergence of other observations on this point may arise from variation in the character of the carbon black used. Cracking tends generally to occur at right angles to the direction of mechanical strain, calender grain being of minor influence. The organic accelerator appears to have little effect on the cracking behaviour, but smoothness of surface is beneficial to the resistance of the sample. Exposure to the mercury-vapour lamp gives results qualitatively in line with natural exposure, but with less marked differences.

D. F. TWISS.

Evolution of hydrogen sulphide from vulcanised rubber. E. WOLESENSKY (Bur. Stand. J. Res., 1930, 4, 501–513).—Samples of vulcanised rubber were heated at various temperatures in a current of hydrogen and the hydrogen sulphide evolved was determined as lead sulphide. In one case a sample heated for 5 hrs. at 285° yielded 8.4% of its total sulphur content in this form. At 25° hydrogen sulphide could be qualitatively identified, and if the temperature of destructive distillation was reached (about 300°) the yield exceeded 50%. Removal of free sulphur, or of non-rubber constituents, did not affect the result, nor is the presence of moisture in the nitrogen necessary. The reaction affords an additional explanation of the formation of hydrogen sulphide during vulcanisation and may be a factor in ageing.

C. IRWIN.

Rinmann's green in accelerated rubber mixtures. R. DITMAR and K. H. PREUSSE (Gummi-Ztg., 1930, 44, 1842).—Rinmann's green maintains its colour during vulcanisation in the presence of accelerators of the dithiocarbamate or thiuram disulphide class, but loses its colour with most other organic accelerators; it activates the accelerator and effects a stiffening of the vulcanite.

D. F. TWISS.

Rubber as material for electrotechnical work. H. HEERING (Kautschuk, 1930, 6, 129–136).—The knowledge of the dielectric characteristics of rubber covering, e.g., the influence of vulcanisation, compounding, moisture, and pressure on the electrical resistance, insulating power, power factor, and dielectric strength, is reviewed. The use of a layer of unvulcanised rubber between compounded rubber insulation and a metal conductor, which is frequent in England, is incidentally criticised as ineffective. (Cf. also Curtis and co-workers, B., 1926, 503; 1927, 916).

D. F. TWISS.

Rubber lining in the chemical industry. J. COURBIER and T. BOUSSAND (Chim. et Ind., 1930, 23, 1099–1100).—The method of producing an adherent rubber or ebonite lining on iron or steel is described and the limitations of dimensions of pipes etc. to which a satisfactory lining can be applied are mentioned. A table gives the limits of concentration and temperature within which resistance to different reagents can be assured. Soft rubber linings have a high resistance to abrasion, and

are used on the interiors of tube-mills, breakers, etc.

C. IRWIN.

Antimony pentasulphide. SCHÜRMAN and BÖHM.—See VII.

PATENTS.

Separation of rubber from latex. SIEMENS-ELEKTRO-OSMOSE GES.M.B.H. (B.P. 307,830, 2.3.29. Ger., 14.3.28).—Latex is mixed with a liquid, such as glycerin, of low electrical conductivity so that it is possible to use continuous high-tension current, e.g., at above 250 volts, to effect deposition of the rubber on an electrode or on a porous mould in front of the electrode. If desired, compounds yielding multivalent cations may be added, such as lead, prior to the introduction of the poorly conducting liquid, to deposition at the cathode instead of at the anode.

D. F. TWISS.

Electrical separation of rubber from rubber latex. SIEMENS & HALSKE A.-G. (B.P. 309,947, 4.3.29. Ger., 18.4.28. Addn. to B.P. 307,747; B., 1930, 471).—In the process of the prior patent the support (either the electrode or the adjacent porous mould), bearing its rubber deposit, is dipped into a weak acid solution immediately after removal from the latex. This treatment increases the firmness of the deposit and facilitates its removal from the support.

D. F. TWISS.

Electrophoretic deposition of solids from dispersions, e.g., caoutchouc from latex. SIEMENS-ELEKTRO-OSMOSE GES.M.B.H. (B.P. 305,630 and Addn. B.P. 307,912, 5.2.29. Ger., [A] 8.2.28, [B] 17.3.28).—(A) In the ordinary anodic deposition of rubber or yeast from aqueous dispersions by electrophoresis, the concurrent anodic liberation of oxygen or of acids may be disadvantageous. This feature is avoided by the addition of inorganic salt solutions containing multivalent cations, e.g., thorium nitrate, or of positively charged colloids, e.g., bovine serum, so that the originally negatively charged particles alter the direction of their migration and are deposited on the cathode or on a porous cathodic diaphragm of non-conducting material. It is advisable first to adjust the dispersion to p_H 7. (B) In connexion with the preceding process the p_H of a dispersion such as latex is adjusted to a value 7 by initially bringing it into contact with a gas or gaseous mixture of feebly acid reaction, e.g., carbon dioxide, preferably after the addition of a protective colloid such as globulin.

D. F. TWISS.

Preparation of goods of rubber or similar material from rubber latex or the like. DUNLOP RUBBER CO., LTD., R. F. MCKAY, and E. W. MADGE (B.P. 328,980, 9.11.28).—Homogeneous, non-porous, anodic rubber deposits are obtained by immersing two dissimilar electrodes, e.g., a zinc anode and a depolarised carbon cathode, in natural, preserved, or artificial latex, which may have been concentrated and/or compounded, without the independent addition of electrolytes.

D. F. TWISS.

Manufacture of rubber articles. MORGAN & WRIGHT, Assees. of W. A. GIBBONS (B.P. 307,790, 7.1.29. U.S., 13.3.28).—A hollow, annular form comprising a number of sections, one of which is relatively short

and one at least of which has a conduit communicating with the interior and also with sources of fluid pressure, has several small, evenly distributed perforations over its surface. The form, covered with a filtering material, *e.g.*, fibre coated with clay, is immersed in rubber latex, filtration being effected by evacuating the interior of the form. The rubber deposit so obtained is dried and vulcanised on the form, the sections of the latter being subsequently removed. D. F. TWISS.

Manufacture of rubber articles. GOODYEAR TIRE & RUBBER Co., Assees. of B. W. ROWLAND (B.P. 329,897, 12.7.29. U.S., 10.11.28).—A rubber suspension, which may have been concentrated and compounded, is formed into a film which is coagulated on one side, *e.g.*, by applying a quantity to a cylindrical surface which has been treated with coagulant such as 40% acetic acid. A form or mandrel which also may have been coated with coagulant, is then rolled across this film, thereby causing the layer of freshly coagulated latex to adhere to it. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER Co., LTD., C. HAYES, E. W. MADGE, and F. C. JENNINGS (B.P. 329,381, 19.2.29).—If an aqueous solution of phenol is added to alkali-preserved latex and the alkalinity of the latter is then reduced, *e.g.*, in the case of ammoniacal latex, by the addition of formaldehyde, the latex retains its mobility for a short time during which it may be formed into any desired shape by moulding, spreading, etc., and then changes into a firm continuous coagulum. Although tannic acid alone will not cause the formation of a continuous coagulum, one third of the phenol can be replaced by tannic acid without affecting the result. For latex of 60% concentration produced by a centrifuging process, the proportion of phenol necessary is about 5% on the rubber, but in the presence of compounding ingredients the proportion is smaller. D. F. TWISS.

Manufacture of rubber-coated articles. DUNLOP RUBBER Co., LTD., and D. F. TWISS (B.P. 328,686, 5.2.29).—Chemical plant, coils, pipes, and perforated or woven sheet are coated with soft or hard rubber by spraying with an aqueous dispersion of rubber or similar material, drying, and vulcanising. [Stat. ref.] D. F. TWISS.

Liner [for rubbered fabrics] and treatment of same. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,749,743, 4.3.30. Appl., 6.4.27).—Liners, for use between the adhesive surfaces of freshly rubbered fabrics, are coated with nitrocellulose which has been treated previously, or is treated subsequently, with certain softening agents such as phthalic esters. Old liners may advantageously receive similar treatment. The softening agent checks hardening, cracking, and peeling of the nitrocellulose film. D. F. TWISS.

Manufacture of foils and the like [from synthetic rubber-like substances]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,000, 5.1.29).—Hard flexible foils, such as films, bands, lacquer coatings, or filaments of great strength, low extensibility, and considerable resistance to water and chemical influences, are obtained from soft films of the desired form prepared

from synthetic polymerisation products of diolefines, especially butadiene, by treating them with oxygen or oxygen-containing gases or with agents supplying oxygen, *e.g.*, peroxides. The films can, if desired, contain additional substances, such as fillers, pigments, and softening agents, and may also have been vulcanised before the described treatment. The process may be accelerated by raising the temperature, *e.g.*, to between 50° and 200°. D. F. TWISS.

Porous body [containing rubber]. H. BECKMANN (B.P. 306,129, 31.1.29. Ger., 17.2.28).—Porous bodies suitable for filter-plates, electrolytic diaphragms, lubricating pads, etc. are made by mixing granular or fibrous substances with latex (sufficient to give approx. 15% of rubber in the finished product) and sulphur. A coagulant is then added, *e.g.*, a magnesium salt or sulphurous acid, so as to form a rubber gel permeating the entire mass. The mass is then moulded and vulcanised while moist to any desired shape. A porous, moulded body is thus obtained in which the grains or fibres are bound together by a highly permeable but chemically resistant material. D. F. TWISS.

Treatment of rubber and products obtained thereby. J. MCGAVACK, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,742,609, 7.1.30. Appl., 20.9.28).—Increased flexing life can be imparted to rubber, *e.g.*, in pneumatic tyres or hose, by the addition, either singly or mixed, of derivatives of thymol, *e.g.*, thymoquinone, nitroso-thymol, aminothymol, or of α - or β -naphthaquinone. Such treatment also improves the flexing qualities of spray-dried rubber to the level of ordinary plantation rubber. D. F. TWISS.

Dyeing of rubber. E. G. CROAKMAN, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,742,757, 7.1.30. Appl., 8.11.27).—Certain basic dyes of the triarylmethane series, *e.g.*, Victoria-blue B base, are normally unable to withstand hot vulcanisation, but in the additional presence of an aromatic nitro-compound, particularly a nitrophenol such as 2:4-dinitrophenol, coloured finished products are obtainable having shades more or less of the same order as the dye or mixture of dyes, before vulcanisation. D. F. TWISS.

Colouring rubber and like materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,761, 28.3.29).—Rubber and similar materials are coloured by mechanically working with an aqueous paste or suspension of a water-insoluble colouring matter (*e.g.*, Brilliant Indigo 4G) containing an added organic substance or substances, such as amyl alcohol, glycerin, glyceryl acetates, triaryl phosphates, mineral oil, etc., which are less volatile than water. It is often advantageous initially to produce a coloured stock containing an excessive proportion of colour, *e.g.*, 10% or more, and then to use this for colouring further quantities. D. F. TWISS.

Rubber composition and method of preserving rubber. P. C. JONES, Assr. to B. F. GOODRICH Co. (U.S.P. 1,741,778, 31.12.29. Appl., 21.1.29).—Tetra-substituted hydrazines, especially tetraaryl hydrazines, such as tetraphenylhydrazine, are effective in retarding the deterioration of rubber with age. D. F. TWISS.

Incorporation of lampblack into rubber and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,812, 13.5.29).—Lampblack is advantageously incorporated into rubber, either natural or synthetic, after first mixing it with a fluid, incompletely polymerised substance, such as butadiene, or a vinyl ester. The proportion of the liquid polymerisable, polymerised material may be 20–500% of the lampblack.

D. F. TWISS.

Manufacture of rubber pastes. I. G. FARBENIND. A.-G. (B.P. 305,490, 4.2.29. Ger., 3.2.28).—Concentrated latex pastes are obtained without evaporation of water by mixing the latex with a water-soluble cellulose ether. On heating, the cellulose ether is precipitated in a gelatinous form, and carries with it the rubber globules. By suitable means, such as clarification, filtration, or centrifuging, the water can then largely be removed from the mixture, whereby a paste is produced containing a high proportion of rubber. Such pastes can be mixed with compounding ingredients and then dried or may be re-diluted. In the case of artificial rubber emulsions such as those obtainable by polymerisation of emulsified butadiene hydrocarbons it is desirable, before the concentration process, wholly or partially to remove the emulsifying agent. The cellulose ether, which in an example quoted for ordinary preserved latex amounted to 10% on the rubber, forms an excellent filler for the latter.

D. F. TWISS.

Vulcanisation of rubber and the like. IMPERIAL CHEM. INDUSTRIES, LTD., S. COFFEY, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 329,456, 4.4.29).—Vulcanisation is accelerated by the *N*:*N*-dithio-derivative of a secondary amine (*e.g.*, dimethylamine, piperidine), which can be produced from the latter by the action of sulphur chloride.

D. F. TWISS.

Rubber or like products [anti-agers]. GOODYEAR TIRE & RUBBER CO., Assees. of A. M. CLIFFORD (B.P. 313,486, 19.4.29. U.S., 12.6.28).—The 2-hydroxy- and 2-anilino-1:4-naphthaquinone 4-anils obtained by the action of aniline on β -naphthaquinone-4-sulphonic acid, as also 2:4-dianilino- α -naphthol, are anti-agers for rubber.

C. HOLLINS.

Chlorination of rubber. M. DESENISS and A. NIELSEN (B.P. 328,818, 16.5.29).—Stable chlorination products of rubber suitable for the manufacture of films, filaments, varnishes, etc. are produced by chlorinating dissolved or dispersed rubber at about 80°, *e.g.*, by passing chlorine into a boiling solution of rubber in carbon tetrachloride under reflux until hydrogen chloride is no longer formed. Vulcanised rubber chlorinated as a dispersion in carbon tetrachloride yields similar products, the whole of its sulphur being eliminated as sulphur chloride. Films prepared from the chlorinated solutions, unlike those from cellulose esters, are practically non-inflammable.

D. F. TWISS.

Manufacture of brake liners and the like. J. E. POLLAK. From RUBBER LATEX RES. CORP. (B.P. 329,021, 11.2.29).—A wire reinforcement of very open structure is filled with a doughy composition of asbestos and rubber latex (or aqueous emulsion of rubber), the mass is dried by vacuum, and the product—a mass much

reduced in volume but still open-textured and containing gelled rubber—is compressed and finally vulcanised. To prevent coagulation of the latex by the asbestos, the latter may be washed with dilute hydrochloric acid or the former stabilised by the addition of blood.

B. M. VENABLES.

Treatment of latex and product obtained thereby. J. MCGAVACK, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,762,729, 10.6.30. Appl., 14.10.27).—See B.P. 298,628; B., 1929, 864.

Composition of matter comprising resins and rubber in aqueous dispersion and their preparation. Treatment of aqueously dispersed colloidal substances [rubber latex]. A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,762,153—4, 10.6.30. Appl., [A] 30.6.26, [B] 4.11.26).—See B.P. 283,686; B., 1928, 309.

Vulcanisation of caoutchouc and product derived therefrom. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,763,619, 10.6.30. Appl., 12.6.28).—See B.P. 297,051; B., 1929, 949.

Antioxidant or age retarder [for rubber]. A. M. CLIFFORD, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,763,579, 10.6.30. Appl., 1.3.28).—See B.P. 307,013; B., 1930, 432.

Painting [rubber] balls and apparatus therefor. DUNLOP RUBBER CO., LTD., J. WRIGHT, and H. TREVASKIS (B.P. 330,365, 27.4.29).

Hardness etc. of non-rigid objects (B.P. 329,629). Materials in sheet form (B.P. 329,691).—See I. Carbon black (B.P. 327,979).—See II. Insulating materials (B.P. 329,275 and 329,981).—See XI. Emulsions (B.P. 329,266).—See XII. Paint, varnish, etc. (U.S.P. 1,750,087).—See XIII.

XV.—LEATHER; GLUE.

South African tanning materials. II. Trees and plants other than the black wattle. C. O. WILLIAMS (Union S. Africa, Dept. Agric., Div. Chem. Series 93, 1930, Sci. Bull. 74, 68 pp.; cf. B., 1929, 405).—The average percentage of tannin in the bark of *Acacia decurrens* (green wattle) increased with the age of the tree, was greatest in the bark near the base of the tree, and decreased regularly the higher up the tree the bark was obtained. The rate of decrease was not so great as with *A. mollissima*. The tannin content, which was less than that of the bark of *A. mollissima* of the same age, varied directly with the thickness of the bark. The colour of the infusion increased with the age of the tree. A much lower yield of bark, approximately the same amount of tannin in trees of the same age, and a deeper-coloured infusion were obtained from *A. decurrens* than from *A. mollissima*. A number of barks of exotic trees were analysed and contained on a 10% moisture basis: tans, soluble non-tans (%), degrees red and yellow, respectively: Green wattle (*A. decurrens*), 23.5–42.1, 10.8; 2.7–6.6, 2.8–9.3; silver wattle (*A. dealbata*), 14.3–28.2, —, 7.3–16.9, 9.1–17.9; golden wattle (*A. pycnantha*), 35.0–43.4, 10.4, 4.4–6.1, 7.0–8.8; Blackwood (*A. melanoxylon*), 6.8–18.1, 10.1, 4.2–7.9, 6.4–11.7; saligna wattle (*A. saligna*), 12.1–

30.3, 8.8—12.2, 1.7—5.2, 2.5—6.3; *A. cyclopis*, 6.7—13.0, 8.1—8.7, 2.8—10.0, 4.6—14.7; *A. longifolia*, 7.6, 10.1, 4.7, 6.6; *Eucalyptus astringens*, 27.5—45.4, 11.6—13.7, 2.9—5.6, 4.9—21.6; *E. sideroxylon*, 7.1—30.1, 6.3—9.1, 3.1—16.6, 5.6—49.0; *E. diversicolor*, 3.6—16.3, —, 2.2—8.1, 3.5—11.0; *E. Smilthii*, 11.8, —, 6.4, 13.3; *E. paniculata*, 8.6, —, 6.9—12.6; *E. cladocalyx*, 8.1, —, 6.2, 16.8. The tannin content of other eucalyptus trees was very low. Other results were: *Pinus halepensis*, 17.9, —, 3.6, 6.8; *P. longifolia*, 16.6, —, 5.2, 14.1; *P. insignis*, 13.2, —, 3.9, 7.1; *P. patula*, 8.6, —, 4.3, 7.0; *Callitris calcarata*, 22.1, —, 7.1, 9.9; *C. rhomboidea*, 15.2, 10.3, 3.3, 5.5; *C. robusta*, 21.9, 7.3, 6.9, 12.2; *Quercus pedunculata*, 9.9, —, 7.3, 17.8; *Casuarina cunninghamiana*, 9.2, —, 5.7, 9.5; *Salix babylonica*, 7.3, —, 9.3, 26.7. The following indigenous trees and plants had: *Acacia karroo*, 16.7, 8.2, 6.7, 8.8; *A. arabica* (pods), 21.5, 24.9, 30, 8.8; bark, 15.3, 8.7, 5.4, 17.1; *A. caffra*, 13.1, 13.8, 7.8, 10.3; *Elephantorrhiza burchelli* (roots), 15.6—17.3, 16.4—23.6, 2.9—11.8, 4.9—38.4; *Schotia speciosa*, 16.3, 10.7, 12.2, 23.1; *Rhus Thunbergii*, 35.0, 17.7, 6.8, 17.7; *R. incana*, 19.2, 7.9, 14.7, 26.8; *R. laevigata*, 18.5, 6.2, 11.4, 18.1; *R. natalensis*, 15.2, 7.3, 8.2, 14.6; *Protorhus longifolia*, 10.2, 4.3, 11.4, 16.9; *Harpephyllum caffrum*, 18.2, 8.7, 9.1, 23.7; *Sclerocarya caffra*, 10.7, 6.5, 15.8, 19.1; Cape sumac (*Osyris compressa*) leaves and bark, 15.7—26.9, 15.4—25.4, 3.7—10.8, 6.9—21.3; *O. abyssinica* (leaves), 21.3, 19.1, 4.1, 9.5; *Rhizophora mucronata*, 33.6, 11.4, 7.7, 17.0; *Bruguiera gymnorrhiza*, 27.9, 10.0, 6.5, 9.5; *Curtisia faginea*, 21.5, 10.3, 15.2, 28.9; *Olinia radiata*, 17.2, 15.4, 3.7, 8.8; *Eugenia Gerrardii*, 16.7, 7.1, 13.0, 24.6; *Elaeodendron croceum*, 16.4, 8.2, 14.9, 22.9; *Pterocelastrus variabilis*, 13.6, 20.6, 5.7, 7.1; *Protea mellifera*, 16.4, 12.3, 5.6, 14.7; *P. grandiflora*, 13.6, 16.9, 4.8, 7.4; *P. caffra*, 12.7, —, 7.7, 11.7; *Brabeium stellatifolium*, 13.3, 9.7, 3.5, 5.2; *Leucadendron argenteum*, 13.2, 7.6, 6.9, 8.8; *Leucospermum conocarpum*, 12.6, 5.2, 5.0, 6.6; *Zizyphus mucronata*, 13.9, 7.3, 6.1, 9.0; *Jatropha zeyheri* (roots), 20.5, 15.6, 9.4, 22.5; *Phyllanthus discoideus*, 9.9, —, 7.2, 19.9; *Myrsine melanophloeos*, 11.6, 5.7, 7.3, 9.7; *Garcinia Gerrardii*, 11.3, 10.2, 4.9, 11.7; *Terminalia sericea* (galls), 10.2, 7.1, 24.5, 42.1; *Baikiea plurijuga*, 19.9, 4.9, 12.5, 17.0; *Brachystigia spicaformis*, 19.6, 9.1, 5.5, 9.4; *Lonchocarpus mossambicensis*, 21.1, 14.2, 19.0, 47.6. The tannin content of the yellow-wood barks was low.

D. WOODROFFE.

Automatic apparatus for extraction of tanning materials for [tannin] analysis. G. PARSY (J. Soc. Leather Trades' Chem., 1930, 14, 117—119).—The tanning material is placed in an inverted, narrow-necked bottle, from which the bottom has been cut and replaced by a two-holed stopper fitted with a delivery tube from a steam generator and a reflux condenser. The neck of the bottle is provided with a stopper and U-tube, to which is attached a siphon and delivery tube drawn out to a fine nozzle. The U-tube is supported by a flat-bottomed porcelain basin. The extraction is commenced with water at 50—60°, and gradually the temperature rises until finally it is at 100°.

D. WOODROFFE.

Apparatus for cooling tannin solutions during filtration for analysis. L. SHEARD (J. Amer. Leather Chem. Assoc., 1930, 25, 248—249).—Two shallow, rectangular troughs are arranged one above the other. The upper one is provided with holes which are fitted with rubber stoppers each carrying a funnel, the stem of which reaches to just above the collecting bottles, which stand in the lower trough. A rubber band is fixed on the lower part of the funnel stem and on it rests a conical-shaped glass or metal cover. The upper trough is fitted with a hinged cover, which may be closed during filtration, and with a water-inlet pipe at one end; at the other end is an outlet pipe connecting with the lower vessel, which is similarly fitted with an exit pipe. This apparatus is used in filtering tannin solutions in accordance with the regulations of the official method of tannin analysis, except that the temperature is regulated to 20—21°, as the author considers the range of 5° allowed in the official method is too great, especially for crude quebracho extracts.

D. WOODROFFE.

Determining the p_H of tan liquors by the hydrogen electrode. T. F. G. HEPBURN (J. Soc. Leather Trades' Chem., 1930, 14, 261).—Poisoning of the electrode is reduced in the following way. A freshly-coated Rideal electrode is immersed in distilled water and hydrogen is bubbled through so that the whole cylinder is sprayed by it as the hydrogen emerges. After 3 min. the water is replaced by the tan liquor, so that the electrode is completely immersed therein, and the hydrogen is sprayed over it. A constant reading is attained in 2 min.

D. WOODROFFE.

Measurement of the colour of tanning extracts. G. BALDRACCO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 100—109).—For this measurement the Lovibond tintometer gives inexact results, but if the tanning liquid is used in suitable concentration, so that tanning is complete, practical tests with hides give useful data.

T. H. POPE.

Analytical controls of sulpholeates used in tanning. II. Sulphoricinoleates (II). III. Sulpholeates. M. GERONAZZO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 142—144, 144—150).—II. The results of a more strict chemical analysis of 37 samples of sulphoricinoleates are in agreement with the indications of the simple technical analysis proposed (B., 1928, 580), the value of which is thus confirmed.

III. Sulpholeates with a basis of fish oils should remain clear and liquid at the ordinary temperature, such properties not being due to dilution with mineral oil or other organic solvent. Fish and cetacean oil sulpholeates are brownish-yellow or brownish-red, bluish-green fluorescence usually indicating the presence of mineral oil. Most sulpholeates exhibit the odour due to sulphonation, which becomes more evident in their aqueous emulsion, as well as the characteristic odour of the original fatty material and that of any solvent added. The reaction towards litmus paper is usually acid, but the use of highly acid products may be harmful in certain tanning operations. The characters of technical importance in an emulsion of sulpholeates are colour, intensity, duration, emulsive capacity, and behaviour towards ammonia, these being observed after shaking 2 c.c.

of the sulpholeate with 20 c.c. of water for 2 min. The value of the sulpholeate is directly related to the duration of the emulsion and to the emulsive capacity (*loc. cit.*), which should be determined by a tanning test. The behaviour towards ammonia is examined by adding ammonia until the emulsion has a distinct odour of it, and then shaking; destruction of the emulsion with formation of a clear liquid indicates derivation from sulphonated matter only, whilst persistent turbidity shows the presence of extraneous matters such as emulsive oils (mineral or resin) or of other emulsifying agents insoluble in ammonia. The titre is determined as previously described (*loc. cit.*). The above method of technical examination is applied to 47 samples of sulpholeates of various types, the results being in agreement with those of more accurate chemical analysis.

T. H. POPE.

Tanning problem. L. MEUNIER and K. LE VIET (*Cuir tech.*, 1929, 22, 432—439; *Chem. Zentr.*, 1930, i, 785).—The terms tanning agent, intensity of tanning, and astringency are defined, and the effect of centrifuging on the capillary water is considered. Determinations of the astringency of various leathers were made.

A. A. ELDRIDGE.

Limits of acidity and temperature in "clearing" semi-tanned sheepskins. P. S. BRIGGS (*J. Soc. Leather Trades' Chem.*, 1930, 14, 244—253).—Elongation-temperature curves were derived for pieces of semi-tanned sheepskins (Egyptian, Smyrna, Syrian, and Mosul tannages) in water, 0.1N-, 0.02N-, and 0.01N-sulphuric acid. Maxima were observed in one case at 80° in water, diminishing as the concentration of the acid "clearing" solution was raised to 60° for 0.1N-sulphuric acid. The maximum was as low as 37° for some tannages, indicating that the temperature of "clearing" should not exceed 30°, otherwise the skins may be damaged, producing harsh leather and loss in area.

D. WOODROFFE.

"Red stains" and "red heat" on salted hides. F. STATHER (*Collegium*, 1930, 151—153; cf. B., 1930, 433).—The author observes that "red stains" and "red heat" are not caused by different bacteria as suggested by Lloyd and co-workers (cf. B., 1930, 251). The characteristics of the bacteria which cause "red heat" agree closely with those of the bacteria which cause "red stains" viz., *Micrococcus roseus*, *Sarcina lutea*, and *S. auriantica*. The chief difference is their reaction to common salt. The "red heat" bacteria give optimum growth on a medium which contains 16—30% of sodium chloride, and only poor growth when only 6—8% was present. The "red stain" bacteria showed no growth on a medium containing 16% of sodium chloride, but when grown on media increasing in salt content to 20% practically no difference was found between them and the "red heat" bacteria, except that the latter developed much better and more quickly. Cultures of "red heat" bacteria were made on media of decreasing salt contents. D. WOODROFFE.

Violet specks on salted raw hides. F. STATHER, G. SCHUCK, and E. LIEBSCHER (*Collegium*, 1930, 153—161).—Small reddish- or bluish-violet specks are often observed on the flesh side of salted raw skins. They

are caused by chromogenic micro-organisms, and disappear during soaking and liming. No increased loss of hide substance was observed on stained skins, but violet specks were found on the grain side of skins which had been stained on the flesh side, and they rendered such skins unfit for pale shades. D. WOODROFFE.

Salt stains. II. Grain damage on limed hide and chrome-tanned leather. F. STATHER and G. SCHUCK (*Collegium*, 1930, 161—169; cf. B., 1929, 140).—Determinations of the calcium, phosphoric acid, and sulphuric acid contents of a number of salt-stained and unstained raw calf-skins showed them to be the greater in the salt-stained skins. The chromium contents of salt-stained and unstained pieces were determined after tannage and found to be the same except for one finished leather in which the chromium content was the greater in the tanned salt-stained piece. The stains are not removed by liming, bating, nor pickling. They are fixed by tannage and generally have a darker green colour than the normal tanned leather, but sometimes they are brighter coloured. Dyes are absorbed differently by the stained and the unstained portions. Fat and season are only slightly or not at all absorbed by the salt-stained grain, which cracks on glazing if badly stained. D. WOODROFFE.

Bacteriology of the violet specks on salted raw hides. F. STATHER and E. LIEBSCHER (*Collegium*, 1930, 170—175; cf. preceding abstract).—*Micrococcus roseus*, *M. luteus*, and *M. pyogenes albus* have been identified amongst the bacteria on salted raw hides showing violet specks. Several harmless forms of *Proteus* were found and also a species of bacteria (*Actinomyces*) which formed long threads, coloured the medium reddish, and liquefied gelatin.

D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leathers. VI. W. J. CHATER (*J. Soc. Leather Trades' Chem.*, 1930, 14, 133—153; cf. B., 1930, 574).—Shrinkage curves have been derived for a number of commercial sole leathers in different stages of tannage. The initial shrinkage temperature (I.S.T.) of the fully-tanned leathers was in every case less than that of the half-tanned products, and was increased by the removal of water-soluble matter from the fully-tanned leathers. It is shown that the I.S.T. is increased by treating the fully-tanned leather with buffer solutions, attaining a maximum at p_H 5.0, and then diminishing slightly with further increase in the p_H of the buffer solution. The p_H of the half-tanned leather is diminished as the tannage becomes more complete, which would explain the change in I.S.T. Samples of pelt after being tanned with quebracho, valonia, oakwood extract, and myrobalans, respectively, were found to have varied I.S.T. values. D. WOODROFFE.

Sodium hydrogen sulphide in the manufacture of glove leather. V. CASABURI (*Cuir tech.*, 1929, 22, 489—494; *Chem. Zentr.*, 1930, i, 784).—A study of conditions for depilation.

A. A. ELDRIDGE.

Testing of patent leather. F. FEIN (*Collegium*, 1930, 117—118).—Tests were carried out on two patent leathers A and B. The tensile strength and stretch of the patent film on A were slightly less than those

of the leather itself, whereas the same properties of the film on *B* were about 30% less than those of the leather. Of the two samples, *A* was better than *B*. This affords a useful method of testing patent leathers. Sample *B* would crack the more readily.

D. WOODROFFE.

Identification of formaldehyde in leathers. P. CHAMBARD (J. Soc. Leather Trades' Chem., 1930, 14, 258—261).—Different leathers were tested for formaldehyde by the Schiff and the Grosse-Bohle (Z. Unters. Nahr. Genussm., 1907, 14, 89) reactions. A positive reaction was obtained with the Schiff test on pelt, chamois, alum-tanned and formaldehyde leathers; it is consequently useless on leather. Positive reactions were given by the Grosse-Bohle test on undyed formaldehyde-tanned leathers, whether freshly tanned or aged, but not with dyed formaldehyde leathers. The colour was developed slowly on aged leathers. This test is therefore satisfactory for detecting formaldehyde in leathers.

D. WOODROFFE.

Effect of different methods of preparation of sole leather on resistance to wear. A. GOLDENBERG (Cuir tech., 1929, 22, 414—422; Chem. Zentr., 1930, i, 786).

Determining the insoluble matter [in tan liquors] by the sedimentation method. V. KUBELKA and V. NEMEC (J. Soc. Leather Trades' Chem., 1930, 14, 254—258).—Cf. B., 1929, 991.

PATENTS.

(A) **Production of a bate or drench for skins.**
(B) **Bating or drenching limed, unhaired hides or skins.** I. G. FARBERIND. A.-G. (B.P. 304,214 and 304,294, [A] 16.1.29, [B] 18.1.29. Ger., [A] 16.1.28, [B] 18.1.28).—(A) Bacteria capable of hydrolysing albumin and fat are cultivated on a medium containing these substances (e.g., blood and animal fat) and are then removed, the liquor being used for bating or drenching purposes. (B) The above bating liquor (0.3—0.5%) is allowed to react for 1 hr. on rinsed, limed, unhaired calf skin at 35—37°.

D. WOODROFFE.

Manufacture of glutinous products. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 328,197, 22.12.28. Addn. to B.P. 313,101; B., 1929, 653).—Finely-ground dry yeast (10 pts.) or products of yeast autolysis are treated with aldehydes (e.g., paraldehyde, 1 pt.), their polymerisation products, or with substances capable of developing them, prior to, during, or after treatment with weakly alkaline or acid reacting agents or salts or a strong alkali (e.g., 30 pts. of 8% sodium hydroxide solution). The product, a glue, is left for 3 hrs.

D. WOODROFFE.

Treatment of fish skins. A. EHRENREICH (U.S.P. 1,762,053, 3.6.30. Appl., 24.1.28. Belg., 26.1.27).—See B.P. 281,919; B., 1928, 133.

Process for hardening colloids. A. MILLER, Assr. to AGFA ANSCO CORP. (U.S.P. 1,763,533, 10.6.30. Appl., 2.2.24. Ger., 27.11.23).—See B.P. 231,564; B., 1925, 479.

Dyeing of chrome leather (B.P. 304,608).—See VI.

XVI.—AGRICULTURE.

Soil evaluation and examination. A. VON NOSTITZ (Landw. Jahrb., 1930, 71, 837—854).—In the mechanical analysis of soils, the effects of the use of hard water for sedimentation, of the various methods of dispersion, and the fluctuations of temperature during the process are considered, together with the relationship between the mechanical composition and water relationships of soils.

A. G. POLLARD.

Improvements in the Kühn method for the mechanical analysis of soils. L. PINTO (Z. Pflanz. Düng., 1930, 17A, 102—112).—Kühn's method is modified by determining the chalk and organic matter in the sand and clay fractions. Boiling the soil suspension for 2 hrs. did not appreciably affect the proportion of clay recorded, but markedly decreased the necessary washing period in the elutriation cylinder.

A. G. POLLARD.

The phosphate question [in soils]. O. ARRHENIUS (Z. Pflanz. Düng., 1930, 16A, 307—310; cf. B., 1930, 386).—Evidence from culture experiments indicates that silica cannot, even partially, replace phosphate as a plant nutrient and actually tends to reduce the efficiency of phosphates in this connexion.

A. G. POLLARD.

Determination of the root-soluble phosphate in soils by the seedling method. A. NEMEC (Z. Pflanz. Düng., 1930, 16A, 354—363).—Low values for the assimilable phosphate in soils, determined by the seedling method as compared with field trials and other methods, were due to deficient nitrate in the soils and consequent abnormal assimilation by the plants. Effects of increasing applications of nitrogenous fertilisers on the Neubauer phosphate values of soils are recorded.

A. G. POLLARD.

Examination of the Neubauer method and its application to easily assimilable nitrogen [of soils]. R. THUN (Z. Pflanz. Düng., 1930, 16A, 257—283).—Changes in the Neubauer values for assimilable potash and phosphate in soils with season and with fertiliser treatment are recorded and discussed together with the selection of "limiting values" for particular crops. Qualitative examination of the nitrate content of soils may be made by immersing the cut stems of seedlings grown by Neubauer's method in diphenylamine solution and observing the intensity of the blue colour produced (cf. Niklas and Vogel, B., 1924, 142). Comparison tests with fertilised soil form a basis for the calculation of the nitrogen requirement of the soil. Results of such tests are confirmed by field trials.

A. G. POLLARD.

Neubauer analyses and their relation to field trials. K. HESSE (Landw. Versuchs-stat., 1930, 110, 1—31).—Irregular results of Neubauer tests are ascribed to lack of uniformity of experimental conditions, notably the lighting of the growing seedlings, moisture conditions of the soil before and during the experiment, and the season during which the trials are made. With comparable conditions Neubauer results agree well with those of field trials; in general, better agreement is obtained for potash than for phosphates. The amount of minerals assimilated by plants in seedling trials are

dependent on the time of application of the fertilisers. The intake of potash and phosphate by the seedlings is not affected by nitrogenous manuring.

A. G. POLLARD.

Lime requirement of lime-deficient moor soils.

B. TACKE [with T. ARND, W. SIEMERS, A. POOCK, I. SAFFRON, and A. SPIECKER] (Z. Pflanz. Düng., 1930, 9B, 216—234).—The effect of increasing quantities of lime on crop production in moor soils is examined in pot cultures. Maximum crops corresponded in most cases with partial neutralisation of the total acidity, the actual proportions varying with the crop. Excessive liming produced markedly decreased yields. The repeated use of physiologically acid or alkaline fertilisers changes the "lime condition" of these soils to a considerable extent.

A. G. POLLARD.

Von Csiky's method for determining the lime requirement [of soils]. J. WOLTERS DORF (Z. Pflanz. Düng., 1930, 16A, 290—297).—In determinations of the hydrolytic acidity of soils by leaching with calcium acetate solution (von Csiky, B., 1929, 788), the relationship between the amount of calcium acetate used and the acidity developed is represented by a hyperbolic curve. Thus the end-point of the reaction, i.e., the true hydrolytic acidity, is not actually observed, but must be calculated after experimental determination of at least two points on the curve. The retentive power of the absorbing complex of soils for hydrogen ions is sufficiently great to demand relatively high proportions of other ions for its complete displacement. The calcium ion exhibits relatively greater displacing power than the ions K^+ , Na^+ , Mg^{2+} , or NH_4^+ , and is more strongly absorbed by the soil. Von Csiky's method does not include consideration of these observations and gives results which although not generally far in error must nevertheless be suspect.

A. G. POLLARD.

The chlorine question [in soils]. O. ARRHENIUS (Z. Pflanz. Düng., 1930, 16A, 310—314).—Sand cultures are described showing the effects of varying concentrations of sodium chloride on the crop yields of wheat, barley, oats, clover, sugar beet, and sugar cane.

A. G. POLLARD.

Determination of exchange acidity in potassium chloride extracts of soils. R. ALBERT (Z. Pflanz. Düng., 1930, 16A, 305—307).—Ferrous compounds may occur in potassium chloride extracts of soil and cause unsatisfactory results in p_H determinations with the quinhydrone electrode. The mechanism of the reactions involved is discussed.

A. G. POLLARD.

p_H determinations [of soil] in water and potassium chloride solutions. H. ELLEDER (Z. Pflanz. Düng., 1930, 16A, 283—289; cf. B., 1930, 630).—Examination is made of the factors causing differences in the recorded p_H values of soils as determined in aqueous and in N -potassium chloride suspensions. The more alkaline values for potassium suspensions of chalky soils cannot be correlated with the calcium content (whether carbonate or total), but are more closely related to the lime and magnesia content (cf. Goy and von Burow, B., 1929, 830).

A. G. POLLARD.

Physiological reaction of potash salts. W. ZIELSTORFF and K. NEHRING (Z. Pflanz. Düng., 1930, 17A,

67—79).—In pot cultures neither "40% potash salts" nor kainite affected soil reaction, but potassium and magnesium sulphate mixtures tended to produce slight acidity. In field trials none of these materials affected soil reaction.

A. G. POLLARD.

Relationships of the different acids and bases in soil to the electrometric titration.

S. GOY, P. MÜLLER, and O. ROOS (Z. Pflanz. Düng., 1930, 16A, 342—354; cf. B., 1930, 254).—Comparative determinations of acidity, p_H values, and electrometric titration values of soils are recorded. Exchange acidity does not exist in soils with $p_H > 5.5$. For practical purposes electrometric titration can be utilised for determinations of hydrolytic and exchange acidity, neutralisation values, and lime requirement. The authors' system of soil classification is preferable to individual determinations of p_H value, titratable acidity, or buffer capacity for characterising the acid injury of heavy soils. The assimilable potash and phosphate contents of soils may be determined from electrometric titration values.

A. G. POLLARD.

Nitrogen losses from "Kalkammonsalpeter" by the volatilisation of ammonia.

W. SELKE (Z. Pflanz. Düng., 1930, 9B, 207—215).—Laboratory experiments indicate possible losses of free ammonia from "Kalkammonsalpeter" when mixed with moist soil. Under field conditions, unless the fertiliser is applied as a top dressing or used on a very light chalk soil, the absorptive power of the soil is sufficient to retain any free ammonia formed.

A. G. POLLARD.

Nitrogen fertilisers. A. N. VOLSKAYA (Udobren. Urozhai, 1929, No. 4, 221—226).—On sandy and heavy loam soils ammonia fertilisers are as good as, or better than, nitrates with potatoes and oats; ammonium nitrate ranked first, followed by the sulphate, whilst calcium nitrate, calcium cyanamide, and urea gave erratic results.

CHEMICAL ABSTRACTS.

[German] supervisory department for ammoniacal and phosphatic fertilisers. ANON. (Z. Pflanz. Düng., 1930, 9B, 234—235).—Official notification is given of changes in the naming and marking etc. according to the regulations for the sale of fertilisers.

A. G. POLLARD.

Preparation and action of "artificial stall manure." O. FLIEG (Z. Pflanz. Düng., 1930, 9B, 193—207).—During the rotting of straw the pentosan content steadily decreases and serves as a measure of the extent of the rotting process. Satisfactory rotting of straw composts results if the straw is first wetted with water and when heating begins (a few days) it is stacked with the admixture of nitrogenous materials. Addition of lime, potash, or phosphates to the stack is unnecessary. After about 3 months the mixture should contain approx. 80% of water and 0.35% N. If the customary period of time is allowed before use the compost causes no plant injury due to temporary binding of soil nitrate, but exerts a fertilising effect corresponding to its nitrogen content. By addition of soluble nitrogen to the compost in quantity to give a total nitrogen content similar to that of fresh stall manure, an "artificial stall manure" is obtained with a greater efficiency than

the natural material. On light soils similar results are obtained by ploughing-in straw with the addition of an adequate proportion of soluble nitrogen (2%).

A. G. POLLARD.

Physical chemistry of cultivated soils. II. Influence of manuring on the properties and adsorption potential of low-lying loams. R. A. HERZNER (Z. Pflanz. Düng., 1930, 16A, 315—331; cf. B., 1930, 294).—Elutriation and sedimentation processes for the mechanical analysis of soils give higher values than pipette and hydrometer methods. Kozeny's equation ($h/\sqrt{t} = K$), representing the rate of capillary rise (h) in soils, is examined and its relation to other physical properties discussed, together with its possible use in showing the effects of fertilisers on soil-water movements. Measurements of the adsorption potential of soils (*loc. cit.*) are valuable in studies of soil texture and water relationships. Changes in the adsorption potential of soils following manuring result not only from the increased electrolyte content, but also from the altered degree of dispersion of the soil colloids. No definite relationships were observed between adsorption potential and crop increases of manured soils.

A. G. POLLARD.

Relationship between soil reaction, distribution of meadow plants, type and yield of meadows. KLAPP (Landw. Jahrb., 1930, 71, 807—835).—Optimum ranges of soil reaction for the growth of numerous species of meadow grasses etc. are recorded. For the majority of species the growth- p_H range is of such width that the characterisation of soil reaction by classification of the natural flora is indecisive. Highest hay yields are associated with soil reactions of p_H 5.0—6.8, with a preponderance of plants of better feeding value in the less acid areas.

A. G. POLLARD.

Lime-sensitiveness of yellow lupins and the rôle of the nodule bacteria in lupin sickness. R. REINCKE (Z. Pflanz. Düng., 1930, 17A, 79—102).—Chlorosis in yellow lupins is characteristic of a definite early stage in the growth of the plants and is distinct from and independent of injury produced by nitrogen deficiency, although both may result from excessive liming. Nitrogen deficiency following liming is due to decreased activity of the nodule bacteria. Chlorosis is rare in blue lupins, but lime injury to the nodule bacteria occurs. The latter is not caused by the increased alkalinity of soil following liming, but is the result of increased calcium concentration of the soil solution, which in turn changes the ionic ratios within the plant root.

A. G. POLLARD.

Influence of silicic acid, peat, and humus on the solubility and intake of phosphates [by plants]. W. JESSEN and W. LÄSCH (Z. Pflanz. Düng., 1930, 17A, 48—66).—Both colloidal silica and peat increase the solubility of dicalcium phosphate in water, but the effects are modified by the presence of other fertiliser salts. In sand cultures silica and peat increased the phosphate assimilation of plants only when phosphorus was supplied in a difficultly soluble form or in mixed fertilisers tending to render the phosphate insoluble. Peat increased the crop produced by dicalcium phos-

phate in physiologically alkaline nutrients. In the presence of peat silica was ineffective. With physiologically acid nutrients neither peat nor silica increased the crop yields. Peat and silica increased the efficiency of tricalcium phosphate in all cases examined, crop increases being the more marked in physiologically alkaline media.

A. G. POLLARD.

Nutrient intake and dry matter production by buckwheat (*Fagopyrum esc.*) and the yield law of higher plants. K. MATWALD (Z. Pflanz. Düng., 1930, 17A, 12—42).—Growth curves for buckwheat in pot cultures are recorded. The rate of dry matter production in leaves, stems, and roots and the corresponding intake of nitrogen and calcium are examined. The rate of calcium intake follows closely the total production of dry matter. The nitrogen intake is relatively more rapid and is closely paralleled by the dry matter production of leaves and roots. Uniform relationships, which are expressed in S-form curves, exist between the period of growth and the total dry matter of the plant, the total nitrogen intake, total leaf surface, dry weight of leaves and of roots, nitrogen intake of leaves and stems, and the calcium intake of stems.

A. G. POLLARD.

Influence of colloidal silica on the assimilation [by plants] of the phosphoric acid of rock phosphates. A. REIFENBERG (Z. Pflanz. Düng., 1930, 17A, 1—11).—The citric solubility and assimilability of rock phosphates depend on the degree of dispersion of the particles. The ease of formation of aggregates (probably by perikinetic coagulation) in aqueous suspensions increases with the fineness of division. The formation of aggregates is largely prevented by colloidal silica, and stable silica-phosphate sols can be obtained. The favourable influence of silica on the assimilation of phosphates by plants is attributed to this effect.

A. G. POLLARD.

Influence of nutrition on the size distribution of potato starch grains. G. BREDEMANN and O. NERLING (Z. Pflanz. Düng., 1930, 16A, 331—341).—The average size of starch grains from potatoes fertilised with nitrogen is less than those from unfertilised crops, the difference being greatest before the dying back of the potato haulm. The effects of potash and phosphate fertilisers are smaller. Results vary with the nature of the fertiliser used and with the time of its application, the general tendency being to reduce the average grain size.

A. G. POLLARD.

Tomato, a sensitive and rapid indicator of the phosphorus-poverty of soil. L. MEYER (Fortschr. Landw., 1929, 4, 684—686; Chem. Zentr., 1930, i, 887).—Lack of phosphorus is detectable 14 days after sowing; the method gives results in accord with those of Neubauer and of von Wrangel.

A. A. ELDRIDGE.

Toxicity of sulphur to spores of *Sclerotinia cinerea* as affected by the presence of pentathionic and other sulphur acids. O. N. LIMING and H. C. YOUNG (J. Agric. Res., 1930, 40, 951—962).—Comparative tests of the toxicity of sulphuric, sulphurous, di-, tri-, and penta-thionic acids to spores of *Sclerotinia cinerea* are recorded. The last-named acid alone showed

any marked toxicity. Filtrates from wetted sulphur contained pentathionic acid and were toxic. Sulphur freed from pentathionic acid was not toxic, but regained its toxicity and pentathionic acid content after aeration. In field tests sulphur to which oxidising agents were added (*e.g.*, permanganates) was more effective than ordinary sulphur in controlling apple scab.

A. G. POLLARD.

Factors concerned in arsenical injury to foliage.

S. F. POTTS (J. Econ. Entomol., 1930, **23**, 469—470).—Moisture accumulating on leaf surfaces contains appreciable amounts of carbon dioxide and weakly acid matter derived from the leaves. The solubility of lead and calcium arsenates in such solutions is greater than in distilled water which is used in laboratory tests of solubility. Field trials show calcium arsenate to be more soluble in leaf moisture than the lead compound. Lime used in conjunction with lead acetate in sprays tends to form calcium carbonate and arsenate. On foliage, rain quickly removes the carbonate and some arsenate and the leaf surface again becomes acid with an increase in the proportion of soluble arsenic. Some cases of the protective action of lime used in lead arsenate sprays may be ascribed to such formation of soluble arsenate and its removal by rain.

A. G. POLLARD.

Pyrethrum and soap, a chemically incompatible mixture. R. C. ROARK (J. Econ. Entomol., 1930, **23**, 460—462).—The use of soap in pyrethrum sprays leads to partial saponification of the pyrethrins I and II and consequent loss of insecticidal value. This is minimised by the use of the least necessary amount of neutral soap and preparation in the cold. Saponin and other spreaders are recommended as substitutes for soap.

A. G. POLLARD.

Petroleum products. CARPENTER.—See II. **Solubility of the phosphoric acid of monocalcium phosphate.** STOLLENWERK.—See VII. **Sugar-beet structure and sugar content.** ARTSCHWAGER.—See XVII. **Ash of cows' milk.** SCHNECK and GÖRGEL.—See XIX.

PATENTS.

Production of mixed fertilisers. F. JOST (B.P. 312,169, 18.5.29. Ger., 21.5.28. Addn. to B.P. 306,046; B., 1930, 611).—In a modification of the prior process, a mixture of nitric acid with an excess of calcium phosphate and one or more alkali chlorides is distilled in a current of air and, if desired, under reduced pressure; the vapours evolved are absorbed by ammonia, sodium hydroxide, milk of lime, etc. to yield non-hygroscopic, nitrogenous fertilisers; the residue, consisting of calcium phosphates and alkali nitrates and chlorides, is used, after removal of insoluble constituents, for the production of mixed fertilisers.

L. A. COLES.

Manufacture of salt mixtures suitable for use as fertilisers containing carbamates or conversion products of carbamates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,326, 15.2.29).—The carbamates obtained by the process described in B.P. 328,005 (B., 1930, 612) are not separated from the solution, but are worked up, with or without conversion into other products, together with the salts formed during their production; *e.g.*, a mixture of potassium carbamate and

ammonium nitrate is obtained by passing carbon dioxide into a solution of potassium nitrate in liquid ammonia and subsequently removing excess ammonia by evaporation.

L. A. COLES.

Manufacture of fertiliser salts. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,641, 16.1. and 2.5.29).—The salts are formed by the double decomposition, in the presence of liquid or gaseous ammonia free or practically free from water, of two or more salts of strong mineral acids which are at least slightly soluble in liquid ammonia. The products may be recovered together by evaporation of the ammonia or, where practicable, may be separated by filtration before the evaporation. In an example gypsum is stirred for about 6 hrs. at the ordinary temperature with a mixture of liquid ammonia and ammonium nitrate, and ammonium sulphate and calcium nitrate are recovered by filtration and by evaporation of ammonia from the filtrate, respectively.

L. A. COLES.

Manufacture of fertiliser salts containing potash and nitrogen or potash, nitrogen, and phosphorus.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,883, 1.7.29).—Potassium carbamate, alone or mixed with other substances, is treated with acids (sulphuric, nitric) or acid salts; residual acids, if present, are neutralised, preferably with ammonia.

L. A. COLES.

Utilisation [as manure] of the final liquors in the potassium industry.

KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 329,495, 6.5.29. Ger., 6.12.28).—The liquors mixed with dilute phosphoric acid or phosphate solutions are treated with sufficient ammonia to neutralise the acid; magnesium ammonium phosphate is precipitated, removed, washed, and dried at about 100° to yield a fertiliser containing 45·7% P_2O_5 , 9%N, 25·9% MgO , and 11·6% H_2O , which may be mixed with ammonium nitrate. The liquor is heated with lime to recover the ammonia contained in it.

L. A. COLES.

[Dusting-powders for] disinfecting seeds. M. ENGELMANN, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,749,575, 4.3.30. Appl., 29.10.26).—The powders comprise mixtures of copper carbonate or other basic copper salts with acids (*e.g.*, phosphoric, boric, benzoic, benzenesulphonic, oxalic, tartaric acid), with acid salts (*e.g.*, sodium bisulphate, calcium hydrogen phosphate), or with salts having an acid reaction (*e.g.*, copper or ferrous sulphate, zinc chloride). The mixtures may be prepared by grinding a copper salt, *e.g.*, copper sulphate, with a base, preferably magnesium oxide, in quantity insufficient to convert the salt completely into the basic salt.

L. A. COLES.

Adhesive compositions for catching insects.

I. G. FARBENIND. A.-G. (B.P. 306,906, 26.2.29. Ger., 27.2.28).—The products obtained by exhaustive chlorination, first at room temperature and then on a water-bath, of vegetable or animal oils or fats (linseed, wood, tall, fish oils), after the remaining hydrochloric acid has been removed with a stream of air and later of a little ammonia gas, are mixed with resins, waxes (carnauba wax), and machine oil.

L. A. COLES.

Immunising grain (B.P. 304,741).—See IX.

XVII.—SUGARS; STARCHES; GUMS.

Structure of sugar beet in relation to sugar content and type. E. ARTSCHWAGER (J. Agric. Res., 1930, 40, 867—915).—Numerous data concerning the anatomical structure of sugar beet are recorded and discussed in relation to the sugar content. A. G. POLLARD.

Preliminary investigations on two cellulosic wastes as sources of xylose. W. C. HALL, C. S. SLATER, and S. F. ACREE (Bur. Stand. J. Res., 1930, 4, 329—343).—An attempt is made to extract crystalline xylose or xylose molasses from peanut hulls or cottonseed bran without materially injuring the plant fibre for use by the cellulose technologist. Both of these cellulosic wastes are rich in furfuraldehyde-forming materials, but, so far, crystalline xylose has been obtained only from cottonseed bran. The process, which has been employed on a semi-commercial scale, consists in heating the material with water under pressure to remove soluble gums which interfere with the crystallisation, followed by treatment with cold 0.12*N*-sulphuric acid and washing with water, whereby a large proportion of mineral matter is removed. Finally, the xylose is extracted by cooking with 0.16*N*-sulphuric acid under pressure, and the extract is decolorised, evaporated, neutralised with baryta, and allowed to crystallise. F. R. ENNOS.

Purification of potato and maize syrups by activated carbon ("Norit"). W. TAEGENER (Z. Spiritusind., 1930, 53, 155—157).—Bone charcoal for the purification and decolorisation of the syrups has been replaced by an activated carbon, termed "Norit" (special glucose quality), which is prepared in the form of a powder by the carbonisation of certain plant materials. It is added direct to the syrup solution at 70—75° and is stirred vigorously for 20 min. prior to being separated in the filter-press, through which it allows, relatively to bone charcoal, the syrup to pass more freely even at high concentrations. The desired standard colour can be controlled by the amount of carbon added, and as its power of adsorption is much higher than that of bone charcoal, a relatively small quantity is required. Prefiltration of the syrup is rendered unnecessary by its use, and it does not require frequent reactivation since the filter runs longer before the carbon is completely exhausted. Generally, 0.7—1.4% of active carbon is required, when calculated on a syrup of $d_{1.43}$. The preparation of maize syrup differs from that of potato syrup, and previous to the treatment with carbon, oil and excess proteins must be removed from the maize wort. In addition, as the carbon is more active in acid solution, the maize wort is partly neutralised. Reactivation of the carbon is effected by alternate extraction with dilute hydrochloric acid and sodium hydroxide solutions followed in each case by washing with water. After drying, the carbon has 50—60% of its original activity. C. RANKEN.

Solvents for gum arabic. R. TAFT and L. MALM (Trans. Kansas Acad. Sci., 1929, 32, 49—50).—Gum arabic (purified by precipitation from aqueous solution with alcohol) dissolves in 1.7 pts. of water at 25°, 1.6 at 50°, or 1.5 at 90°. Gum arabic is very sparingly

soluble in solvents other than water and certain aqueous mixtures. CHEMICAL ABSTRACTS.

Determination of water. LEPPER.—See VII. **Chlorine in soils [for sugar beet etc.].** ARRHENIUS.—See XVI. **Lactose in milk.** GOHR. **Determining sugar in milk.** MÜNCHBERG. **Sucrose and lactose of milk-chocolate.** RINCK and KAEMPF. **Effect of heat on sugars in chocolate.** LECOQ.—See XIX.

PATENTS.

Production of gum from locust beans. A. MÜLLER, ASS. to S. NEUMAN (U.S.P. 1,749,833, 11.3.30. Appl., 28.11.28. Ger., 8.10.28).—A solution in cold water of a mixture of the ground beans, freed from skin and seeds, and alkylated (methyl) naphthalenesulphonic acid is boiled by direct or indirect heating with steam.

L. A. COLES.

Digestor (B.P. 329,600). **Refractometers (B.P. 314,994).**—See I. **Esters of carbohydrates (B.P. 305,661).**—See III.

XVIII.—FERMENTATION INDUSTRIES.

Research work in the yeast field. L. H. LAMPITT (J. Inst. Brew., 1930, 36, 250—260).—The problems surrounding the life and activities of yeast are critically reviewed and a plea is made for more work on standardised lines on simple substances. C. RANKEN.

Boiling of worts. R. WORSSAM (J. Inst. Brew., 1930, 36, 260—265).—The relative advantages of boiling worts by steam-heating apparatus in the boiling vessel or by direct fire-heating beneath it are discussed. The annual depreciation of a steam-heated copper is less than that of the fired coppers, and for economy in the consumption of coal the former has an enormous advantage. Steam-jacketed vessels have the serious drawbacks that a central boil is not attained, nor does the violent upheaval of the wort occur, which is essential for circulation and aeration. In addition, the inner and outer pans are liable to leak at the joints between the two. With the modern steam heater, which is placed in the centre of the vessel, a central boil and vigorous circulation of the liquid are assured. The use of superheaters for raising the temperature of the steam supply to the coppers has no economic value unless the heat is drawn from sources which otherwise would be lost. It is essential that the steam-heating apparatus be served by a steam trap able to remove the condensed water rapidly. No material differences could be traced between the worts boiled by the two methods, and, if caramelisation did occur in a fire-copper, it could only do so by the overheating of the bottom of the vessel with consequent damage.

C. RANKEN.

Titrimetric determination of carbon dioxide in beer. J. DE CLERCK (Bull. trim. Assoc. anc. Elèves Ecole Brass. Louvain, 1929, 29, 30—37; Chem. Zentr., 1930, i, 300).—A modification of Cannizzaro's method consists in the use of sodium hydroxide for absorption and the titration of the solution to the colour of phenolphthalein in a buffer solution of p_H 8.4.

A. A. ELDRIDGE.

Detection of fruit wine in grape wine by means of the sorbitol method. G. REIF (Z. Unters. Lebensm.,

1930, 59, 99—104).—The precipitation of dibenzylidene-sorbitol in Werder's test (cf. B., 1929, 619) is largely prevented by lævulose, sucrose, invert sugar, starch sugar, dextrin, etc., but not by dextrose. After fermentation for 10 days with pure wine yeast the inhibitory influence of the sugars is diminished except in the case of dextrin, in the presence of which the test is completely negative. In this case the distillate residue should be extracted with hot absolute alcohol and the extract tested after complete removal of the alcohol. In performing the test it is recommended that the viscous distillate be diluted with a few drops of water and warmed, the optimum quantities of the reagents being 0.2—0.25 c.c. of benzaldehyde and at least 1 c.c. of sulphuric acid (1:1). Experiments with grape wine containing various percentages of sweetened cider are described.

H. J. DOWDEN.

PATENTS.

Manufacture of malt. O. SLEEMAN (B.P. 329,574, 30.7.29).—The apparatus, which is rotatable on a horizontal axis, consists of a container which is divided by perforate partitions into two outer compartments constituting air spaces, and an inner compartment comprising a germination and dispersion chamber in free communication with each other. To secure a flow of air through the germination chamber, each air space is provided with an air pipe and door, so that the one air space acts as a supply and the other as an exhaust for the air and gases. The direction of flow may be reversed. Slow rotation of the container causes the grain to fall from the germination chamber to the dispersion chamber and back to the germination chamber.

C. RANKEN.

Automatic fermentation apparatus. A. P. HARRISON, Assr. to STANDARD BRANDS, INC. (U.S.P. 1,750,267, 11.3.30. Appl., 9.6.27).—The apparatus for the production of yeast by an addition process consists of a series of fermenters at progressively lower levels, each of which has approximately double the capacity of that preceding. Connecting each fermenter to the next is a pipe in the form of a \cap , which controls the maximum level of the liquid. The capacities of the tubes are sufficient to empty the fermenters to which they are attached in the same period of time. Yeast nutrients may be supplied independently to each fermenter, and valves fitted to the lead-off tubes permit the removal of an aliquot portion of the liquid.

C. RANKEN.

Apparatus for pasteurising beer and other liquids. R. F. GRAHAM (B.P. 329,733, 8.3.29).—Liquid containers are automatically fed on to a perforated, rotatable carrier provided with a fixed guide which spirals round the carrier. The moving containers are sprayed with pasteurising liquid from a tank with perforated bottom placed above the carrier. The tank is divided into 5 co-axial compartments each of which supplies pasteurising liquid, the temperatures of which rise gradually from the innermost compartment outwards. A similarly divided tank is placed under the carrier to collect the pasteurising liquid, which, after heating, is returned to the upper tank.

C. RANKEN.

Apparatus for obtaining yeast. O. LÜHRS, Assr. to ZELLSTOFF-FABR. WALDHOF (U.S.P. 1,760,706, 27.5.30.

Appl., 29.2.28. Ger., 12.3.27).—See B.P. 287,052; B., 1928, 797.

Manufacture of yeast. A. P. HARRISON, Assr. to STANDARD BRANDS, INC. (U.S.P. 1,761,789, 3.6.30. Appl., 6.10.25).—See B.P. 259,572; B., 1927, 666.

Electrophoretic deposition of solids (B.P. 305,630 and 307,912).—See XIV.

XIX.—FOODS.

Detection of hard wheat grits. E. BERLINER and R. RÜTER (Z. Getreidew., 1929, 16, 212—213; Chem. Zentr., 1930, i, 606).—A criticism of Kühl's test.

A. A. ELDRIDGE.

Detection of hard wheat grits. H. KÜHL (Z. Getreidew., 1929, 16, 213—216; Chem. Zentr., 1930, i, 606).—A reply to Berliner and Rüter (preceding abstract) and statement of exact conditions.

A. A. ELDRIDGE.

Xylene value [of adulterated butter]. E. HANKE and F. DEUTSCHMANN (Milchwirt. Forsch., 1929, 8, 525—532; Chem. Zentr., 1930, i, 607).—The minimum permissible value in van Raalte's xylene test is 19.0.

A. A. ELDRIDGE.

Freshness of milk. K. L. PESCH and U. SIMMERT (Milchwirt. Forsch., 1929, 8, 551—575; Chem. Zentr., 1930, i, 607).—A simple indicator method for the determination of the p_H of milk is described. Resazurin is added to the milk, which is then kept at 20°, the resistance to reduction to a red shade being a measure of the freedom from bacteria.

A. A. ELDRIDGE.

Viscosity of milk and milk products. I. Skim milk, full-cream milk, and cream. II. Fresh and treated milk and cream. W. MOHR and F. OLDENBURG (Milchwirt. Forsch., 1929, 8, 429—461, 576—592; Chem. Zentr., 1930, i, 765—766).—I. Considerable differences were apparent in the results obtained with different forms of apparatus. The Lawaczek viscosimeter is preferred.

II. Determinations were made with naturally or artificially acidified milk. Ageing affects the state of aggregation of the fat.

A. A. ELDRIDGE.

Electrical conductivity of milk. II. Results of practical measurements on the milk of cows from different herds. J. KRENN (Z. Unters. Lebensm., 1930, 59, 32—62; cf. B., 1929, 574).—Observations on herds of several hundred cows and on individual animals have afforded further evidence that a high value for the electrical conductivity is indicative of abnormal secretion due to diseased udders. Suspected cases were almost invariably confirmed by bacteriological and clinical examinations. Although considerable variations occur between different animals, the value of the electrical conductivity for normal milk should not exceed 46×10^{-4} . As the value of κ rises so does the sodium chloride content, but the values for d , solids-not-fat, and refraction all decrease. The limits within which normally secreted milk may vary are given as d 1.0310—1.0360, fat-free solids 8.80—over 10%, and refraction 39.0—over 41.0. The lower values frequently quoted are ascribed to milk from sick cows. On the other hand, the lowering of f.p. remains practically constant, even

when the composition of the milk is so abnormally low as to suggest that water has been added.

H. J. DOWDEN.

"Alkali number" of the ash of cows' milk, with special reference to fodder and the lactation period. A. SCHNECK and B. GÖRGEL (Milch. Zentr., 1930, 59, 49—53, 65—70, 81—85, 97—101).—The results hitherto recorded for the potash, soda, and lime contents of milk ash have shown considerable variation, especially in the sodium value, e.g., 3.85—13.02%, resulting in wide differences in the "alkali number" (ratio K_2O/Na_2O). The earlier values for this ratio range from 2 to 4, but the more recent work of Nottbohm (cf. Milch. Forsch., 1927, 4, 336) gives an average value of 6 (max. 10). Further investigation has been made on the milk of stall-fed cows which received special diets, normal, rich, and poor in mineral matter. The perchlorate method of analysis was used throughout, and was found to give results in good agreement with those obtained by the platinichloride method. Mixed milk from groups of 10 cows in full milk gave alkali numbers of 2.2—2.4, whereas colostrum milk gave values of 1.2—1.5, and was characterised by a low potassium content. The results obtained with different animals showed considerable variation: thus the calcium value in one case was 25—30% higher than the average. Observations were made on two animals over a period of 230 days in order to study the changes during lactation. After 4 days the composition was normal, and thereafter the potassium value slowly decreased, the sodium value remaining more or less unchanged. The influence of fodder and of supplementing the diet with added mineral matter by dosing the cows with carbonates of potassium, sodium, or calcium was inconclusive, and appeared to depend on the physiological requirements of the animals, since during the period of feeding with fodder of low mineral content the highest individual value for the alkali number, viz., 4.7, and the highest sodium content, viz., 7.4, were recorded. No significant change in the calcium content was observed during the lactation period.

H. J. DOWDEN.

Titrimetric determination of lactose in milk.

H. GÖHR (Z. Unters. Lebensm., 1930, 59, 90—94).—The method is based on the reduction of potassium ferricyanide in the presence of sodium carbonate, followed by iodometric determination of residual ferricyanide. Fat and albumin are removed from 5 c.c. of milk by adding 1 c.c. each of potassium ferrocyanide solution (150 g./litre) and zinc sulphate solution (300 g./litre). The mixture is made slightly alkaline with caustic soda, diluted to 100 c.c., and, after 15 min., the precipitate is filtered off. A portion of the clear filtrate (2—6 c.c.) is diluted to 20 c.c. and treated with 10 c.c. of potassium ferricyanide solution (16.46 g. together with 70 g. of anhydrous sodium carbonate in 1 litre). After heating on a water-bath for 20 min., and then cooling, 10 c.c. of potassium iodide-zinc sulphate solution (50 g. of zinc sulphate, 25 g. of potassium iodide, and 250 g. of sodium chloride in 1 litre) and 10 c.c. of 9% acetic acid are added, iodine being then titrated with 0.05*N*-sodium thiosulphate. After deducting the value for a blank test, the lactose content is read from

tables derived from a solution of pure lactose, using the modification of the Hagedorn-Jensen method (cf. A., 1923, ii, 265) described by Issekutz and Both (cf. A., 1927, 600).

H. J. DOWDEN.

Determination of chlorine and sugar in small quantities of milk. F. MÜNCHBERG (Milch. Zentr., 1930, 59, 161—163).—In determining chlorine in milk, 2 c.c. of halogen-free nitric acid (*d* 1.40), 1 c.c. of 0.1*N*- (or 5 c.c. of 0.02*N*-) silver nitrate, and 1 c.c. of the sample are heated together to boiling. Then 10% potassium permanganate solution is added drop by drop to the boiling liquid till it becomes clear. The liquid is cooled by dilution with 30 c.c. of distilled water and, after addition of 0.5 c.c. of saturated iron ammonium alum solution, the excess of silver is titrated with 0.02*N*-ammonium thiocyanate. (1 c.c. of 0.02*N*-silver nitrate \equiv 0.0007092 g. Cl.) In determining the sugar content, 10 c.c. of the sample are shaken well with 1 c.c. of 20% trichloroacetic acid for several minutes and filtered clear through a dry filter. The rotation observed in the Zeiss pocket polarimeter multiplied by 2.2 gives the percentage sugar content. The results are slightly lower than those obtained by the iodometric and gravimetric methods of determining sugar. W. J. BOYD.

Formulae for the direct calculation of the sucrose and lactose content of milk-chocolate.

A. RINCK and E. KAEMPF (Z. Unters. Lebensm., 1930, 59, 81—90).

—The method previously employed for the determination of starch syrup and sucrose in marmalade (cf. B., 1922, 191A) has been applied to the determination of sugars in milk-chocolate. A sample (10 g.) is freed from fat with chloroform-ether, and, after drying, the residue is extracted with water containing ammonia and lead acetate to precipitate albumin. The polarisation of the extract is measured before and after Clerget inversion, and for the purpose of determining the total sugar content by the method of Welmans and Steinmann the polarisation of 10 g. of chocolate, subsequently freed from fat, dried, and dissolved in 100 c.c. of water, is also measured. The following formulae have been derived, in which polarisation value = *P*, provisional total sugar = *S*. [Owing to the difference between the polarisation units for sucrose and lactose (1.33 and 1.051, respectively), a table of factors has been calculated for use in deducing the total sugar content.] Thus, (i) *P* before inversion/factor = *S*; (ii) *S*/1.59 = volume of total sugar; (iii) [*S*/(100 - volume)] 1000 = actual total sugar (%); (iv) [(*S* × 0.43) + *P* after inversion]/0.1481 = provisional lactose (%); (v) [(*S* × 1.051) - *P* after inversion] 0.1481 = provisional sucrose (%); (vi) 10*S*: actual total sugar (%) = provisional lactose (%): actual lactose (%). The percentage sucrose is given by the difference between total sugar and the percentage of lactose. Values for milk solids and cacao can also be derived. H. J. DOWDEN.

Effect of heat on the sugars entering into the composition of chocolate pastes. R. LECOQ (J. Pharm. Chim., 1930, 11, [viii], 522—529).—Heating of chocolate pastes at temperatures ranging from 50° to 120° causes a distinct caramelisation of the sugar, which is expressed by an increase in the proportion of reducing sugars at the expense of the initial sucrose. This caramelisation

depends on the duration of treatment and the temperature reached. It is produced to a small extent in cooking chocolate, but it is in overheated chocolate fondants that it is mostly shown, when the inverted sugar may amount to 25% of the total sucrose.

B. W. TOWN.

Soya-bean extract residues from different processes. P. MALKOMESIU and W. SCHRAMM (Landw. Versuchs-Stat., 1930, 110, 33—47).—Examination of various samples of soya-bean cakes shows that the method of production does not affect the digestibility of the product. Differences in the digestible protein contents of various samples are traceable in part to differences in the original material and in part to the extraction method. The fat and lecithin contents are entirely controlled by the process used. A. G. POLLARD.

Pollen analysis of honey. I. C. GRIEBEL (Z. Unters. Lebensm., 1930, 59, 63—79).—To assist in the detection of imported honey the forms and structures of a large number of different types of pollen, both native and foreign, are described and illustrated by photomicrographs. H. J. DOWDEN.

Deterioration and spoilage of honey in storage. G. E. MARVIN (J. Econ. Entomol., 1930, 23, 431—438).—Granulation of honey in storage favours fermentation by sugar-tolerant yeasts with the production of carbon dioxide, alcohol (rarely > 5%), and a non-volatile acid. Such fermentation may be prevented by heating to 71°, sealing, and cooling rapidly to avoid discoloration. A. G. POLLARD.

Methods used to detect heated honeys. G. H. VANSSELL and S. B. FREEBORN (J. Econ. Entomol., 1930, 23, 428—431).—The Fliche test for heated honey is unreliable since small amounts of hydroxymethylfurfuraldehyde may be produced without heat, especially in solidified samples. The diastatic activity of honey depends on the amount of pollen present, and, unless a simultaneous pollen count is made, is insufficient as a test of heating. A. G. POLLARD.

Viscosity of pectin sols. II. Effects of citric acid and various sugars. A. OHN (Ind. Eng. Chem., 1930, 22, 635—639; cf. B., 1927, 91).—The effect of citric acid, dextrose, lævulose, sucrose, or lactose on the viscosity and gelation of non-heated pectin sols has been investigated. Citric acid decreases the viscosity of pectin or pectin-sucrose sols, whilst the carbohydrates increase the viscosity of pectin sols; sucrose has the greatest effect. The pectin-acid-carbohydrate jellies containing sucrose are firmer and more stable than those containing dextrose, lævulose, or lactose. Crystalline formation appears at a lower hydrogen-ion concentration in sucrose than in dextrose or lactose jellies, whilst under similar conditions in lævulose jellies no crystalline formation occurs. L. S. THEOBALD.

Determination of water. LEPPER.—See VII.

PATENTS.

Manufacture of bread. J. MATTI, Assr. to AUGMENTINE HOLDING SOC. ANON. (U.S.P. 1,750,720, 18.3.30. Appl., 5.4.28. Ger., 21.4.27).—An emulsion of paraffin oil (55%), glycerin (35%), and water (10%) is added to

a bread dough in the proportion of 0.3% of the flour weight. The process is claimed to improve the quality of the bread and to give an increased yield.

E. B. HUGHES.

Production of bakery goods, pastries, confectionery, etc. H. BOLLMANN and B. REWALD (B.P. 328,075, 23.2.29).—In the preparation of such products, egg yolk and the whole or part of the fat normally employed may be replaced by a mixture comprising food fat and an amount of lecithin (from soya bean) not less than 5% of the fat. E. B. HUGHES.

Self-preserving [condensed] milk product. T. O'CONNOR (U.S.P. 1,749,153, 4.3.30. Appl., 8.2.26).—The product, suitable for use in bakeries, comprises 7—14% of sodium chloride, not less than 28% of milk solids, about 20% of dextrose, and not less than 25% of water. L. A. COLES.

Flavouring tea. L. ARONSON and A. FELDBLET (U.S.P. 1,750,768, 18.3.30. Appl., 21.12.27).—Tea is impregnated with citric acid solution and dried, then exposed for $\frac{1}{2}$ hr. to the vapours of essential oils (lemon, orange, etc.) dissolved in alcohol. After redrying it is blended with an equal weight of untreated tea. E. B. HUGHES.

Treatment of seaweed. A. CARTIER and P. GLOESS (F.P. 633,696, 8.9.26).—Seaweed is heated with a suspension of magnesia in water, whereby the unpleasant odour and taste is removed. The product is suitable for the manufacture of foodstuffs and electrical insulators. A. R. POWELL.

Drying apparatus (U.S.P. 1,749,451). **Mixing machine** (U.S.P. 1,745,993). **Congealing of liquid substances.** (B.P. 329,129).—See I. **Table salt** (B.P. 312,088).—See VII. **Vitamin-A** (B.P. 306,881).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Tests for aloes. A. H. WARE (Pharm. J., 1930, 124, 596—597).—The colour reactions described by Schorn (B., 1930, 485) are unsatisfactory. Details of improvements in tests previously described by the author (A., 1925, i, 1122) are given. H. E. F. NOTTON.

Oil from cascara sagrada. A. JERMSTAD (Pharm. Acta Helv., 1929, 4, 90—98; Chem. Zentr., 1930, i, 845).—When the residue from the alcoholic extraction of cascara sagrada is distilled with steam an oil is collected which has d_{20}^{15} 0.8923, n_D^{20} 1.4568, acid value 3.7, saponif. value 189.4, iodine value 86.2. It contains palmitic, oleic, linoleic, and linolenic acids; glycerol is not present. The unsaponifiable matter contains docosane and an oil, b.p. (?) 285—289° or (?) 185—189°, n_D^{19} 1.4901. A. A. ELDRIDGE.

New constituent of peppermint oil. H. CARLES (Parf. mod., 1930, 22, 615—637; Chem. Zentr., 1930, i, 759).—Peppermint oil from the 1927 crop had an exceptionally high density (0.915) and α_D up to -10° ; it contained up to 10% of a constituent having d_{20}^{15} 0.965, $\alpha_D + 81^\circ$, n_D^{20} 1.4807, b.p. 95°/20 mm. or 196°/atm. pressure. A. A. ELDRIDGE.

Portuguese petitgrain oil. L. S. GLITCHITCH and V. R. NAVES (Parfums de France, 1929, 7, 223—228;

Chem. Zentr., 1930, i, 604).—Sweet orange (branch) oil from Algeria, d_{20}^{15} 0.8585, $\alpha_D^{20} + 40^\circ 55'$, n_D^{20} 1.4726, contained 2.33% of esters (as linalyl acetate), together with palmitic and geranic acids, guaiacol (?), methyl methylantranilate and anthranilate, pyrrole, furfuraldehyde, citral, pinene, dipentene, *d*-limonene, a terpene, d_{20}^{15} 0.8274, n_D^{20} 1.4812, $\alpha_D + 5^\circ 42'$, *d*-linalool, *d*-terpineol, geraniol, nerol, acetic and higher fatty acids, bisabolene (?), farnesol (?), and only traces of camphene.

A. A. ELDRIDGE.

Philippine eucalyptus oil. A. P. WEST and H. TAGUIBAO (Philippine J. Sci., 1930, 42, 1—11).—The oil from several species of eucalyptus grown in the Philippines has been examined. The highest yields of oil (3½—5%, which were greater than those from the same species grown in Australia) were obtained from *E. globulus*, *tereleornis*, *polyanthemos*, and *citriodora*. For reliable comparison the yields of oil (which is almost entirely located in the leaves) should be calculated on a moisture-free basis. The constants and cineol content of the oil of *E. globulus* compared favourably with the corresponding figures for Australian oil from the same species.

E. LEWKOWITSCH.

Composition of the oil of *Dracocephalum Moldavica*. L. B. N. RUTOVSKI and I. V. VINOGRADOVA (Riechstoffind., 1929, 4, 137—138; Chem. Zentr., 1930, i, 758).—The oil (yield 0.01—0.17%) has d_{20}^{20} 0.9038—0.9491, $\alpha_D - 5.92^\circ$ to 0° , n_D^{20} 1.4764—1.4874, acid value 2.32—18.64, ester value 34.64—170.33, ester value after acetylation 204.0—288.11, aldehyde content 25—68%. Citral, thymol, geraniol, nerol, an aldehyde, m.p. 60—62°, a terpene (? limonene), and a monocyclic sesquiterpene are present.

A. A. ELDRIDGE.

Determination of allylthiocarbamide in black mustard. R. MEESEMAECKER and J. BOIVIN (J. Pharm. Chim., 1930, [viii], 11, 478—784).—5 G. of finely-powdered mustard (*Brassica nigra*) and 100 c.c. of water are heated in a closed flask on a water-bath at 68° for 1 hr.; 100 c.c. of ammonia solution are then added and the mixture is kept for 15 min. The solution is cooled, made up to 200 c.c. with distilled water, 20 c.c. of Courtonne's reagent are added, and, after shaking, the liquid is filtered. 100 C.c. of the filtrate are neutralised, then acidified with 5 c.c. of 0.5*N*-sulphuric acid, 10 c.c. of 0.1*N*-iodine are added, and the mixture is set aside for 15 min. After the addition of 10 c.c. of benzene, the excess of iodine is titrated against 0.05*N*-thiosulphate. The process is based on the fixation of iodine by thiosinamine. (1 C.c. of 0.1*N*-iodine is equivalent to 0.0058 g. of thiosinamine and 0.00485 g. of allylthiocarbamide.) The maximum of allylthiocarbamide is obtained by maceration at 68° and the above method allows the production to be followed under varying conditions. This has been done with different varieties of mustard. For an "oil-free" meal the optimum temperature for maceration to obtain the highest yield of essence is 40°.

E. H. SHARPLES.

PATENTS.

Manufacture of stable emulsions of paraffin. E. GLÜCKSMANN, Assee. of A. MÜLLER (G.P. 446,598, 15.7.25. Cf. B.P. 255,456; B., 1927, 892).—Emulsions

having the consistency of a soft fat and stable up to 40° are obtained by the process disclosed in the B.P.; they may be flavoured by addition of essences to the water in which the paraffin is emulsified.

A. R. POWELL.

Preparing vitamin-A in free or combined condition. T. SHIMIZU (B.P. 306,881, 18.2.29).—The sterol-free unsaponifiable matter of substances rich in vitamin-A is treated with suitable bile acids, which form a crystalline vitamin-A-choleic acid complex ($C_{24}H_{40}O_4$)₅. $C_{27}H_{44}O_2$. H_2O , m.p. 179°. This compound is stable towards oxygen and insensitive to light; on treatment by the usual methods, it is decomposed into choleic acid and crystalline vitamin-A, $C_{27}H_{44}O_2$. H_2O , m.p. 187°. The above operations are preferably carried out in absence of oxygen.

E. H. SHARPLES.

Manufacture of the hormone of the anterior lobe of the hypophysis. SCHERING-KAHLBAUM A.-G. (B.P. 329,605, 14.10.29. Ger., 13.10.28).—Lime is removed from the urine of pregnant warm-blooded animals by treatment with a soluble oxalate and, after removal of the precipitate, the hormone is precipitated from the solution in the usual manner.

E. H. SHARPLES.

Preparation of compounds of alkaline-earth nitrites with methylxanthines. H. P. KAUFFMANN (B.P. 311,231, 2.5.29. Ger., 7.5.28).—Compounds useful as vascular agents are obtained by evaporating a solution of an alkaline-earth nitrite to which caffeine, or an alkaline-earth salt of theobromine or theophylline, or a mixture of alkaline-earth hydroxide and theobromine or theophylline, has been added.

C. HOLLINS.

Manufacture of double compounds of the acridine series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,212, 18.1.29).—The double salts containing both 3:7-diaminoacridine salt (*N* = 5) and 3:7-diamino-*N*-alkylacridinium salt are much more soluble in water than is either component. Hot aqueous or alcoholic solutions of 3:7-diamino-*N*-methylacridinium chloride and 3:7-diaminoacridine hydrochloride are mixed and the double salt is salted out or recovered by evaporation; or the acridine base is added to the acridinium hydrogen sulphate in aqueous suspension at 60°.

C. HOLLINS.

Manufacture of pharmaceutical products. W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,760,781, 27.5.30. Appl., 16.12.27. Ger., 20.12.26).—See B.P. 282,453; B., 1929, 958.

Purified ethylene (U.S.P. 1,741,559).—See III. **Lyophilic products** (B.P. 329,305). **Emulsions** (B.P. 329,266).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Developers and development. I. Physico-chemical basis of a rational composition for developer solutions. K. TSCHIBISSOV. II. Metol-quinol and metoquinone developers. III. Effect of dilution of developer on photographic properties.

K. TSCHIBISSOV and V. TSCHELZOV (Kinotechnik, 1929, 11, 227—231, 315—321, 373—376; Chem. Zentr., 1930, i, 473—474).—I. The relations between chemical constitution and developing power are considered, together with concentrations applicable to practice and the use of alkali hydroxide.

II. Substitution of sodium hydroxide for carbonate increases the developing power of quinol, but diminishes that of metol.

III. Variable results were obtained owing to hydrolysis, oxidation, inadequate diffusion, etc. Solutions of different concentrations do not behave regularly towards changes of temperature. A. A. ELDRIDGE.

Test for thiosulphate. BAINES.—See VII.

PATENTS.

Preparation of light-sensitive layers. M. P. SCHMIDT and W. KRIEGER, ASSRS. to KALLE & Co. A.-G. (U.S.P. 1,760,780, 27.5.30. Appl., 7.8.28. Ger., 6.9.27).—See B.P. 296,725; B., 1930, 303.

Desensitisation of silver bromide emulsions and plates or films covered with such emulsions. B. HOMOLKA, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,760,756, 27.5.30. Appl., 20.12.27. Ger., 15.6.27).—See B.P. 292,140; B., 1928, 875.

[Lenticular] films for colour photography. KODAK, LTD., ASSEES. of H. E. HASTINGS (B.P. 330,151, 21.6.29. U.S., 2.11.28).

Printing of kinematograph films. FILM OZAPHANE SOC. ANON. (B.P. 330,024, 14.3.29. Fr., 24.1.29).

[Preparations of records for] coloured talking-picture photography. BRIT. TALKING PICTURES, LTD., ASSEES. of L. DE FOREST (B.P. 319,779, 26.9.29. U.S., 28.9.28).

Cellulose ester waste (B.P. 311,368).—See V.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Explosive compositions. A. STETTACHER (B.P. 312,316, 14.5.29. Switz., 24.5.28).—Plastic explosive mixtures for use especially for filling mines, torpedoes, and detonators comprise 70—90 pts. of pentaerythritol tetranitrate precipitated in a finely-crystalline form, and 30—10 pts. of nitroglycerin and/or glycol dinitrate (cf. B., 1929, 152, 998). L. A. COLES.

Composition rods igniting by friction [match substitutes]. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 329,796, 25.4.29).—The rods, which can be extinguished and re-ignited repeatedly, comprise an organic foundation yielding practically no ash, having a speed of combustion less than that of weakly nitrated cellulose, and containing, *e.g.*, cellulose nitrate or acetate mixed with ammonium oxalate, camphor, naphthalene, etc., and an ignition mixture free from phosphorus, *e.g.* potassium chlorate and sulphur; binders, materials capable of regulating combustion, fillers, pigments, etc. may also be added. L. A. COLES.

Production of nitric acid (B.P. 329,427).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Red-water trouble and the remedy at West Palm Beach. L. O. WILLIAMS (J. Amer. Water Works' Assoc., 1930, 22, 791—795).—The presence in the finished water of 9 p.p.m. of dissolved oxygen and 5 p.p.m. of carbon dioxide caused severe corrosion in the iron mains which, owing to lack of systematic flushing, became coated with a soft mass containing ferrous carbonate, ferric hydroxide, and ferric oxide. Small portions of this material dislodged by the normal variations in the rate of flow were the cause of frequent complaints of red water. By the addition of lime, a protective coating of calcium carbonate has been deposited in the mains and, as the portions of rust scale which are not thus coated have proved to be much harder than formerly, the complaints have ceased. C. JEPSON.

Identification and determination of nitrates in water. G. BINI (Atti R. Accad. Lincei, 1930, [vi], 11, 593—596).—An examination of reagents suggested for the identification of nitrates in the presence of nitrites shows that quinolsulphonic acid, for which details of preparation are given, is the most suitable. This reagent gives with nitrates, according to concentration, colours varying from pale green to brown, and will detect 1 pt. of nitrate in 10^5 pts. of water. The presence of 20 pts. per 10^5 of nitrite is without influence. In this respect this reagent is to be preferred to pyrogallolsulphonic acid. F. G. TRYHORN.

PATENTS.

Utilising [for water sterilisation] the oligodynamic action of metals and metal compounds. G. A. KRAUSE (B.P. 306,547, 20.2.29. Ger., 24.2.28).—Oligodynamically active metals, alloys, and compounds are deposited by heat upon carriers, containers, etc. for use in sterilising water; *e.g.*, ceramic material, silica gel, clay, charcoal, etc. impregnated with solutions containing compounds of silver, gold, thallium, copper, etc. is heated to decompose the compounds, or alternatively, the metals are applied by spraying or electrical atomisation processes. [Stat. ref.] L. A. COLES.

Production of a balanced environment for fish life. J. T. and M. E. TRAVERS, ASSRS. to OHIO SANITARY ENG. CORP. (U.S.P. 1,747,804, 18.2.30. Appl., 18.5.28).—The water contains travertine, composed of calcium carbonate and clay in the process of decay, preferably having a porosity not less than 100% and/or $d\ 2.3-2.5$, and/or a calcium carbonate content of 90—96%. The travertine is capable of adsorbing the noxious odour-producing compounds in the excrement of fish. S. K. TWEEDY.

Disinfecting or sterilising devices for use in connexion with bottle and like washing apparatus. R. HAIMES (B.P. 330,249, 2.3.29).

Water-softening apparatus. UNITED WATER SOFTENERS, LTD., and H. S. LAWRENCE (B.P. 330,120, 17.5.29. Addn. to B.P. 302,744).

Air filters etc. (B.P. 328,948, 329,099, and U.S.P. 1,747,694).—See I. Water-softening agent (U.S.P. 1,750,817).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUGUST 15 and 22, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat transmission: modern methods of expressing convection data. M. FISHENDEN (Gas World, 1930, 92, 632—637).—Mathematical treatment of heat transfer is applied to problems of the coal-gas industry. Data and calculations are given for radiations of varying wave-length, natural or forced convection, and heat transfer in tubes or through partition walls. A number of results, from different observers, have been correlated to give figures for natural convection from horizontal cylinders suspended in gases, and for forced convection in tubes. An example is given of the application of calculations, derived in this way, for the case of hot air flowing through a horizontal tube, with or without insulation.

R. H. GRIFFITH.

Maintenance and working of simple types of autoclaves. A. W. C. HARRISON (Ind. Chemist, 1930, 6, 244—248; cf. B., 1930, 643).—Packing materials, discharge arrangements, pipe-lines, and accessories are discussed. Processes using ammonia entail the addition of 7 or 8 times as much as that theoretically needed, and the greater part of this can be recovered by blowing off and condensing at a suitable point during the cooling down. Two valves should be used on the ammonia connexion to avoid leakage and consequent blockage. Gas-heating installations are described and a detailed description is given of the conversion of a batch of Schäffer acid into Brønner acid by amination with ammonia and ammonium sulphite.

C. IRWIN.

Construction and use of homogenisers. C. H. MÖLLERING (Chem. Fabr., 1930, 239—240).—The principle of all homogenising machines consists in the forcing of the material through fine passages, whereby the particles in suspension or emulsified are brought to a uniform size by friction. Power is economised by working at higher temperatures. Pressures of 150—200 atm. are usual, and are obtained either by a three-stage compressor or by centrifugal force. A machine of each type is described. The pressure obtained in centrifugal machines is lower than with pumps, but as they also act as emulsifiers by mechanical mixing the results are equal. The process of homogenisation was first applied to milk, but it is also employed in the margarine, lacquer, and artificial silk industries, and in connexion with pharmaceutical chemicals and cosmetics.

C. IRWIN.

Porous medium for flotation cells. F. C. DYER and H. L. McCLELLAND (Univ. Toronto Fac. Appl. Sci. Eng., Eng. Res., Bull. No. 8, 1928, 419—424).—A résumé of the defects of diaphragms made of canvas, rubber, or ceramic material is given. Since resistance to abrasion was a highly desired quality, linoleum was con-

sidered most worthy of investigation owing to its known good qualities on concentrating tables. Porous linoleum was unobtainable in a ready manufactured state, and drilling or punching was too slow and unsatisfactory owing to the holes closing up; an encouraging result was obtained by stripping off the canvas backing, cutting into strips, and reassembling with a grooved face to a smooth face. As, naturally, the grooves were of different sizes and the distribution of air was not uniform, other grooves were cut by a multiple saw and the result was good, except that it was impossible to prevent the adhesive (durofix) from closing up some of the holes. Experiments were then tried starting with an uncured mix of cork and oils which was granulated, heated between rolls till soft and sticky, and then rolled, the grooves being formed by ridges on one roll; it was found unnecessary to roll the material in the form of strips because it could be guillotined without closing up the ends of the grooves. The finally adopted roll was then formed with only circumferential ridges about 0.0012 in. high and wide, spaced $\frac{3}{8}$ in. apart; this paired with a smooth roll and formed sheets $\frac{1}{8}$ in. thick, which were then sheared at right angles to the grooves into strips $\frac{3}{8}$ in. wide, the reassembled sheet then being $\frac{3}{8}$ in. thick and having the holes spaced at $\frac{1}{8}$ in. apart one way and $\frac{3}{8}$ in. the other. The natural stickiness was sufficient for adherence, and the curing was effected in an oven for several days. Besides more obvious advantages, scale-forming liquors cannot approach the walls of the holes through the substance of the material, so that they do not choke even though scale may build up harmlessly on the surface between the holes. Although linoleum is resistant to a large number of solutions, the authors suggest that the same method of manufacture may be applied to other materials such as tar products (congolem), soft metals, glass, rubber, or clay.

B. M. VENABLES.

Bacterial filtration by the Berkefeld filter and the size of the pores. H. HOEK (Chem. Fabr., 1930, 249—250).—These filters consist of ceramic cylinders, and as the pore diameters may vary it is desirable to test their suitability for retaining bacteria. A number may be immersed in water and connected to air under gradually increased pressure, the pressure at which each commences to pass bubbles being noted. From this the diameter of the largest pores can be calculated. Experiments on the filtration of bacterial cultures are described which indicate that satisfactory cylinders should resist an air pressure of at least 0.55 atm. without passing bubbles. The cylinder dimensions are immaterial; it is the maximum pore size, not the average, that is important.

C. IRWIN.

* The remainder of this set of Abstracts will appear in next week's issue.

Pilot-plant fractionating column. R. McCULLOUGH and L. D. GITTINGS (Ind. Eng. Chem., 1930, 22, 584—587).—A fractionating column suitable for semi-works' scale operation is described. It consists of a 3-in. pipe, 9 ft. long, packed with $\frac{1}{4}$ -in. glass Raschig rings. It is fitted with a hot-air jacket, and is itself also electrically heated. The boiler consists of a piece of 12-in. pipe. The upper 2 ft. of the column is air-cooled and acts as a partial condenser. An air-cooled final condenser is also fitted. The column was used to fractionate nitrodiphenyls at 30-mm. pressure which it effected at 3.0 lb./hr., and chlorodiphenyls at atmospheric pressure for which the rate obtained was 2.1 lb./hr.

C. IRWIN.

Laws of motion of particles in a fluid. R. G. LUNNON (Trans. Inst. Min. Met., 1930, 38, 402—420).—The limitations of Rittinger's law are greater than has previously been assumed. Stokes' law (resistance proportional to speed) holds within 5% when vd is between 0 and 0.002 for water and between 0 and 0.03 for air (v = velocity and d = diam. of sphere in c.g.s. units). Similarly, Allen's law (resistance proportional to the $3/2$ power of the speed) holds when vd is between 0.3 and 3 for water and between 5 and 50 for air. Finally, Newton's law (resistance proportional to the square of the velocity) holds when vd is between 10 and 1000 for water and between 150 and 15,000 for air. The equations for the motion of a sphere are applicable to the motion of irregular particles with certain small changes. The only exception occurs when the particles are closely packed together and are just lifted by a rising fluid.

W. E. DOWNEY.

Separation of gases by adsorption with charcoal. K. PETERS and K. WEIL (Z. angew. Chem., 1930, 43, 603—612).—The adsorption isotherms of argon, krypton, and xenon at temperatures from -80° to 0° have been determined. At pressures from 0 to 100 mm. Hg these obey the relationship $a = \alpha^{1/n}$, where a is the mass of adsorbed gas, p the pressure over the adsorbed phase, and α and n are constants. At higher pressures this no longer holds good, but as separation by adsorption is facilitated by low pressure, only this pressure range is considered. From the experimentally obtained isotherms, isobars and "isosteres" (analogous to vapour-pressure curves) for the different gases are obtained. The activated carbon used for these experiments was Bayer A carbon. The optimum conditions for separation are given by these curves, and this may be carried out by either fractional adsorption or fractional "desorption." In the first case due consideration must be given to time of contact, or adsorption will not be complete; if desorption be used it is essential to select the correct temperature for the particular case. It is readily possible to effect a complete separation of the three gases mentioned by these methods and also to separate traces of a less volatile component from the more volatile (e.g., radium emanation from liquid air), when present in quantity too small for chemical separation.

C. IRWIN.

Modern fire extinguishers. E. BIESALSKI (Z. angew. Chem., 1930, 43, 603—608).—Dry fire extinguishers invariably depend on sodium bicarbonate to which a little kieselsuhr has been added. This is dis-

tributed by a current of carbon dioxide from a container filled with the liquefied gas. Carbon dioxide snow may also be used, produced by the expansion of the liquefied gas through a jet. Carbon dioxide gas is suitable for permanent sprinkler installations. Liquid fire extinguishers of the acid and soda type are most suitable for extinguishing burning solid materials where penetration is an advantage. Other makers employ water and compressed carbon dioxide or a concentrated solution of sodium or potassium carbonate. The latter solution will extinguish a heavy oil, when burning, by emulsification. "Erkalen" is a water-soluble organic compound marketed for extinguishing fires of lignite and cork dust. Carbon tetrachloride has many advantages, and the alleged danger of phosgene formation is negligible except perhaps in confined spaces. Methyl bromide is an efficient fire extinguisher, but is believed to be dangerous in use. Foam extinguishers employ carbon dioxide with either saponin or other foam producers in aqueous solution. The volume of foam is 8—12 times the volume of the water used. Its electrical conductivity, however, makes it unsuitable in presence of high voltages. The Minimax foam generator employs sodium bicarbonate and aluminium sulphate, or oxalic acid, to generate carbon dioxide, and is connected to a water main.

C. IRWIN.

Determination of humidity. G. ALIVERTI (Nuovo Cim., 1929, 6, cxvii—cxxiii; Chem. Zentr., 1930, i, 1009).—A discussion of the use of various types of hygrometer.

A. A. ELDRIDGE.

Graphical methods in the gas industry. SCHUMACHER.—See II. Measurement of colour and gloss. DESBLEDS.—See V. Silica grinding. CAWOOD.—See VII.

PATENTS.

[Boiler] furnaces. J. J. DESCHAMPS (B.P. 330,200, 1.10.29. Fr., 1.10.28).—The water-tubes forming the walls of a boiler furnace are provided with fins of such a diameter and pitch that ash accumulates on them to such an extent as to cover the whole surface of the tube proper. In the case of nearly horizontal tubes for water-bottoms, the fins are longitudinal blades, radial on the upper parts and curved on the lower.

B. M. VENABLES.

[Boiler] furnace. C. J. HUBER, ASSR. to FLYNN & EMMICH Co. (U.S.P. 1,751,861, 25.3.30. Appl., 2.6.25).—A furnace has a coking shelf adjacent the opening from the supply hopper and an arch immediately above it. Air is drawn through a hollow metallic girder in the arch and between double walls of the hopper into the fire.

B. M. VENABLES.

Furnaces for heating articles. H. A. DREFFEIN (B.P. 302,652, 27.11.28. U.S., 19.12.27).—A furnace for heating metal plates and the like is provided with a combustion chamber down a longitudinal side of the goods chamber which near the burner is in unobstructed communication with the latter, but owing to the longitudinal velocity of the flame the heat reaches the goods only by radiation, and there may even be a back-circulation into the combustion chamber at this point. Further along the gases are permitted to enter the goods chamber through ports in a dividing wall.

B. M. VENABLES.

[Heating] furnaces [for metal bars etc.]. INTERNAT. CONSTRUCTION CO., LTD., E. P. JONES, and J. H. MIDDLETON (B.P. 330,160, 2.7.29).—A single chamber for heating bars and two chambers for heating the plates rolled therefrom are combined with two fireplaces in one structure, the bar chamber lying transversely between the fires and the plate chambers.

B. M. VENABLES.

Apparatus for heating [metal sheets etc.]. E. M. FREELAND and F. MCGOUGH, Assrs. to FOLLANSBEE BROS. Co. (U.S.P. 1,750,884, 18.3.30. Appl., 31.5.27).—A reverberatory furnace for heating sheets etc. has the hearth formed of live-rolls which are themselves heated from below by the products of combustion passing through flues, though their shafts may be water-cooled.

B. M. VENABLES.

Heat exchanger. (A) W. M. CROSS, (B) W. B. PHILLIPS, Assr. to W. M. CROSS (U.S.P. [A] 1,751,725 and [B] 1,751,757, 25.3.30. Appl., [A] 7.1.26, [B] 14.1.28).—In (A) corrugated sheets are arranged ridge to ridge in pairs forming longitudinal passages for the inner fluid. The pairs are kept apart by spacers embossed on the sheets at the ends and intermediately, thus forming transverse passages for the outer fluid. (B) In an exchanger as above, separate spacers are provided and the preferred arrangement of the adjacent sheets of different pairs is ridge to valley, so that the transverse passages have parallel but sinuous walls.

B. M. VENABLES.

Drying apparatus. A. T. OAKLEY and P. LUPKE, JUN., Assrs. to ESSEX RUBBER Co. (U.S.P. 1,750,813, 18.3.30. Appl., 10.11.26).—A rotary drum dryer for materials which are sensitive to heat, such as pulverised reclaimed rubber, is provided with interior stationary conduits for drying and moist gases, respectively, perforated throughout their length and supported on a structural steel bridge extending right through the drum in such a manner that they may expand independently of each other.

B. M. VENABLES.

Drying ovens. S. L. GROOM (B.P. 329,972, 24.1.29).—A mechanism is described by which plate-like objects which can be handled only on one side are transferred from the upward run of a vertical conveyor to the downward run without turning over.

B. M. VENABLES.

Dryer. C. J. BLEIL (U.S.P. 1,749,525, 4.3.30. Appl., 12.4.28).—The apparatus comprises a number of superposed circular hearths, inclined alternately to the centre and circumference, with rakes driven by an axial shaft; the transfer passages through the hearths are provided with double doors operated out of phase by the stirrers, so that they form air-locks permitting different conditions to be maintained in different chambers. Some chambers may be heated by wet steam in coils, others by dry steam mixed with the material, and some chambers may be subjected to a vacuum.

B. M. VENABLES.

Drying process and apparatus. A. A. CRIQUI, Assr. to BUFFALO FORGE Co. (U.S.P. 1,751,472, 25.3.30. Appl., 1.5.26).—The material is subjected alternately to strong blasts of air in a direction which holds it on its support, then to feebler currents in an opposite direction; e.g., it may be placed on a pervious conveyor

and exposed to concentrated downward and diffused upward currents of heated air. B. M. VENABLES.

Pulverising machine. W. J. ARMSTRONG, Assr. to JEFFREY MANUF. Co. (U.S.P. 1,751,611, 25.3.30. Appl., 2.10.26. Renewed 12.6.29).—A disintegrator is provided with a pocket in advance of the feed hopper and out of the path of the hammers, which pocket is fitted with an abutment and a shelf in addition to two walls at right angles, all of which surfaces are intended to deflect and crush material flung by the hammers.

B. M. VENABLES.

Pulverising machine. W. K. LIGGETT, Assr. to JEFFREY MANUF. Co. (U.S.P. 1,751,000, 18.3.30. Appl., 20.5.18. Renewed 30.7.29).—A disintegrator of the hammer type is fed from the upper circumference; alongside the hopper, but outside the zone swept by the hammers, is a crushing zone in which particles that are too large to enter the hammer zone are swept round like an eddy.

B. M. VENABLES.

Pulveriser mill. R. O. GROMAN, Assr. to FULLER-LEHIGH Co. (U.S.P. 1,750,636, 18.3.30. Appl., 11.2.27).—The invention relates to methods of obtaining even distribution of air to a mill which may be of the ball-and-ring type.

B. M. VENABLES.

Abrading and crushing machine. P. L. CROWE (U.S.P. 1,750,573, 11.3.30. Appl., 27.7.28).—The machine has an electric motor built round it and the draught that carries away the ground coal or other material has previously cooled the motor. The abrasion is effected inside a conical hopper with apertures for the exit of ground coal and with an internal worm which tends to raise any material sliding along it. The moving unit comprises a number of double conical elements assembled on a vertical shaft, the largest diameter of one of them nearly closing the bottom of the hopper. The ground material passes mainly through the walls of the hopper to a fan surrounding it.

B. M. VENABLES.

Machine for kneading, crushing, and similar operations. B. ZETTERGREN (U.S.P. 1,750,558, 11.3.30. Appl., 2.10.28. Swed., 23.12.27).—A mill in which a roller is dragged round inside a cylindrical casing by means of a crank and pair of links is described.

B. M. VENABLES.

Crusher heads for homogenising apparatus. A. P. ANDERSEN (B.P. 312,351, 3.5.29. Denm., 26.5.28).—Each of the grinding surfaces (with central feed) is provided with concentric grooves which are not opposite each other.

B. M. VENABLES.

Determination of moisture in aggregate [by unskilled persons]. R. E. ROBB (U.S.P. 1,749,606, 4.3.30. Appl., 17.8.28).—In a vessel suitable for suspending from a weighing-beam, water is placed to a definite level, determined by a lower draw-off cock. The lower cock is closed and a definite weight of dry aggregate of the same quality as that to be tested is added to the vessel and the water that runs out of a higher cock is collected in a measuring cylinder. The gross weight of the weighing vessel is then counterpoised by adjusting a sliding weight on the scale beam and the weight is clamped in that position. The standard aggregate is then discarded and the weighing vessel refilled with water up to the lower cock, then moist

aggregate is added (the overflow being caught) until the vessel again balances; there will then be present the same weight of dry aggregate as was taken of the standard, but the volume of water that has overflowed out of the upper cock will be greater by exactly the amount of original moisture in the sample, so that the height of water in the measuring cylinder will indicate directly on a scale moisture percentage of dry weight. No calculation and no weighing other than the occasional dry standard are necessary. B. M. VENABLES.

Material classifying devices. H. G. LYKKEN (B.P. 330,145, 14.6.29. U.S., 5.12.28).—An air separator is placed above a disintegrator and comprises a number of cylindro-conical walls which are co-axial, but not parallel, and adjustable relatively to each other. A bladed element to produce whirling is also provided which telescopes on the cylindrical part of the inner baffle to vary the effective area of the passages between the blades. B. M. VENABLES.

[Compressed air] atomisers and powder blowers. A. S. FISHER & NIELSEN, and P. A. FISHER (B.P. 330,022, 12.3.29).—For a paint or powder sprayer, a trigger valve is described which prevents dribbling at the paint jet. B. M. VENABLES.

Separation of materials of different sp. gravities. T. M. CHANCE and H. O. STAPLES (U.S.P. 1,750,090, 11.3.30. Appl., 26.3.29).—It is known to separate coal from rock by flotation in a fluid medium composed of sand and water kept in a state of agitation sufficient for it to behave as a liquid of intermediate density. The principle is extended to elevate the coal alone to any desired point, *e.g.*, from the mine to the surface, leaving the waste matter behind. An apparatus for effecting the separation and transport of coal or other materials is described. B. M. VENABLES.

Separation of solids from gases and liquids. J. E. POLLAK. From F. ARLEDTER (B.P. 329,992, 24.1.29).—The fluid is admitted through the wall, at an intermediate level, of a funnel-shaped tank which is provided with an outlet for solid matter at the bottom and peripheral overflow for clear fluid at the top. The fluid may be admitted tangentially to produce a swirling motion. [Stat. ref.] B. M. VENABLES.

Releasing the pressure of mixture of solids and liquids existing under high pressure. W. R. TATE, H. P. STEPHENSON, H. P. DEAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,106, 9.5.29).—The pressure of an abrasive liquid is released in a tandem hydraulic ram, the other piston of which operates on clean liquid, which may be caused to do useful work. B. M. VENABLES.

Filter. F. B. LEOPOLD, Assr. to McKENNA BRASS & MANUF. Co., Inc., and L. DE MARCUS (U.S.P. 1,750,879, 18.3.30. Appl., 10.11.26).—A non-porous or sand filter is combined with a porous or paper filter; the latter forms a check on the condition of the former, and for convenience of inspection is placed in the cover of the apparatus. Connexions are provided so that, when cleaning, the sand filter only receives a back-flush. B. M. VENABLES.

Filtering device. C. A. and P. X. MCGEEHAN (U.S.P. 1,750,185, 11.3.30. Appl., 22.8.27).—A number

of disc filter elements are mounted with spacers on an outlet pipe which can be rotated by hand. Fixed but flexible scrapers are attached to the outer casing which forms the inlet chamber and serves to remove collected sludge and/or filter aid (if used). B. M. VENABLES.

Filter device. F. L. M. KENNEY (U.S.P. 1,749,730, 4.3.30. Appl., 22.6.28).—A simple device for insertion in a pipe-line or attachment to a tap is described; a filter disc is clamped between inlet and outlet chambers. B. M. VENABLES.

Centrifugal machine. W. H. BATH, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,750,154, 11.3.30. Appl., 5.12.28).—A cushioned gyrating bearing for a supercentrifuge is described. B. M. VENABLES.

Clarifier bowl. L. D. JONES and A. U. AYRES, Assrs. to SHARPLES SPECIALTY Co. (U.S.P. 1,749,538, 4.3.30. Appl., 4.9.25).—For a centrifuge, a form of neck is described by which the depth of liquid retained may be easily varied by changing a weir ring. B. M. VENABLES.

Viscosimeters. A. E. ROBINSON (B.P. 330,042, 2.4.29).—A viscosimeter of the falling-ball type is provided with electric contacts at the bottom to indicate the time of arrival of the ball when used in an opaque fluid. B. M. VENABLES.

Method and apparatus for crystallisation. E. W. KOPKE, Assr. to F. L. ALLEN (U.S.P. 1,749,588, 4.3.30. Appl., 19.1.27).—The apparatus comprises a horizontally elongated vessel with stirrers spaced on a horizontal shaft. Alternating with the stirrers are vertical, stationary, flat cooling coils extending across substantially the whole transverse area of the vessel; these are supplied in parallel with regulable quantities of cooling medium so that a rapid initial cooling and slower final cooling may be effected. The liquid, *e.g.*, masse-cuite, is supplied continuously. B. M. VENABLES.

Fractionating apparatus. E. H. HARRIS, Assr. to B. M. HERR (U.S.P. 1,750,306, 11.3.30. Appl., 6.8.26).—A fractionator in which the gradation of temperature is continuous and not stepped is constructed of a bundle of inclined tubes for the vapour being treated, around which are passed a number of circulations of heating or cooling fluid, the temperatures of which are thermostatically controlled; a heater, cooler, and pump are included in each circuit. To prevent sudden changes of temperature at the walls dividing one circulation from the next, baffles transverse to the tubes are provided which are spaced in such a manner to give a varying velocity (and hence heat transmission) of the outer fluid, the spacing being precalculated according to the laws of fractionation and of heat transmission. B. M. VENABLES.

Evaporator. W. SIECK, JUN., Assr. to W. GARRIGUE & Co. (U.S.P. 1,750,434, 11.3.30. Appl., 16.1.28).—A bundle of inclined tubes is supplied with heating medium inside so that vapour produced outside can immediately leave the heating surface and thus improve the transmission of heat. Definite circulation is provided for by partitioning off a lower, inclined, longitudinal compartment for the return flow of liquid. Means are also provided for the removal of precipitate. B. M. VENABLES.

Chemical apparatus. [Precipitation tanks.] W. H. ALLEN, Assr. to PARKER RUST PROOF Co. (U.S.P. 1,750,617, 18.3.30. Appl., 14.3.27).—Precipitates are removed from the bottom of a tank by a worm or other type of conveyor working in a sediment chamber below the tank. This chamber communicates with a stand-pipe normally extending to above the level of the liquid in the tank, but it is made telescopic or flexible so that to discharge sediment it may be lowered below liquid level. A perforated false bottom may be placed in the tank. B. M. VENABLES.

Dust separators and collectors. DORMAN, LONG & Co., LTD., and M. R. KIRBY (B.P. 330,163, 5.7.29).—An inner whirling chamber is situated within an outer settling chamber. The former has a cylindrical outer surface with tangential slots to permit exit of concentrated dust-laden air to the outer settling space, but it (the inner chamber) is formed from one or a number of intercoiled, helical passages; alternatively, it may be cylindrical and the whirling produced by a paddle-wheel. B. M. VENABLES.

Apparatus for filtering air. J. C. HINES (U.S.P. 1,751,999, 25.3.30. Appl., 26.5.27).—A method of distributing oil or viscous liquid over labyrinth plates is described, the edges of the plates being bent back to prevent oil being blown off. B. M. VENABLES.

Thermal-conductivity gas-analysis apparatus. J. C. PETERS, JUN. (U.S.P. 1,751,715, 25.3.30. Appl., 27.2.26. Renewed 4.2.30).—The sample chamber of an apparatus that compares the heat loss from two electric-resistance wires (in the sample and a standard gas) by means of a Wheatstone bridge (cf. U.S.P. 1,504,707) is lined with non-corrodible material, and the heat conductivity between the lining and wall is maintained at a high definite value by the interposition of solder, varnish, or the like. B. M. VENABLES.

Light filter. I. SHOJI, Assr. to SANKYO KABUSHIKI KAISHA (U.S.P. 1,751,220, 18.3.30. Appl., 3.8.26. Jap., 24.12.25).—The use of hexamethylenetetramine-triphenol as a film of less than 0.25 mm. is claimed as a filter for ultra-violet light; colouring matter to filter visible light may be added, and the film may be placed between two glass plates or applied to bottles or electric bulbs or even used without a rigid base. A method of manufacture is described in which phenol and formaldehyde are condensed in the presence of ammonia. B. M. VENABLES.

Light filter. C. S. McNAIR (Austr.P. 9581, 27.9.27).—A solution of alum, copper sulphate, and, preferably, an iron salt, together with free sulphuric acid to prevent the formation of basic salts, is contained in a red glass vessel or, alternatively, contains red colouring matter, e.g., a cobalt salt, to neutralise its colour, and is used to cut off heat rays. J. S. G. THOMAS.

Heat-treatment process and apparatus in which a hot liquid mass is employed. C. F. HAMMOND, Assr. to W. SHACKLETON (U.S.P. 1,765,148, 17.6.30. Appl., 1.4.27. U.K., 15.4.26).—See B.P. 278,768; B., 1927, 928.

Heat exchanger. J. HEIZMANN (U.S.P. 1,767,794, 24.6.30. Appl., 31.3.28. Fr., 28.2.28).—See B.P. 297,643; B., 1928, 878.

Heat interchanger. A. E. LEEK (U.S.P. 1,767,989, 24.6.30. Appl., 22.4.27. U.K., 8.12.26).—See B.P. 279,663; B., 1928, 39.

Air- and gas-washing apparatus. E. M. BASSLER (U.S.P. 1,766,588, 24.6.30. Appl., 20.8.23).—See B.P. 296,159; B., 1928, 774.

Apparatus for collecting suspended dust. H. WARING, Assr. to ASSOCIATED LEAD MANUFACTURERS, LTD. (U.S.P. 1,761,377, 3.6.30. Appl., 9.3.27. U.K., 28.8.26).—See B.P. 282,849; B., 1928, 216.

[Centrifugal] fluid-mixing device. P. JANES (U.S.P. 1,766,643, 24.6.30. Appl., 1.3.28. Austr., 30.7.27).—See B.P. 305,877; B., 1929, 498.

Filter for absorbing ultra-violet light. T. SUZUKI and S. SAKURAI, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,766,411, 24.6.30. Appl., 8.1.27. Jap., 20.12.23).—See B.P. 242,721; B., 1926, 76.

Production of chemically stable articles. J. K. WIRTH (U.S.P. 1,767,421, 24.6.30. Appl., 19.11.25. Ger., 11.4.25).—See B.P. 292,334; B., 1928, 606.

[Suspension-type] furnace roofs. SCHEIDHAUER & GIESING A.-G. (B.P. 313,175, 7.6.29. Ger., 8.6.28).

Process and apparatus for manufacture of shaped objects from fusible materials. I. G. FARBERIND A.-G. (B.P. 308,614, 22.3.29. Ger., 23.3.28).

Pulverising apparatus (B.P. 328,437).—See II. **Dehydration** (U.S.P. 1,749,455).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Origin of coal and oil. E. TERRES and W. STECK (Gas- u. Wasserfach, 1930, 73, Sond.-Heft, 1—5).—Recent views on the origin of coal are briefly outlined. Peat moss (sphagnum) has been transformed into peat in the laboratory by subjecting it to bacterial decay for 9—10 months; during the process only the cellulose appeared to have suffered complete decomposition. By heating the product at 280—300° under pressure it was converted into brown coal containing 35.2% of a bituminous oil. By similar treatment of the sludge formed by the putrefaction of yeast or egg albumin, artificial coals possessing swelling and caking properties have been obtained. These products also contained an oil similar in composition to that extracted from the artificial brown coal. It is concluded that whereas the lignites and brown coals are transformation products of lignin, the bituminous coals originated in protein-containing substances. A. B. MANNING.

Determination of oxygen in coal. F. SCHUSTER (Gas- u. Wasserfach, 1930, 73, 549—551).—The significance of the results obtained in the ultimate analysis of coal is discussed with reference to the interpretation of the oxygen content as determined "by difference." It is shown that combustion produces chemical changes in the mineral constituents of the coal, and that consequently the percentage of ash determined is not necessarily identical with the content of mineral constituents. This affects the percentage of oxygen as determined by difference. Experiments were made to determine the oxygen in coal by a method similar to that used by

Ter Meulen and Heslinga ("Neue Methoden der organisch-chemischen Analyse," Leipzig, 1927). The apparatus and manipulative details are described. The method consists essentially in heating the coal in a combustion tube through which a stream of pure dry hydrogen is passing. The gases produced pass over a heated column of asbestos fibre, where hydrogen and any oxygen unite, and the products, containing also carbon monoxide and dioxide, pass over a column of a mixture of asbestos and nickel freshly reduced from nickel oxide. This column is maintained at 420° and catalytically induces the reactions $\text{CO} + 3\text{H}_2 \rightarrow \text{CH}_4 + \text{H}_2\text{O}$, $\text{CO}_2 + 4\text{H}_2 \rightarrow \text{CH}_4 + 2\text{H}_2\text{O}$. The mixture of gases passes over a short plug of nickel-asbestos mixture maintained at 150°. Sulphur and chlorine are here abstracted from the gas. Finally the gases pass through three weighed U-tubes containing, respectively, (a) a solution of silver sulphate acidified with sulphuric acid, (b) anhydrous calcium chloride, (c) soda-lime and anhydrous calcium chloride. Ammonia and chlorine which has escaped reaction are retained by (a) and are determined by distillation and titration with potassium thiocyanate, respectively; the water produced is calculated from the increase in weight of the tubes. The last U-tube serves to absorb any unchanged carbon dioxide. The percentage of oxygen is calculated from the water produced. It is pointed out that the presence of oxides and sulphides in the mineral constituents leads by this method to oxygen percentages higher than the true value, and to low oxygen content as determined by difference. Experiments were made to determine the differences in the oxygen content as found by the two methods. With three dried coals of ash percentage 7.6, 9.7, 10.3, the differences were +0.7, +1.8, +2.5% O respectively, the hydrogenation method giving the highest results. Coal ash was prepared from the three coals by the usual method and was reduced in the apparatus described. Appreciable quantities of reducible oxygen were shown to be present. It was concluded that the hydrogenation method of Ter Meulen and Heslinga, although quite applicable to pure organic substances, gives results as open to criticism as are those of the difference method when applied to coal, especially when the oxygen content of the coal substance is under consideration.

H. E. BLAYDEN.

Selection of coal for gas producers. E. J. C. BOWMAKER and J. D. CAUWOOD (J. Soc. Glass Tech., 1930, 14, 16—29 r).—Determinations of the percentage of ash, volatile matter, Fe_2O_3 in ash, softening temperature of ash, and aniline solubility were made on samples of 11 coals. The solubility in aniline was determined by Soxhlet extraction of 0.5 g. of powdered dried coal until the aniline in the siphon tube was almost colourless. From the data so obtained the suggested specification for a suitable coal was: ash not above 5%, volatile matter 35—38%, solubility in boiling aniline not above 6%, Fe_2O_3 (in ash) not above 15%, of the dry coal. It was stressed that this applied to Kerpely producers, and might need modification for conditions widely differing from those obtaining with this type of plant.

M. PARKIN.

Functions of coke ovens. E. G. STEWART (Gas World, 1930, 92, 651—659).—Coke-oven developments

are followed with reference to the steel and gas industries, and the function of this type of plant in England, Germany, and America is discussed. The circumstances under which coke ovens can successfully compete with horizontal and vertical retorts for gasworks purposes are described, and it is shown that they are only advantageous when a capacity of about 1000 tons, or more, of coal per day is required. The coke ovens in course of construction at Beckton are described in full, and the arrangements for handling coke and for purification of gas are discussed.

R. H. GRIFFITH.

Activated carbons. G. SIMONIN (Caoutchouc et Gutta-Percha, 1929, 26, 14,357—14,359, 14,468—14,470, 14,516—14,519, 14,551—14,553, 14,594—14,595, 14,632—14,636, 14,701—14,702, 14,742—14,744, 14,778, 14,821; 1930, 27, 14,866—14,868, 14,903—14,904, 14,940—14,941, 14,983—14,985, 15,022—15,023, 15,064—15,065).—After indicating the chief physical properties of activated carbons, it is shown that the activity is not appreciably influenced by the nature of the gas from which the vapour is removed. If care is taken to avoid too high temperatures it is possible to recover methyl and ethyl alcohols, acetone, ether, ethyl acetate, and carbon disulphide unaltered from active carbon in which they have been adsorbed from air. The most rapid method for expulsion of the adsorbed vapours from the carbon without chemical alteration is by treatment with steam; below 130° the steam may be adsorbed to some extent, but above this temperature it is possible substantially to avoid such adsorption. Investigation of the tarry matter retained in the carbon after removal of benzol from coal gas revealed the presence of surprisingly little real tar, probably because of rapid resinification of the major portion of such small quantities as may be present in the gas, by contact with the charcoal. Inactivation of carbon in the removal of benzol from coal gas is attributed to the effect of sulphur, which converts the active carbon molecules into a carbon disulphide. Re-activation by calcination before and after treatment with hydrochloric acid is tentatively explained by the decomposition of the disulphide by iron naturally present; the acid not only decomposes the ferrous sulphide formed in the first calcination, but effects a uniform and intimate dispersion of the regenerated iron salt in readiness for the second calcination. Similarly, it is possible to remove the inactivating sulphur and so to revivify carbon by impregnation with ferric chloride solution followed by heating, e.g., for 2 hrs. at 900°.

D. F. TWISS.

Flash point of activated charcoals. M. SWIDEREK (Rocz. Chem., 1930, 10, 365—384).—Two constant flash points are obtained for activated charcoals, according to the rate of heating; the value obtained when the temperature is raised slowly is higher than when the rate of heating exceeds a certain characteristic value for each type of charcoal. The lower flash point is not characteristic of the given type of charcoal, as its value depends on various factors, such as the temperatures of activation and of drying, content of activating substance, moisture content, etc. The value of the upper point is a function of the temperature at which the charcoal was activated, but is independent of the content of

activators, of various secondary processes which might have taken place during the preparation of the charcoal, and of the initial velocity of heating, provided only that the rate of heating at temperatures close to the flash point is sufficiently low (about $3^{\circ}/\text{min.}$). The adsorptive power of the charcoal stands in no relation to its flash point; this observation militates against the probability of the theory that activated charcoal is a chemisorbent. Charcoals reheated at temperatures above those used for their activation have a lower flash point than have those activated originally at the higher temperature, and this difference is directly proportional to the difference between the two temperatures of activation. The flash point has a maximum value for charcoals the granules of which have a mean diameter of about 1.3 mm.

R. TRUSZKOWSKI.

Benzol recovery, with special reference to gas-works' practice. W. H. HOFFERT (Gas World, 1930, 92, 667—671).—The cost of benzol recovery from coal gas, and its refining, is partly dependent on the amount of unsaturated hydrocarbons in the crude liquid. The value of the benzol as a component of town's gas is correspondingly affected. Special methods adopted for the treatment of benzols which deposit gums on storage are mentioned.

R. H. GRIFFITH.

Importance of benzol recovery for the German gas industry. H. KEMMER and G. BAUER (Gas- u. Wasserfach, 1930, 73, 509—519. Cf. Firth, B., 1930, 129).—Nomograms are constructed showing the gain or loss, expressed as a variation in the price of gas, resulting from the recovery of benzol from coal gas, as a function of the prices of coal, coke, and crude benzol. Other methods of calculating the effect of benzol recovery are discussed, and it is concluded that, at the prices now ruling in Germany, such recovery would be economically advantageous. In addition to the direct financial gain, the removal of benzol from the gas would result in improved operation of the purifying plant, the distributing system, etc.

A. B. MANNING.

Application of graphical methods in the gas industry. SCHUMACHER (Gas- u. Wasserfach, 1930, 73, 494—499, 524—529).—Curves and diagrams are constructed to illustrate various relationships and operations of importance in the gas industry, e.g., calculation of the calorific value of coke from its ash and moisture content, flue-gas losses as a function of the temperature and composition of the gas, gas-analysis procedure, regulation of automatic gas-heated water-circulating apparatus, operation of refrigerators, distribution and utilisation of the water supply in a gasworks, etc.

A. B. MANNING.

[Coal-]gas dehydration. E. W. SMYTH (Gas World, 1930, 92, 639—648).—Practical results from works where gas dehydration is practised are reviewed, and alternative methods for partial drying of gas are discussed. It is found necessary to apply oil films to the water of the ordinary wet gasholder, and to substitute some other suitable liquid for the water in wet gas meters, when dried gas is prepared. American procedure depending on compression is compared with other types of plant, and alterations in operation due to seasonal changes of temperature are mentioned. Obser-

vations by nine different companies, including actual or potential financial advantages, are recorded.

R. H. GRIFFITH.

Naphthalene [in coal gas]. F. W. J. BELTON (Gas J., 1930, 190, 884—886).—Measures adopted to lessen the amount of naphthalene in town's gas are described, and the gradual diminution of troubles due to blockage of service pipes is traced, following the introduction of new plant.

R. H. GRIFFITH.

Experiences with large "double-gas" [mixed coal- and water-gas] generators. W. SCHROTH (Gas- u. Wasserfach, 1930, 73, Sond.-Heft, 18—26).—The plant that has been in operation at Dresden during the last five years is described. It consists of three generators, each 3 m. in diam. in the gasifying zone and 2 m. in diam. in the carbonising zone. The "blow" gases pass through a dust separator to a regenerator, wherein any combustible constituents are burned with secondary air, and thence to a waste-heat boiler. The sensible heat of the water-gas produced during the "run" is utilised in carbonising the coal in the upper part of the generator. The coal used should have an ash of high m.p. It must, moreover, while passing through the plastic stage, offer little resistance to the passage of gas. The suitability of a coal for this process is judged by (a) a crucible coking test; (b) a determination of caking power by Kattwinkel's method (B., 1926, 257, 905), but using only 4 pts. of sand to one of coal; and (c) measurement of the maximum pressure required to force a current of nitrogen through a column of the coal which is heated over the plastic range (cf. Layng and Hathorne, B., 1925, 195). The blow gases are relatively high in hydrogen and carbon monoxide because of the high reactivity and volatile matter content of the coke entering the gasifying zone. For efficiency of operation the steam used should be highly superheated, otherwise an undesirable amount of combustible matter is discharged with the ash. The losses during stoppages are greater than in a water-gas generator, and it is, therefore, desirable to operate the plant continuously, controlling the gas output by regulating the rate of supply of steam during the "run." Loss of gas during the clinkering period is avoided by the provision of a special exhauster, which is operated during that period, and adds the gas then evolved to the total gas. Some experimental results illustrating different methods of running the plant are tabulated; they show the possibility of raising the rate of production from 30,000, the guaranteed rate, to 50,000 m.³ of gas per 24 hrs. The thermal efficiency of the plant averages 65%, the calorific value of the gas being about 4000 kg.-cal./m.³

A. B. MANNING.

Law of flame speeds. II. Law applied to mixtures of combustibles having different theoretical flame propagation temperatures. Y. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 120—125 B; cf. A., 1930, 1000).—Generally, the law previously proposed does not hold, but it can be applied to those gaseous mixtures containing a sufficient amount of one combustible, so that the second combustible has the same theoretical flame propagation temperature as the first. This application of the law is supported by the work of Bone (A.,

1927, 26, 630) and Payman (B., 1923, 436 A), and is further demonstrated by experiments to determine the concentrations of hydrocarbons and lead tetraethyl in gaseous mixtures containing air having uniform flame speeds of 65 cm./sec. and of 35 cm./sec.

S. K. TWEEDY.

Determination of viscosity on small samples of tar. F. M. POTTER and J. S. SACH (Roads, 1930, 8, 231—232).—The apparatus described by Mallison (B., 1921, 197 A) is shown to provide a rapid means of checking the viscosities of tars, and the precautions necessary to obtain consistent results are discussed.

D. G. MURDOCH.

Origin and formation of acetic acid of pyroligneous liquor. II. C. PADOVANI and A. MARIOTTI (Annali Chim. Appl., 1930, 20, 205—210).—Experiments similar to those on Libyan esparto (B., 1929, 309) have been made on beech wood of the percentage composition: moisture 12.95, ash 1.49, fats and waxes 2.132, cellulose 52.94, lignin 30.49, methoxyl 1.45. Ordinary destructive distillation gave: charcoal 29.5, pyroligneous liquor 41.9, acetic acid 4.5%. The woody material offers greater resistance than esparto to hydrolysis by cold sodium hydroxide solution under 5 mm. pressure. The percentage of acetic acid, calculated on the weight of wood taken, reaches the value about 6.6 when the concentration of the alkali is raised to about 9% and then remains sensibly constant up to 12% alkali. The maximum proportions of acetic acid removed by the alkali solution for beech wood and esparto are very nearly in the ratio of the lignin contents of the two materials (cf. Pringsheim and Magnus, A., 1919, i, 473). Distillation of the dried residue from the alkali treatment yields about 1.6% of acetic acid (on the original wood), the total yield of acetic acid being thus 8.2%. Digestion of the wood with 2.5% sodium hydroxide solution under 9 atm. pressure (180°) yields 8% of acetic acid, this being increased to 9.5% by distillation of the dried residue. The so-called lignin appears to be composed of two groups of quite distinct components, some (including preformed acetyl groups) being removable by mild hydrolysis in the cold, whereas others are detachable from the cellulose only by more violent and disruptive processes. The amount of methoxyl groups in the alkali solution does not correspond with the difference between the amounts in the original wood and in the residue, but diminishes rapidly as the action proceeds, so that methoxyl groups are destroyed during the attack of the wood and the subsequent concentration of the liquors.

T. H. POPE.

Heat calculation for flash distillation [of petroleum hydrocarbons]. G. H. FANCHER (Chem. Met. Eng., 1930, 37, 307—308).—In calculating the heat added during vaporisation of hydrocarbons by means of Turner and Harrell's equation (B., 1930, 357), more reliable results can be obtained by taking into consideration the average sp. gr. and the temperature rather than the mol. wt. of the vapour formed. The value of the latent heat used in this calculation should be obtained from the relation $l = 1/d \cdot (110.9 - 0.09 t)$, where l is the latent of vaporisation in B.Th.U. per lb., d the sp. gr. of the liquid vaporised at 60°/60° F., and t

is the temperature in ° F.; this value is more accurate than that obtained from Hildebrand's or Trouton's rules.

D. K. MOORE.

Stabilisation of the knock value, gum content, and colour of gasoline by chemical inhibitors. G. EGLOFF, W. F. FARAGHER, and J. C. MORRELL (Petroleum, 1930, 26, 243—249).—The oxidation products, e.g., peroxides, acids, aldehydes, and ketones, which are formed in benzene on being kept lower the knock value. In a series of tests on Pennsylvanian cracked benzines, accelerated oxidation was found to decrease the benzol equivalent from 40% to 29%. The effect of chemical inhibitors on Mid-continent and Pennsylvanian cracked benzines shows a decrease of the knock value during accelerated oxidation. Such inhibitors are aniline, dimethylaniline, quinol, urea, anthracene, etc. The gum formation on keeping depends on the quantity of inhibitor added, the presence of water, the type of container, the oxidation medium, and on the effects of temperature and light. Oxidation by air or oxygen causes an increase in gum formation. Results are tabulated showing the effect of accelerated oxidation tests on gasolines with or without added inhibitors. The latter prevent gum formation. The colour is influenced by elementary sulphur and no colour stabiliser is known for benzene which has been kept. The anti-knock properties of cracked gasolines can be restored by treating them with fuller's earth, caustic soda, borax, etc.

W. S. E. CLARKE.

Motor oils from redistillation of mazout distillates from naphthenic-base crude oils. I. KLIGERMAN (Azerbaid. Neft. Choz., 1930, No. 2, 77—83).—High-grade lubricating oils are obtainable.

CHEMICAL ABSTRACTS.

Sludge of transformer oils. III. Catalytic effects of several metals on the formation of sludge and acids. T. YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 113—114 B; cf. B., 1930, 542).—The relative catalytic influence of a number of metals on the formation of sludge and of acid in transformer oils has been measured by heating the oil at 140° for 100 hrs. in contact with air and with the metal. With refined oil many metals, and notably lead, zinc, iron and magnesium, tend to reduce the acidity of the oil; this, however, is due in part to the formation of metallic salts. With less highly refined oil copper, brass, silver, and lead increase considerably the quantity of sludge formed, whereas magnesium, tin, aluminium, and zinc have the reverse influence. Metals which tend to accelerate sludge formation increase, in general, the formation of acid.

H. F. GILLBE.

[Refining of petroleum oil crudes by] the Edel-eanu process. J. ROSENBERG (Petroleum, 1930, 26, 137—141).

Heat transmission. FISHENDEN. **Autoclaves.** HARRISON. **Fire extinguishers.** BIESALSKI.—See I.

PATENTS.

Apparatus for pulverising coal and like substances. ASHINGTON COAL CO., LTD., and W. J. DRUMMOND (B.P. 328,437, 24.4.29).—A pulverising mill in which the finely-divided particles are removed from the

grinding zone by means of a current of air is provided with a number of openings in the track where the heavier particles tend to accumulate; these lead to receptacles in which the heavy particles can collect and from which they are periodically and automatically discharged. A current of air is admitted into each receptacle in such a manner as to return any lighter or fully-ground particles back to the mill.

A. B. MANNING.

Rotary retort for low-temperature carbonisation. W. HAUSWALD and E. MILDNER (G.P. 461,703, 21.4.26).—The material to be carbonised is fed into tubes which consist of three sections of different diameter, fitting one into the other. The tubes are subdivided by partitions in the rotating drum in such a manner as to form a carbonising zone which is externally and internally heated. A method of constructing the retort is described.

A. B. MANNING.

Internally heated low-temperature carbonisation plant. METALLBANK U. METALLURG. GES. (Aust. P. 109,716, 4.10.26. Ger., 24.10.25).—The gaseous heating medium leaving the carbonising zone is freed from tar and passed through a multiple-stage countercurrent cooler in such a manner that while the whole of the gas is passed through the first stages of the cooler, only the excess gas which is being withdrawn from the circulating system is passed through the other stages.

A. B. MANNING.

Continuous gasification of granular or pulverulent material, such as pit coal, brown coal, coke, etc. KOHLENVEREDLUNG A.-G. (B.P. 311,775, 15.5.29. Ger., 16.5.28).—The finely-granular material together with steam, air, and/or oxygen is passed through parallel juxtaposed passages formed by dividing up a rotary drum by means of chequer bricks, the material being simultaneously heated to gasification temperatures. The heat required for gasification may be supplied from an external source, *e.g.*, by passing a heating gas along passages formed between the reaction passages by the chequer bricks, or from the combustion of part of the raw material.

A. B. MANNING.

Utilisation of the quenching vapours periodically resulting from the quenching of glowing coke in a pressure vessel. BAMAG-MEQUIN A.-G., and O. HELLER (B.P. 328,852, 25.6.29).—The mixture of steam and water-gas produced when the glowing coke is quenched in a pressure vessel is passed through the water contained in a steam accumulator. By means of an automatic pressure valve the pressure in the steam space of the accumulator is maintained at a value higher than that corresponding with the temperature of the water, and in consequence the entering steam is almost completely condensed, while the water-gas passes through the valve to the points of consumption. During the succeeding intermediate period the steam which has accumulated in the water of the accumulator is drawn off through a separate pipe. Several alternate arrangements of the apparatus for carrying out the process are described.

A. B. MANNING.

Purification of graphites. E. RABETRANO (B.P. 328,697; 25.6.29. Fr., 10.5.29).—The impure graphite is treated with hot, dilute sodium hydroxide under

pressure, and then, after washing, with boiling dilute acid, preferably sulphuric acid.

A. B. MANNING.

Gas generators or producers. HUMPHREYS & GLASGOW, LTD., and A. G. GLASGOW (B.P. 329,146, 4.5.29).—A mechanically clinkered gas generator is provided with one or more pockets for receiving the clinker and ash. The inlet to and outlet from each pocket are interlocked in such a manner that either can be opened only when the other is completely closed. The outlet closure may also be interlocked with the operating gear of the plant so that it cannot be opened while gas is being made in a downward direction through the generator.

A. B. MANNING.

Water-gas generators. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,642—3, 25.1.29).—(A) A deep coke bed is provided in the generator and the air is supplied thereto through several tuyères at different depths, the blow gases being withdrawn at the base. Steam is then supplied to traverse the whole depth of the bed in an upward direction. The blow gases are passed through a combustion chamber, wherein any carbon monoxide is burned with secondary air, and then through a regenerator which is used for preheating the steam. A second regenerator, through which the water-gas is passed during the "make," serves to pre-heat the air during the "blow." (B) The combustion chamber consists of an annular jacket surrounding the lower portion of the generator. It is separated from the coke bed by a thin wall and communicates with the generator through ports near its bottom end.

A. B. MANNING.

Control of gas-making plants. W. T. GLOVER and H. J. TOOGOOD (B.P. 329,167, 16.5.29).—The gas main is provided with a compartment in which the liquor can collect and which communicates with a vessel having an adjustable overflow. The cross-sectional area through which the gas can flow in the compartment is determined by the height of the liquor therein, *i.e.*, by the height of the overflow; this is controlled by means of a movable bell which is exposed on one side to the atmospheric pressure and on the other to the gas pressure in the main.

A. B. MANNING.

Production of mixed oil gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of (A, B) H. G. TERZIAN, and (C) E. L. HALL (B.P. 329,245—6 and 329,598, 11.9.29. U.S., [A] 8.11.28, [B] 3.1.29, [C] 29.12.28).—(A) The fuel bed in the generator is intermittently air-blasted and the blast gases are used for heating the carburettor and superheater. Between air-blasting operations the heat stored therein is utilised to vaporise, or vaporise and lightly crack, liquid hydrocarbons. The resultant oil vapours and oil gas are then passed through the fuel bed in the generator to complete their cracking, the carbon thereby formed being deposited in the fuel bed. At suitable intervals the fuel bed is blasted with steam, the blue water-gas produced being carburetted if desired. (B) Means are provided for extracting tar from the oil gas, produced as above described, both before and after its passage through the fuel bed. (C) The generator fuel bed is blasted with air, the resultant gases being burned in the carburettor and superheater. The step is followed either by a water-gas

production step or by a re-formed oil-gas production step. The latter consists in supplying oil and previously made gas together to the superheater, the gas being used to carry the resulting oil vapours and oil gas through the carburettor and then through the fuel bed. The water-gas produced may be carburetted in the usual manner, if desired. A. B. MANNING.

Continuous production of water-gas, or of producer gas rich in hydrogen or carbon monoxide, from powdered fuel. K. FISCHER (G.P. 461,481, 27.11.24).—The fuel is gasified while suspended in the gasifying agent, the necessary heat being supplied by radiation from a flame maintained in the producer. The sensible heat of the waste gases from the radiating flame may be utilised for heating a boiler or industrial furnace. A. B. MANNING.

Purification of gases. W. D'LENY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,135, 26.4.29).—Hydrogen sulphide and cyanogen are removed from gas by passing it in contact with an alkaline suspension of ferric hydroxide, the suspension being in the form of a continuous, freely falling, liquid film. The suspension may be recirculated, a part being withdrawn continuously for regeneration, which is effected by exposing it, again in the form of a freely falling film, to the action of air. A. B. MANNING.

Removal of ammonia and hydrogen sulphide from gases. C. J. HANSEN, ASSEC. of H. KOPPERS A.-G. (B.P. 316,278, 30.11.28. Ger., 28.7.28. Addn. to B.P. 309,116; B., 1930, 546).—The process described in the prior patent is modified by using the metal thionate liquor to scrub gas which contains ammonia and the metal hydroxide liquor to scrub ammonia-free gas. The two spent liquors are then mixed and re-divided into two parts for regeneration with sulphurous acid and oxidising gas, respectively. Accumulation of sulphur in the hydroxide liquor is thereby avoided, whilst the advantages of separate scrubbing operations are obtained. A. B. MANNING.

Separation of acetylene from mixtures of gases. SOC. CHEM. IND. IN BASLE, ASSECS. of H. ANDRIESENS (B.P. 316,888, 2.8.29. Ger., 4.8.28).—Acetylene is separated from gas mixtures containing methane, hydrogen, and, perhaps, nitrogen by adsorption on activated carbon or similar adsorbent. Ethylene, if present, may be adsorbed at the same time. The adsorbent, when saturated with acetylene, is first evacuated in the cold whereby most of the other gases are removed without loss of acetylene. The latter is then recovered in a high degree of concentration by heating and evacuating the adsorbent. It is advantageous to carry out the adsorption at a low temperature, e.g., -40° . A. B. MANNING.

Washing of gas liquors. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 328,388, 15.3.29).—Phenols and other organic substances are extracted from gas liquors by bringing the latter into intimate contact with tri-aryl or -alkyl phosphates, e.g., tri-*o*-tolyl phosphate. The phenols are recovered by extraction of the phosphoric acid ester with dilute sodium hydroxide, and the bases and neutral oils are removed from the ester by subsequent steam-distillation. A. B. MANNING.

Distillation of tar at a gas-retort plant. BARRETT Co., ASSECS. of S. P. MILLER (B.P. 309,946, 16.2.29. U.S., 18.4.28. Cf. B.P. 303,166; B., 1930, 545).—The gases from a number of coal-distillation retorts are passed directly into a single vertical stand-pipe, which is heat-insulated. Tar or pitch is brought into intimate contact with the hot gases in the stand-pipe and is thereby distilled. The residual pitch is withdrawn from the bottom of the stand-pipe, while the gases and vapours, after a cleaning treatment to remove suspended pitch if necessary, are passed to the condensing system. A. B. MANNING.

Production of higher tar yields in carbonisation processes. P. JUNG (G.P. 461,425, 4.6.24).—The fuels are mixed with chlorides of the earth or alkaline-earth metals, ammonium chloride, ferric chloride, etc., either before or during carbonisation. Metals also may be added to the mixture, which, if desired, may be briquetted before carbonisation. A. B. MANNING.

Preparation of [rubberised]-fabric coating composition. E. I. DU PONT DE NEMOURS & Co. (B.P. 303,368, 29.12.28. U.S., 31.12.27).—Asphalt, petroleum residue, stearine pitch, or other bituminous material is cracked, and the distillate is fractionated or "bodied," i.e., heated in an open container, to obtain a product of suitable viscosity. A coating composition for rubberised fabrics is prepared by mixing this product with a pigment, a drying oil, and a thinner. Part or all of the drying oil in the mixture may be replaced by asphalt, petroleum flux, or other bituminous material. A. B. MANNING.

Feeding pastes of coal and oil or the like into hydrogenation converters. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,044, 21.2.29).—The paste or slurry of coal and oil is continuously circulated through the storage vessel in order to prevent settling, and is withdrawn as required from the circulating system for injection into the hydrogenation converter. A single pump may serve the dual purpose of feeding the injector and circulating the slurry. A. B. MANNING.

Destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,992, 5.1.29. Cf. B.P. 326,184; B., 1930, 498).—In order to maintain the finely-dispersed catalysts continuously in uniform suspension in the liquids or pastes to be hydrogenated, the difference in sp. gr. between the material to be treated and the catalysts is reduced either by making additions of suitable sp. gr. to the former or by incorporating a suitable carrier, e.g., active carbon, powdered pumice, with the latter. A. B. MANNING.

Recovery of refined products from carbonaceous materials such as coal, tars, mineral oils, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,997, 12.11.28).—The reaction products from the hydrogenation under pressure of coal, oil, tar, etc. are fractionated by passing through them, in a suitable column, gases, vapours, or liquids having lower temperatures than the reaction products. A. B. MANNING.

Depolymerisation of hydrocarbons. J. MERCIER (B.P. 302,899, 7.12.28. Fr., 22.12.27).—The hydro-

carbon is vaporised and the vapours are passed up through a zig-zag retort containing a depolymerising catalyst. The successive segments of the retort extend across a vertical shaft, which is traversed by a heating medium; a series of baffles within the shaft direct the flow of heating medium along the segments of the retort. The upper section of the retort is heat-insulated. The vapours issuing from the retort pass into a cooling and expanding chamber, which may also contain a catalyst. The heavy oils condensing therein may be returned for re-treatment; the uncondensed vapours may proceed to a second expansion and cooling unit, or to the bottom of a fractionating tower. A. B. MANNING.

Catalytic synthesis of liquid and gaseous hydrocarbons. A. FOLLIET (F.P. 637,891, 19.7.27).—Mixtures of hydrocarbons, oxides of carbon, and, if desired, hydrogen, are heated at 500–1000° under 5–100 atm. pressure in a metal apparatus encased in a jacket of refractory material, and, after being cooled if necessary, are allowed to expand suddenly into the catalyst chamber. A. B. MANNING.

Ketone fuels. SOC. DES BREVETS ÉTRANGERS LEFRANC ET CIE. (F.P. 637,993, 30.11.26).—The fuels consist of complex ketones produced by the dry distillation of calcium salts, *e.g.*, calcium butyrate, and liquid fuels obtained synthetically (methyl alcohol), by fermentation, or by the distillation of carbonaceous material (benzol). A. B. MANNING.

Purification of [used mineral] oils. L. H. CLARK, ASSR. to SHARPLES SPECIALTY CO. (U.S.P. 1,747,161, 18.2.30. Appl., 11.7.25).—Mineral oils containing decomposition products, *e.g.*, used transformer or switch oils, are emulsified with about 1% of an activated carbon and an aqueous alkaline reagent, *e.g.*, a solution of caustic soda or sodium silicate; the renovated oil is separated by centrifuging. E. LEWKOWITSCH.

Apparatus for gasifying and distilling solid fuel. E. ROSER (U.S.P. 1,765,702, 24.6.30. Appl., 31.12.25. Ger., 6.7.25).—See B.P. 257,479; B., 1926, 972.

Coke oven. E. LECOCQ, ASSR. to SOC. GÉN. DE FOURS À COKE SYSTÈME LECOCQ SOC. ANON. (U.S.P. 1,761,835, 3.6.30. Appl., 23.3.25. Belg., 29.3.24).—See B.P. 231,483; B., 1926, 4.

Carbonisation of coal. W. E. TRENT, ASSR. to TRENT PROCESS CORP. (U.S.P. 1,767,778, 24.6.30. Appl., 27.2.25).—See B.P. 261,954; B., 1927, 99.

Distillation of carbonaceous substances. E. R. SUTCLIFFE (U.S.P. 1,767,231, 24.6.30. Appl., 16.7.24. U.K., 25.7.23).—See B.P. 227,879; B., 1925, 235.

Bleaching of montan wax. T. HELLTHALER, ASSR. to RIEBECK'SCHE MONTANWERKE A.-G. (U.S.P. 1,767,886, 24.6.30. Appl., 18.7.27. Ger., 26.7.26).—See B.P. 303,036; B., 1929, 198.

Emulsions from partially oxidised petroleum wax and their manufacture. A. W. BURWELL, ASSR. to ALOX CHEM. CORP. (U.S.P. 1,768,523, 24.6.30. Appl., 23.3.27).—See B.P. 287,514; B., 1929, 348.

Screens employed for the screening of coal. W. H. BERRISFORD (B.P. 330,554, 9.3.29).

Crushing machine (U.S.P. 1,750,573). **Separation of materials** (U.S.P. 1,750,090).—See I. **Tar-stained wool** (B.P. 309,871).—See VI. **Oxidation of ammonia** (B.P. 309,583).—See VII. **Making roads etc.** (B.P. 330,072 and 330,440). **Covering roads etc.** (B.P. 310,922).—See IX. **Insulating oil** (B.P. 301,876).—See XI. **Emulsions containing rubber** (B.P. 329,965).—See XIV. **Sugar for fermentation** (B.P. 330,275).—See XVII.

III.—ORGANIC INTERMEDIATES.

Absorption of organic solvents by washing with liquids. E. BERL and L. RANIS (Z. angew. Chem., 1930, 43, 600–603).—The relations between vapour pressure and concentration of various organic liquids absorbed in cresol, sulphuric acid, and paraffin oil were determined by means of the Haber-Löwe gas interferometer. The heats of mixing of the different pairs of liquids were also determined, and the theoretical vapour pressures according to Planck's formula, which assumes a process of dissolution without chemical action, were calculated. A large number of liquid pairs are thus classified into three groups: (1) those in which the partial pressures correspond to the formula; (2) those in which they are greater than those indicated by theory, owing to the splitting of associated molecules; and (3) those in which they are less, owing to the formation of a compound. The form of the partial pressure-concentration curve, whether linear, convex, or concave, and the heat evolved on mixing in some cases afford supporting evidence. In some cases, *e.g.*, cresol-tetrachloroethane, contradictory results were obtained by the different methods, and here it is suggested that both disassociation and combination may occur. Absorbents which lower the vapour pressure of the more volatile liquid are naturally the most efficient.

C. IRWIN.

Detection of ethyl *o*-phthalate. J. SUCHODOLSKI (Przemysl Chem., 1930, 14, 265–267).—The presence of ethyl *o*-phthalate in alcoholic solutions can be detected by the following method: the solution is evaporated to dryness with 1 c.c. of 1% sodium hydroxide, 5 c.c. of concentrated sulphuric acid are added to the residue, and, after heating for a further 5–10 min. at 100°, 25–30 mg. of resorcinol are added and the whole is heated for 5 min. at 160–170°. The liquid is then dissolved in 100 c.c. of water, and 50 c.c. of sodium hydroxide solution (*d* 1.4) are added. Under the above conditions permanent fluorescence appears only if ethyl *o*-phthalate was originally present; essential oils, and other substances ordinarily present in perfumes, give either a transient fluorescence or none at all.

R. TRUSZKOWSKI.

Utilisation of soya beans. III. Y. TAKAYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 91–92 B).—Results are summarised of the experiments previously described (cf. B., 1928, 529) on the separation of glutamic acid (as the dibasic calcium salt) from the products of hydrolysis (with 25–50% sulphuric acid) of crude soya-bean protein. Hydrolysis with more concentrated acid gave greater yields. The solubility of dibasic calcium glutamate in water was found to be 1.322, 1.928, 1.979, 3.943, and 5.698 g. of anhydrous salt per 100 g.

of solution at 0°, 19°, 21°, 61°, and 100° respectively. Incipient decomposition, with liberation of ammonia (as sulphate), was observed on prolonged heating of pure glutamic acid with 10*N*-sulphuric acid at 100° and 135–145°.

E. LEWKOWITSCH.

Fractionating column. McCULLOUGH and GITTINGS. —See I. **Acetic acid from pyroligneous liquor.** PADOVANI and MARIOTTI. —See II. **Glycerin by fermentation.** TOMODA. —See XVIII.

PATENTS.

Manufacture of acetaldehyde. C. N. HAND, T. W. BARTRAM, and A. H. MAUDE, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,738,649, 10.12.29. Appl., 12.4.26). —Acetylene at 25 lb./in.² is passed into 25–30% sulphuric acid at 68°, sufficient mercurous sulphate being added to maintain the temperature at 68–80°. Entrained water and acid are removed from the exit gas, and the acetaldehyde is separated from the excess acetylene.

C. HOLLINS.

Production of (A) formic acid (B) formaldehyde [from carbon monoxide and water]. W. C. ARSEM, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,740,140—1, 17.12.29. Appl., [A] 12.9.25, [B] 3.2.26). —(A) The carbon dioxide (55%) and hydrogen (45%) evolved from butyl-acetonic fermentation is led under pressure over a mixed catalyst (reduced copper with alumina, thoria, etc.) and the resulting mixture of carbon monoxide and water vapour is converted into formic acid by passage over a cuprous chloride catalyst at 50–150° and 20 atm. or more. (B) Carbon monoxide and water vapour are passed over zinc oxide at 150–350° and 10–100 atm. Other weak basic oxides may be used. About 10% conversion is obtained.

C. HOLLINS.

Manufacture of alkyl halides. R. H. MCKEE and S. P. BURKE (U.S.P. 1,738,193, 3.12.29. Appl., 8.8.27). —An alcohol vapour is passed with hydrogen halide over chloride of magnesium, calcium, or barium at about 300°.

C. HOLLINS.

Manufacture of furyl alcohol and methylfuran. E. RICARD and H. M. GUINOT, Assrs. to SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (U.S.P. 1,739,919, 17.12.29. Appl., 29.12.27. Fr., 31.1.27). —Hydrogenation of furfuraldehyde in presence of reduced copper at 140° gives mainly furyl alcohol, with 10–20% of methylfuran which is easily removed by distillation; excess furfuraldehyde distils as azeotropic mixture with water and may be returned to the process. The furyl alcohol can be reduced to methylfuran by repeating the hydrogenation.

C. HOLLINS.

[Croton]aldehyde manufacture. A. H. MAUDE, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,738,659, 10.12.29. Appl., 29.6.27). —Aldol is heated at 160° with 3% of 85% phosphoric acid and 1% of aluminium hydroxide, i.e., a mixture of phosphoric acid and aluminium phosphate formed *in situ*. High yields of crotonaldehyde are claimed.

C. HOLLINS.

Manufacture of phenols. W. J. HALE and E. C. BRITTON, Assrs. to DOW CHEM. CO. (U.S.P. 1,737,841—2, 3.12.29. Appl., [A] 21.2.23, [B] 23.9.27). —(A) Diphenyl

ether and aqueous sodium phenoxide solution are heated above 350° in an autoclave for 15 min. The layer of free phenol is removed from the reaction product after cooling, more diphenyl ether (or chlorobenzene, which produces the ether during the heating up) is added, and the process repeated. (B) Diphenyl ether is hydrolysed with aqueous solutions of sodium salts of weak acids, e.g., sodium carbonate, borax, di- or tri-sodium phosphate, having alkaline reaction, or with a suspension of lime. The presence of chlorobenzene in the ether is advantageous.

C. HOLLINS.

Manufacture of phosphoric acid esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,963, 31.1.29. Addn. to B.P. 300,044; B., 1929, 10). —The process of the prior patent is applied to the production of mixed alkyl or mixed aryl alkyl phosphates, e.g., *n*-butyl diisooamyl phosphate, b.p. 121–122°/1.5 mm., diethyl isooamyl phosphate, b.p. 84–85°/2 mm., and phenyl dibutyl phosphate, b.p. 161°/4 mm.

C. HOLLINS.

Manufacture of carbazole derivatives. I. G. FARBENIND. A.-G. (B.P. 328,933, 4.1.29. Ger., 5.1.28). —2-Chlorocarbazole, m.p. 244°, carbazolecarboxylic acid, m.p. 271–272°, and carbazole-2-sulphinic acid are obtained by the usual methods from diazotised 2-amino-carbazole. (Cf. B.P. 303,520; B., 1930, 603.)

C. HOLLINS.

Preparation of disubstituted tetrazoles. A. BOEHRINGER (B.P. 309,949, 5.4.29. Ger., 18.4.28). —Imino-esters, R·C(OAc):NR', in which Ac represents an organic or inorganic acid residue, are treated with hydrazine, and the resulting hydrazidines are converted by nitrous acid into 1:5-disubstituted tetrazoles. The imino-esters are preferably made *in situ*, e.g., from oxime esters or *N*-substituted acid amides; the hydrazidines also are not isolated. *cyclo*Hexanoneoxime *p*-toluenesulphonate in benzene is warmed at 30–35° with alcoholic hydrazine acetate, and the hydrazidine solution so obtained yields with nitrous acid 1:5-pentamethylene-1:2:3:4-tetrazole, m.p. 59°, b.p. 192°/10 mm., similarly obtainable from the *o*-benzenesulphonate of β -leucine lactam.

C. HOLLINS.

Manufacture of adhesive and binding agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,908, 1.11.28 and 22.3.29). —Butadiene with or without an organic diluent (aniline, dimethylaniline, tetrahydrostyrene), or isoprene with a diluent, is heated at 100–200°, the reaction being stopped before solid is formed; the oily product may be hardened below 100°, if desired. Application to paint and glue is indicated. [Stat. ref.]

C. HOLLINS.

Production of gases for the manufacture of oxygenated organic compounds [methyl alcohol]. M. PIER, M. MÜLLER-CUNRADI, G. WIETZEL, and K. WINKLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,763, 24.6.30. Appl., 15.9.23). —See B.P. 228,959; B., 1925, 299.

Concentration of acetic acid. G. SCHWAEBEL and F. SCHLEICHER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,404, 24.6.30. Appl., 10.5.27. Ger., 12.5.26). —See B.P. 295,238; B., 1928, 746.

Production of lactic acid and its derivatives.

M. LUTHER and H. BELLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,715, 24.6.30. Appl., 4.2.28. Ger., 12.2.27).—See B.P. 290,464; B., 1928, 543.

Manufacture of [crystalline] urea.

W. MEISER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,761,893, 3.6.30. Appl., 17.2.27. Ger., 19.2.26).—See B.P. 266,378; B., 1928, 255.

Production of phosphoric esters of aliphatic alcohols.

F. NICOLAI, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,720—1, 24.6.30. Appl., [A] 4.9.28, [B] 4.9.29. Ger., [A, B] 12.10.27).—See B.P. 300,044; B., 1929, 10.

Production of thymol.

H. JORDAN, Assr. to CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (U.S.P. 1,768,257, 24.6.30. Appl., 15.8.27. Ger., 16.8.26).—See B.P. 276,010; B., 1929, 237.

[Manufacture of] formaldehyde-bisulphite [derivatives of] aminoaryl antimony compounds [stibinic acids].

H. SCHMIDT, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,766,361, 24.6.30. Appl., 28.9.28. Ger., 6.10.27).—See B.P. 298,234; B., 1930, 394.

Manufacture of *vic.* - trihalogenobenzenes.

R. HERZ and E. ALBRECHT, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,747, 24.6.30. Appl., 29.8.28. Ger., 5.10.27).—See B.P. 319,149; B., 1930, 95.

Process for introducing sulphocyanic groups into organic compounds.

H. P. KAUFMANN and M. SCHUBERT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,765,678, 24.6.30. Appl., 6.3.28. Ger., 11.3.27).—See B.P. 303,813; B., 1929, 236.

Intermediates derived from aminosulphones.

K. H. SAUNDERS, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,951—2, 24.6.30. Appl., [A] 31.8.28, [B] 10.10.29. U.K., 21.10.24).—See B.P. 245,865; B., 1926, 233.

Preparation of sulphur-containing hydroxyquinones.

F. MAYER and K. ZAHN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,687, 24.6.30. Appl., 11.8.28. Ger., 7.9.27).—See B.P. 296,761; B., 1929, 807.

Manufacture of condensation products of the benzanthrone series.

G. KALISCHER, H. SCHEYER, P. NAWIASKY, and E. KRAUCH, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,768,071, 24.6.30. Appl., 17.6.27. Ger., 28.6.26).—See B.P. 296,490; B., 1928, 847.

Manufacture of intermediate compounds and of

azo dyes therefrom. M. MENDOZA, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,949, 24.6.30. Appl., 25.5.28. U.K., 27.5.27).—See B.P. 297,855; B., 1928, 887.

Hexamethylenetetramine-triphenol for light

filters (U.S.P. 1,751,220).—See I. Depolymerisation of hydrocarbons (B.P. 302,899). Ketone fuels (F.P. 637,993).—See II. Vat dye intermediates (B.P. 305,489).—See IV. Oxidisable compounds (B.P. 316,251).—See XIV. Polyhydric alcohols (B.P. 327,193).—See XVII.

IV.—DYESTUFFS.**Colour lakes.** CLAYTON.—See XIII.**PATENTS.**

Manufacture and use of dyes [derived from naphthazarin]. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 327,860, 1.8.28).—Naphthazarin or a derivative, *e.g.*, methylnaphthazarin (obtained from toluquinol and succinic anhydride), is condensed with aqueous ammonia, preferably in presence of a reducing agent (or the leuco-naphthazarin may be used) and a copper salt. Grey to violet-grey dyes for acetate silk are obtained. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,248, and 328,793, [A], 12.1.29, [B] 1.5.29).—(A) A 1 : 1'-dianthraquinonylamine, containing a 4 : 3-thioxanthone ring and in the other anthraquinone nucleus either a 3' : 4'-acridone ring or an acylamino-group, is treated at 20° with sulphuric acid to give a carbazole derivative. 4-Aminoanthraquinone-2 : 1-thioxanthone is condensed with 5-chloro-1-benzamidoanthraquinone or 4-chloro-5-methoxy-1-benzamidoanthraquinone and the product is carbazolised to give a black-brown vat dye. A grey-green vat dye is obtained by carbazolisation of the product from 4-aminoanthraquinone-2 : 1-thioxanthone and 4 : 3' : 5'-trichloroanthraquinoneacridone. (B) 1-Chloroanthraquinone-2-carboxylic acid is condensed with 2-amino- or 2 : 6-diamino-anthracene and the product is cyclised to an anthraquinoneacridone or anthraquinonediacridone, both of which are red-brown vat dyes. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 305,488, 4.2.29. Ger., 3.2.28).—Phenanthridone is condensed with phthalic anhydride in presence of aluminium chloride at 180—230° to give two isomeric phthaloylphenanthridones, separable by crystallisation from nitrobenzene or trichlorobenzene. The main product is a yellow vat dye; the less soluble product gives paler shades. C. HOLLINS.

Manufacture of vat dyes of the benzanthrone series. I. G. FARBENIND. A.-G. (B.P. 305,679, 9.2.29. Ger., 10.2.28, and Addn. B.P. 328,993, 9.2.29).—(A) Benzanthrone-1-aminoanthraquinones, having a free 2-position and an aroylamino-substituent in the anthraquinone residue, are treated with acid condensing agents (sulphuric or chlorosulphonic acid) to give brown vat dyes. Examples of starting materials are the products from 1-amino-5-benzamidoanthraquinone with 3-bromobenzanthrone, and 1-amino-4-benzamidoanthraquinone with 9-chlorobenzanthrone. (B) Aluminium chloride is used as condensing agent. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,334, 27.11.28. Addn. to B.P. 307,328; B., 1929, 388).—Halogenated (*etc.*) dibenzanthrones and isodibenzanthrones are condensed with other amino-compounds than aminoanthraquinones, preferably those capable of being vatted; *e.g.*, bromodibenzanthrone with aminoanthanthrone (dark reddish-blue), bromoisodibenzanthrone with aminopyranthrone (dark blue),

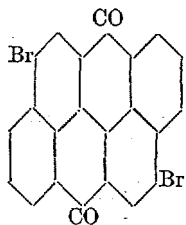
dibromodibenzanthrone with aminoanthanthrone (dark blue), chlorotetrabromoisodibenzanthrone with aminoanthanthrone (dark blue), dibromoisodibenzanthrone with the carbazole produced by heating 4-benzamido-1:1'-dianthraquinonylamine with sulphuric acid (violet).

C. HOLLINS.

Manufacture of halogen derivatives [vat dyes] of the dibenzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,263, 4.6.29. Cf. B.P. 325,525; B., 1930, 453).—Dibenzanthrone or a bromo-derivative is brominated by grinding with bromine together with a carrier (iodine and iron). The product is a reddish-navy-blue vat dye.

C. HOLLINS.

Manufacture of vat dyes and intermediates of the anthanthrone series. I. G. FARBENIND. A.-G. (B.P. 305,489, 4.2.29. Ger., 3.2.28).—A halogenated anthanthrone is heated with sulphur or an agent yielding sulphur. 4:10-Dibromoanthanthrone (annexed formula) heated in nitrobenzene at 130–145° with potassium xanthate yields the 10:10'-dibromosulphide, which is a reddish-violet vat dye. C. HOLLINS.



Manufacture of vat dyes of the 3:4:8:9 [1:2:6:7]-dibenzpyrene -[3:8]-quinone series. I. G. FARBENIND A.-G. (B.P. 310,891, 2.5.29. Ger., 2.5.28).—Dihalogenated (particularly dibrominated) 1:2:6:7-dibenzpyrene-3:8-quinones are improved in shade and in fastness to washing by treatment with an alkaline oxidant, e.g., alkaline hypochlorite. C. HOLLINS.

Manufacture of vat dyes containing sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,705, 13.2.29).—Anthraquinonylthiol-benzanthrones or -dibenzanthronyls, other than those derived from 2- and 3-thiolbenzanthrones, are fused with alkali to give vat dyes. Dyes are thus obtained from 9- α -anthraquinonylthiolbenzanthrone (navy-blue), 9:9'-di- α -anthraquinonylthiol-2:2'-dibenzanthronyl (navy-blue), 1:1'- α -anthraquinonylthiolbenzanthrone (greenish-blue).

C. HOLLINS.

Manufacture of monoazo dyes [for wool]. I. G. FARBENIND. A.-G. (B.P. 305,174, 31.1.29. Ger., 1.2.28). An amine of the benzene series is diazotised and coupled with a sulphonic acid of 2-phenylindole. Examples are: *p*-aminoacetethylanilide \rightarrow 2-phenylindole-7-sulphonic acid (yellow); 2:4:6-trichloroaniline \rightarrow 2-*m*-sulphophenylindole (orange). The dyes are fast to fulling.

C. HOLLINS.

Manufacture of azo dyes [for wool, viscose silk, or acetate silk]. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 329,049, 21.2.29).—A mono-oxamic acid of 4:4'-diaminodiphenyl sulphide, m.p. 108°, or trisulphide, m.p. 122°, is diazotised and coupled with a coupling component. The azo dye may be hydrolysed, diazotised, and coupled further, or in suitable cases may be coupled with diazo compounds. Examples are: the mono-oxamic acid, m.p. above 265°, of the monosulphide [4-*p*-aminophenylthiol-phenyloxamic acid] \rightarrow phenyl-J-acid (bluish-red on viscose silk), 2S-acid (violet on viscose silk), phenyl- γ -

acid (brown on wool), phenylmethylpyrazole (yellow on acetate silk), Brönnner acid (orange on wool), γ -acid (red on wool or viscose silk), or *m*-tolylenediamine \leftarrow 2:4-dinitroaniline (red-brown on acetate silk); the trisulphide-oxamic acid, m.p. above 290°, \rightarrow salicylic acid (yellow on acetate silk or wool), J-acid (yellowish-red on viscose silk), or *m*-phenylenediamine \rightarrow *p*-chloroaniline (orange-brown on acetate silk). C. HOLLINS.

Manufacture of monoazo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 303,838, 10.1.29. Ger., 10.1.28).—A monoaroyl-*p*-phenylenediamine is diazotised and coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. E.g., 6- α -naphthamidocresidine, m.p. 183–184°, obtained from 6-nitrocresidine, is coupled with the 5-chloro-*o*-toluidide for a bluish-violet. C. HOLLINS.

Manufacture of azo dyes [for lakes]. MAJOR & Co., LTD., H. H. HINCHLIFFE, and W. J. DARBY (B.P. 328,292, 19.1.29).—The monoazo dyes from diazotised toluidinesulphonic acids (e.g., *p*-toluidine-3-sulphonic and 5-chloro-*o*-toluidine-4-sulphonic acids) coupled with 2:3-hydroxynaphthoic acid are condensed with 1 or 2 mols. of an arylsulphonyl chloride (e.g., toluene-*p*-sulphonyl chloride) in presence of caustic alkali.

C. HOLLINS.

Manufacture of disazo dyes. E. F. HITCH, H. JORDAN, and A. O. BRADLEY, ASSRS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,739,031, 10.12.29. Appl. 7.8.26).—A non-phenolic arylamine is diazotised and coupled with a non-phenolic middle component; the resulting aminoazo compound is nitrobenzoylated, reduced, diazotised, and coupled with a coupling component. Examples are: β -naphthylamine-6:8-disulphonic acid \rightarrow *m*-toluidine, *m*- or *p*-nitrobenzoylated, reduced, \rightarrow *p*-aminobenzoyl-J-acid (orange on cotton, redder by development with β -naphthol); Brönnner acid \rightarrow α -naphthylamine, *p*-nitrobenzoylated, reduced, \rightarrow S-acid (bluish-brown on cotton, full brown with β -naphthol, yellow-brown with phenylmethylpyrazolone); β -naphthylamine-6:8-disulphonic acid \rightarrow *o*-anisidine, *p*-nitrobenzoylated, reduced, \rightarrow 1-*m*-aminophenyl-3-methyl-5-pyrazolone (yellow on cotton); etc.

C. HOLLINS.

Manufacture of [dis]azo dyes and their application to the dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 329,056, 25.2.29).—A diaminodiphenyl, in which not more than one amino-group is in the 4-position, is tetrazotised and coupled with coupling components of which at least one is an aminonaphtholsulphonic acid or an *N*-derivative thereof; the diphenyl compounds must carry no phenolic, nitro-, carboxyl, or sulphonic groups. Examples are: 3:3'-diaminodiphenyl \rightarrow 2 mols. of γ -acid (brown); diphenylene \rightarrow 2 mols. of phenyl- γ -acid (brown); 2:2'-diaminodiphenyl with salicylic acid and phenyl- γ -acid (brown) or with *o*-cresotic acid and 1:5:7-aminonaphtholsulphonic acid (bluish-red); etc. The dyes give level shades on viscose silk.

C. HOLLINS.

Manufacture of azine dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 328,291, 18.1.29).—An *o*-phenylenediamine is condensed with an 8-acylamino-

1:2-naphthaquinone-4- or -5-sulphonic acid. The acyl groups may be removed by dilute sulphuric acid, the sulphonic group by concentrated acid; the amino-group may be aroylated. From *o*-phenylenediamine and 8-acetamido-1:2-naphthaquinone-5-sulphonic acid are obtained 1-acetamido- α -naphthaphenazine-4-sulphonic acid (greenish-yellow on wool), the 1-amino- (red-orange), 1-benzamido- (reddish-yellow), 1-*o*-chlorobenzamido- (orange), and 1-*m*- or -*p*-nitrobenzamido- (greenish-yellow) compounds, 1-amino- α -naphthaphenazine and its benzoyl (m.p. 218—219°), acetyl (m.p. 233—234°), and *p*-nitrobenzoyl (m.p. 305—307°) derivatives; *o*-phenylenediamine-4-sulphonic acid gives a mixture of 4:8- and 4:9-disulphonic acids (pure yellow on wool), from which the 1-amino- and 1-*p*-nitro-benzamidodisulphonic acids are obtained. 8-Acetamido-1:2-naphthaquinone-4-sulphonic acid yields similar products. The wool dyes are very fast to light; the desulphonated aminonaphthaphenazine may be used for dyeing acetate silk.

C. HOLLINS.

Yellow vat dyes. G. KRÄNZLEIN, H. GREUNE, and H. VOLLMANN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,767,377, 24.6.30. Appl., 27.11.25. Ger., 16.2.24).—See F.P. 593,117; B., 1926, 266.

Preparation of [vat-dye] derivatives of 1:4:5:8-naphthalenetetracarboxylic acids. W. ECKERT and H. GREUNE, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,661, 24.6.30. Appl., 27.1.27. Ger., 9.2.26).—See B.P. 265,964; B., 1928, 225.

Alkaline condensation products [vat dyes] of Bz-1-ethers of benzanthrone. K. THIESS, W. GMELIN, and K. ZAHN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,413, 24.6.30. Appl., 19.7.26. Ger., 26.8.25).—See B.P. 257,618; B., 1928, 83.

Anthraquinone dyes and dyeing therewith. A. SHEPHERDSON and W. W. TATUM, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,761,369 and 1,761,370, 3.6.30. Appl., [A] 9.2.27, [B] 27.1.28. U.K., [A, B] 19.8.26. Renewed [B] 21.10.29).—See B.P. 275,421; B., 1927, 771.

Azo dyes. H. WAGNER, O. SOHST, and J. RACHOR, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,640, 24.6.30. Appl., 27.8.28. Ger., 19.9.27).—See B.P. 297,362; B., 1930, 365.

Manufacture of fast azo dyes. L. LASKA and A. ZITSCHER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,681, 24.6.30. Appl., 27.8.28. Ger., 5.9.27).—See B.P. 309,216; B., 1929, 552.

Manufacture of [chromable] azo dyes. K. H. SAUNDERS, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,947, 24.6.30. Appl., 10.6.26. Renewed 10.9.29. U.K., 4.8.25).—See B.P. 256,775; B., 1926, 866.

Azo dyes derived from aminosulphones. K. H. SAUNDERS, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,946, 24.6.30. Appl., 29.8.25. U.K., 21.10.24).—See B.P. 245,865; B., 1926, 233.

(A) Mordant azo dyes. (B) [Dis]azo dyes. M. MENDOZA and K. H. SAUNDERS, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,948 and 1,766,950, 24.6.30. Appl., [A] 29.9.26, [B] 31.8.28. U.K., [A, B], 13.11.25).—See B.P. 262,243; B., 1927, 101.

[Production of] naphthacarbazolehydroxyazo dyes. H. GRIMMEL and H. CLINGESTEIN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,434, 24.6.30. Appl., 28.6.26. Ger., 26.6.25).—See F.P. 617,211; B., 1927, 902.

Production of complex metal compounds of α -hydroxyazo dyes. H. KRZIKALLA, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,680, 24.6.30. Appl., 27.8.27. Ger., 28.8.26).—See B.P. 296,819; B., 1928, 849.

Treatment of complex metallic compounds of α -hydroxyazo dyes. H. KRZIKALLA and H. KÄMMERER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,767,379, 24.6.30. Appl., 24.3.27. Ger., 31.3.26).—See B.P. 268,754; B., 1928, 667.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Bark of the young woods of spruce, pine, and red beech. C. G. SCHWALBE and K. E. NEUMANN (Cellulosechem., 1930, 11, 113—128).—The properties of the cambial layers of the three woods have been investigated with a view of formulating a simple process for the removal of bark prior to pulping. Woods felled in spring and in autumn have been examined, and their chemical characteristics are differentiated. In the spring woods the cambial cells are soft and the bark is easily removed, the contrary being the case in the autumn woods, which are of the greatest practical interest. Extraction of all three woods with water gave negligible amounts of free sugars. By means of acid hydrolysis higher yields of sugars are obtained from the cambial layers of the coniferous woods than from the heart-wood, indicating higher concentration of hemicelluloses. Tests for pentoses among the sugars produced by hydrolysis gave negative results, hexoses alone being found. Similar treatment of the bark of the deciduous wood (beech) shows that the concentration of easily hydrolysed hemicelluloses is lower than in the body of the tree, as is also the pentosan content. The hexosan content being even lower, both in the bark and in the wood, it is held that the pentosans are very difficultly hydrolysed, concentrated acids or alkalis being necessary. Treatment with lime- and baryta-water failed to affect the pentosan content of beech bark, but in the case of the wood the content was lowered, due to the presence in the pentosan complex of acid groupings of low mol. wt. Extraction with ether shows the bark of both classes to yield larger extracts than do the corresponding woods, the ratio being higher in beech, although the absolute quantity is lower. In order to attempt to trace the differences in character of the barks of the two types of wood, the saps flowing through the trees in spring were investigated, without positive result.

T. T. POTTS.

Extraction of pure cellulose from plant raw materials (Reports 2—4). Pretreatment prior to chlorination. I.—III. K. KANAMARU (J. Soc. Chem. Ind., Japan, 1930, 33, 78—80 B, 80 B, 81—82 B).—The effect produced by various treatments prior to chlorination on the extraction of cellulose by Cross and Bevan's method has been investigated. I. The best pretreatment, in order to remove non-cellulosic material and to cause swelling of the cell-walls without loss or

degradation of cellulose, in the case of wood sawdust (previously extracted with ether and alcohol) is to immerse the material in the concentrated caustic soda solution at room temperature for 24 hrs. (Applied in the determination of cellulose, this method gives the purest product in maximum yield by the subsequent chlorination.) Purification with hot dilute caustic solution lowers the yield of cellulose and α -cellulose. Treatment with 20–60% calcium chloride solution at 60° is also effective. Pentosans can be removed completely by digesting a 1.2–1.3% suspension of α -cellulose in water for 70 hrs. at 35° with the powder (to the amount of 0.07–0.08% of malt extract) obtained by pouring a water extract of dry malt meal into alcohol (p_H adjusted to 4–5).

II. The best conditions (concentration, temperature, etc.) for the pretreatment are discussed theoretically; the experimental results are interpreted on the basis of the theories of von Weimarn on the dispersion of cellulose in salt solutions and of Katz on swelling.

III. Rice straw has been investigated as in I. The fibrous tissue of the straw is porous and contains a large amount of non-cellulosic material, so that pretreatment is itself a process of cellulose extraction: treatment with hot dilute caustic soda is the most effective, but partial loss of cellulose is unavoidable. Pretreatment of the straw with malt extract (which can be used to remove all pentosans) causes a loss of 6–8% in weight (mostly pentosans) and increases the yield of cellulose, but greatly diminishes the content of α -cellulose. Treatment with malt extract converts the cellulose of straw, wood, and cotton into a form soluble in alkali.

E. LEWKOWITSCH.

Wood-pulp testing: a plea for standardisation. G. H. GEMMELL (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 381–386).—The necessity for the standardisation of pulp-testing methods is urged. The fat and rosin content of various woods has been investigated, and it is concluded that the fatty-alcoholic extract is responsible for most of the "pitch" troubles of the papermaker. The addition of paraffin of d 0.8–0.85 to the pulp during bleaching is recommended.

T. T. PORTS.

Microscopical examination of pulps for rosin. T. T. PORTS (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 339–340).—A modification of the Sudan III test is described. The pulp is stained in a 0.2% solution of Sudan III in 70% alcohol for 1 hr., washed rapidly with 50% alcohol, cleared in 50% glycerin, and mounted in glycerin jelly. Rosin and fats are stained red, the fibres remaining colourless. Attention is drawn to the distribution of rosin in pulps.

T. T. PORTS.

Viscose. III. Determination of free and thiocarbonate-carbon disulphide in viscose. Their change during ripening of viscose. IV. Volumetric analysis of viscose containing sodium sulphite. Change of each component during the ripening of viscose. K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 50–51 B, 52–54 B; cf. B., 1930, 322).—III. With T. TAKAHASHI.] The free carbon disulphide content of viscose is determined by shaking an approx. 10% aqueous solution of viscose with ether for 15 min.,

treating the ethereal solution with ethyl-alcoholic potassium hydroxide, acidifying with acetic acid, neutralising with calcium carbonate, and titrating the aqueous layer with iodine. The total carbon disulphide, except cellulose xanthate, content is obtained by trituration of the viscose with solid sodium chloride, washing with excess of saturated sodium chloride solution, extraction of the filtrate with ether after previous acidification with acetic acid, and then proceeding as described above. The difference between these two values is due to sodium thiocarbonate. In the ripening of viscose containing sodium sulphite the free carbon disulphide decreases and the thiocarbonate-carbon disulphide increases.

IV. [With S. MIYOSHI.] A method is described for the volumetric determination of the components cellulose xanthate, sodium hydroxide, carbonate, sulphide, thiocarbonate, thiosulphate, and sulphite of viscose, and is applied to the determination of the variation of these components during the ripening process.

A. I. VOGEL.

Viscose. V. Sodium sulphate in viscose containing sodium sulphite. VI. Influence of sodium sulphite on viscose. K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 144–145 B, 146–147 B).—V. [With T. TAKAHASHI.] A method for the determination of sodium sulphate in viscose containing sodium sulphite is described; the content of sodium sulphate remains constant during ripening. In the case of cellulose xanthate, sodium sulphite corresponding to 0.14% of the viscose was oxidised to sulphate.

VI. [With S. MIYOSHI.] The changes undergone by viscose containing added sodium sulphite, with regard to content of sodium hydroxide, carbonate, sulphide, thiocarbonate, thiosulphate, sulphite, and cellulose xanthate alkali, during ripening have been studied, and compared with those occurring under the same conditions in viscose without added sodium sulphite. A part of the sodium sulphite is converted into sulphate, but the remainder does not change during ripening; the sulphite thus eliminates dissolved oxygen and retards the decomposition of cellulose xanthate. The formation of thiocarbonate from the carbon disulphide set free in the decomposition proceeds slowly. Both processes retard the coagulation of the viscose. The requisite quantity of commercial sodium sulphite ($\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$) is 3 kg. per 100 kg. of pulp.

C. W. SHOPPEE.

Viscose. XXIX. Properties of viscose prepared from alkali-cellulose immersed for a long time in alkali, and of the resultant spun fibres. T. NAKASHIMA, J. MURAKAMI, and S. OHORA (J. Soc. Chem. Ind., Japan, 1930, 33, 44 B).—Reduction in viscosity of viscose leads generally to weak fibres, but under certain conditions strong fibres can be spun from cellulose, the viscosity of which has been reduced by prolonged immersion in alkali.

A. I. VOGEL.

Viscose. XXXI. Effect of caustic alkali on cellulose (Part 2). XXXII. Effect of chlorination on cellulose. G. KITA (J. Soc. Chem. Ind., Japan, 1930, 33, 142–143 B, 143–144 B).—XXXI. [With T. NAKASHIMA, S. OHORA, and J. MURAKAMI.] The viscosity of commercial cellulose in cuprammonium solution is unexpectedly decreased by steeping in caustic alkali in an

atmosphere of hydrogen, and the copper number of the product obtained is distinctly diminished (cf. Nakashima, B., 1929, 917). To find whether cellulose by removal of oxidised products after milling would exhibit the original viscosity (cf. Waentig, B., 1927, 327), the products obtained from cotton paper by thorough milling in a hollander or by dissolution in concentrated zinc chloride solution and reprecipitation, and subsequent steeping for a long period, have been examined. In both cases the viscosity in cuprammonium solution is lower than that of the original cellulose, even if this has been treated by steeping. It is concluded that the decrease in viscosity produced by milling is due to other causes than oxidation.

XXXII. [With S. MAZUDA and T. SUZUKA.] The copper number of cellulose diminishes with repeated chlorination; the viscosity of the chlorinated product is slightly less than that of the original material, but is unaltered by repetition of the chlorination. The viscose threads spun from the chlorinated products possess the same properties as the original material, and are superior from many points of view. C. W. SHOPPEE.

Elimination of "dirt" from paper stock. R. H. CLAPPERTON (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 341—354).—A summary of the methods employed for the removal of dirt from raw materials, half-stuffs, and pulps is given. Centrifugal separation of beaten pulps is advocated, and the operation of this method by means of the "Erkensator" is described at length. T. T. POTTS.

Rosin sizing [of paper]. S. R. H. EDGE (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 289—294).—A continuation of previously published work (cf. B., 1928, 564). Further experimental evidence is given in support of the contention that aluminium sulphate (papermakers' "alum") is essential to sizing. Unbeaten pulps can be sized with rosin, "self-sizing" increasing with degree of beating. Acid aluminium resins have been prepared and found to have considerable sizing action when added to pulp; 2% added to a moderately beaten pulp of p_H 5.0 has, in the presence of a trace of free alum, the effect of hard sizing. T. T. POTTS.

Eliminating personal factors from the measurement of colour and gloss [of paper]. B. DESBLEDS (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 235—282).—The Toussaint "photoelectric colorimeter" is described, and the application of the instrument to the matching of coloured papers is illustrated by means of "colour curves." Light is passed successively through 6 light-filters, covering the visible spectrum, on to a sample of the paper under test. The light reflected from the paper is received by a potassium photoelectric cell, and the intensity measured by means of a galvanometer. The galvanometer deflections plotted against the optimum wave-lengths of the light-filters furnish a "colour curve." Gloss is measured by comparing the galvanometer deflections obtained by means of reflected light from the test sample with that from a standard. T. T. POTTS.

Homogenisers. MÖLLERING. **Medium for flotation cells.** DYER and McCLELLAND.—See I. **Colour lakes.** CLAYTON. **Aircraft finishes.** GARDNER.—

See XIII. **Nitrocellulose.** DEMOUGIN, also BRESSER. —See XXII. **Water purification in the paper mill.** BERRIMAN.—See XXIII.

PATENTS.

Degreasing and opening wool and other fibrous materials. AKTIS PATENT-VERWERTUNGSGES.M.B.H., Assees. of A. UHLMANN (B.P. 304,295, 18.1.29. Ger., 18.1.28).—The material is immersed in water or dilute alkali or acid in a glass or porcelain vessel and exposed to the magnetic field produced by a high-frequency current of, e.g., 3×10^6 cycles per sec. [Stat. ref.]

D. J. NORMAN.

Treatment [oiling] of wool. C. A. BROWN and G. J. ESSELEN, JUN., Assrs. to MANUF. IMPROVEMENT CORP. (U.S.P. 1,749,410, 4.3.30. Appl., 23.7.27).—The oiling of wool is more effectively accomplished by the use of a chemically and mechanically stabilised emulsion of oil in water.

D. J. NORMAN.

Degumming of fibres. E. C. DUHAMEL (U.S.P. 1,748,832, 25.2.30. Appl., 15.3.29. Fr., 5.3.28).—Ramie and like fibres are immersed in a hot suint bath (d 1.056 or above) and then rinsed in warm or cold water.

H. ROYAL-DAWSON.

Retting of textile fibres. M. MARCUS, Assr. to (Mrs.) K. H. CHRISTENSEN (U.S.P. 1,746,316, 11.2.30. Appl., 28.5.28).—Retting is effected by nitrogen-fixing bacteria at 38°.

D. J. NORMAN.

Manufacture of waterproof fabrics, papers, and the like. S. KINUGASA and S. HASHIMOTO (B.P. 314,015, 6.6.29. Jap., 21.6.28).—After impregnation with a solution of soap, the material is dipped into the clear upper liquor obtained by mixing calcium carbonate with an aqueous solution of alum and acetic acid.

F. R. ENNOS.

[Manufacture of] vulcanised fibre. A. F. JACOBS (B.P. 330,394, 30.5.29).—Sheets of cotton cellulose in the form of absorbent paper are treated with a solution of zinc chloride and pressed together between heated rollers; after washing out the zinc chloride, the wet material is immersed in a bath of soluble oil ("hydraulic oil"—an emulsifiable product containing sulphonated oil) and water, and is subsequently dried and pressed between heated platens and rollers. F. R. ENNOS.

Pulp-treating process. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,742,219, 7.1.30. Appl., 1.12.25).—High- α -cellulose pulp is obtained by digesting wood under conditions of high temperature and pressure with a sulphite liquor containing approx. 3—4% each of free and combined sulphur dioxide and agitating the resulting pulp for 0.5—2 hrs. at about ordinary temperature with alkaline solutions of sufficient concentration to remove a substantial proportion of the non- α -cellulose constituents of the pulp without causing mercerisation, e.g., 5—6% caustic soda solution. The action of the alkali is facilitated if the raw sulphite pulp is first treated with 3—4% of bleaching powder. The purified pulp is finally bleached at not above 32°.

D. J. NORMAN.

Production of high- α -cellulose fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,742,218, 7.1.30. Appl., 28.4.25).—Recovery of the heat and sodium compounds from the alkaline digestion liquor used in the

production of α -pulp from sulphite pulp is effected by recirculating the spent liquor and washings with addition of sufficient fresh alkali (to compensate for losses) over further quantities of pulp until the liquor has $d 1.074$ — 1.1154 . The liquor is maintained at this concentration by diverting some of the liquor to a soda-recovery plant and proportionally increasing the flow of water through the countercurrent washer to maintain equilibrium.

D. J. NORMAN.

Manufacture of cellulose esters. G. W. MILES and C. DREYFUS, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,742,611, 7.1.30. Appl., 14.4.26).—Cellulose or its conversion products may be esterified by organic acids alone without the addition of acid anhydrides if the reaction is carried out in the presence of ortho- and/or meta-phosphoric acid. Catalysts, *e.g.*, sulphuric acid, may be used and the cellulose pretreated with glacial acetic acid or mixtures thereof with phosphoric acid. Fourteen examples are given.

D. J. NORMAN.

Cellulose esters or ethers and their uses. G. P. DAVIES, W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,934, 25.10.28).—Aqueous emulsions of cellulose ester or ether solutions, applicable as spraying enamels, are prepared by dispersing in water in a colloid mill, with the aid of a suitable emulsifying agent, a solution of a cellulose ester or ether in a solvent which (a) is immiscible or but slightly miscible with water and is not freely soluble in water, (b) gives a cellulose ester or ether solution of low interfacial tension against the water-phase, and (c) is sufficiently non-volatile to prevent precipitation of the ester or ether during the drying of the film even though the ratio of solvent to water is low and yet is sufficiently volatile to avoid unduly increasing the time of drying. Suitable solvents include methyl cyclohexanone, cyclohexyl acetate, and butyl propionate, and as emulsifying agents there may be used gelatin or casein for pigmented emulsions, or soap, preferably prepared from oleic acid in the non-aqueous phase and caustic soda in the aqueous phase, for unpigmented emulsions.

D. J. NORMAN.

Manufacture of cellulose butyric esters. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 328,259, 22.1.29).—Cellulose tributyrate is dissolved in aqueous butyric (or other aliphatic) acid or a mixture of such acids and hydrolysed for 16—18 hrs. at 45—50° until after precipitation by water the butyrate is soluble in methyl alcohol. Alcohol may be added to homogenise the aqueous acid solution.

C. HOLLINS.

Construction of spinning pots or boxes utilised in the manufacture of artificial silk. J. BRUHL (B.P. 312,041, 6.3.29. Fr., 19.5.28).—The walls of the pots or boxes are made by superimposing layers of fibrous material, such as cellulose or asbestos paper or fabric impregnated with synthetic resin, the resin also serving as an adhesive; the asbestos is arranged at the points at which there is liable to be attack by the material under treatment.

F. R. ENNOS.

Device for use in spinning of artificial silk. LUSTRAFIL, LTD., and G. DOUGILL (B.P. 330,130, 29.5.29).—The spinning funnel is provided with a number of asymmetric constrictions, the inner surfaces of which act as thread guides.

F. R. ENNOS.

Nozzles for spinning artificial silk. E. BRILL (B.P. 314,381, 18.6.29. Ger., 26.6.28).—The nozzle sleeve and base, the latter being provided with spraying apertures, are moulded in one piece from a basic porcelain paste to which is added a quantity of rare earths, *e.g.*, beryl or zircon, and are afterwards baked, whereby the entire surface, including the inner walls of the apertures, are coated with a smooth, hard glaze.

F. R. ENNOS.

Apparatus for the manufacture of artificial threads and the like. COURTAULDS, LTD., F. T. WOOD, and E. H. WELLS (B.P. 330,266, 4.3.29).—The spinning machine is so constructed that the current of air which is drawn in near the rotating guide-wheels is kept separate from that drawn in near the centrifugal boxes, until both currents are about to leave or have actually left the machine.

F. R. ENNOS.

Manufacture of artificial threads, bands, films, etc. I. G. FARBENIND. A.-G. (B.P. 307,758, 12.3.29. Ger., 12.3.28).—In order to avoid aeration of the spinning solution while it passes from the place of manufacture to that of coagulation, the use of gaseous pressure is avoided; the warm solution is transported by means of a mechanical pump or by gravity while subjected to a partial vacuum.

F. R. ENNOS.

Spinning of viscose. I. G. FARBENIND. A.-G. (B.P. 313,503, 12.6.29. Ger., 12.6.28).—The spinning bath contains a sulphonic acid of a lower homologue of the aliphatic series, *e.g.*, methiononic acid, acetaldehyde-disulphonic acid, with or without a mineral acid, a salt, or an organic compound other than the sulphonic acid.

F. R. ENNOS.

Manufacture of artificial fibres. I. G. FARBENIND. A.-G. (B.P. 307,829, 1.3.29. Ger., 14.3.28).—Artificial fibres of denier even less than unity and of high tenacity are produced by spinning non-matured viscose, made from non-matured alkali-cellulose, into a bath containing one or more acid esters formed from a polybasic inorganic acid (sulphuric or phosphoric) and a mono- or poly-hydric aliphatic or aromatic alcohol.

F. R. ENNOS.

Precipitating baths for making threads or the like from viscose. I. G. FARBENIND. A.-G. (B.P. 303,514, 3.1.29. Ger., 5.1.28).—The improved results obtained, particularly in the spinning of unmatured viscose solution, by adding to the coagulating bath organic compounds of high mol. wt. having tanning properties (*e.g.*, sodium carbazolesulphonate) are most marked when the bath is approximately saturated with the organic compound. To effect this with a relatively small quantity of the compound the acid content of the bath is adjusted to 25—42% (calc. as sulphuric acid).

D. J. NORMAN.

Manufacture of artificial silk and like products. W. P. DREAPER (B.P. 328,627, 25.1.29).—Regenerated cellulose filaments or films containing vulcanised rubber are obtained by adding to the spinning solution rubber latex together with reagents which in contact with the coagulating solution will yield sufficient free sulphur to vulcanise the rubber present. Alternatively, the sulphur may be introduced by direct precipitation subsequent to the spinning operation. Vulcanisation is

effected at not above 90° by using an accelerator, such as diphenylguanidine, or a super-accelerator, optionally in the presence of anti-agers. Suitable proportions are 10% of rubber and 1% of free sulphur on the weight of cellulose.

D. J. NORMAN.

Manufacture of filaments, threads, films, etc. from cellulose derivatives. BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 328,911, 5.11.28).—Cellulose ester or other products may be washed free from soluble salts without becoming delustred if the washing is effected with aqueous solutions containing protective agents; e.g., a 10% solution of sodium chloride or sodium acetate. (Cf. B.P. 246,879, 259,265—6; B., 1926, 317, 1008, 976.)

D. J. NORMAN.

Gas container for aircraft. K. HUERTTLE, Assr. to GOODYEAR-ZEPPELIN CORP. (U.S.P. 1,763,586, 10.6.30. Appl., 27.1.28).—See B.P. 304,775; B., 1930, 610.

Conversion of difficultly soluble or insoluble carbohydrate ethers into a soluble state. O. LEUCHS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,767,382, 24.6.30. Appl., 24.6.26. Ger., 27.6.25).—See B.P. 277,111; B., 1927, 860.

Preparation of bodies from acylcelluloses. E. HUBERT, L. LOCK, and O. LEUCHS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,822, 24.6.30. Appl., 17.4.25. Ger., 29.4.24).—See Swiss P. 115,676; B., 1927, 296.

Apparatus for spinning solutions of cellulose esters or ethers. M. KLEIN, Assr. to RUTH-ALDO Co., INC. (U.S.P. 1,767,216, 24.6.30. Appl., 27.3.28. Fr., 22.6.27).—See B.P. 292,561; B., 1929, 595.

Production of artificial threads by the cuprammonium stretch-spinning process. H. WINKELMANN, Assr. to AMER. BENBERG CORP. (U.S.P. 1,761,860, 3.6.30. Appl., 19.1.28. Ger., 20.1.27).—See B.P. 283,923; B., 1929, 390.

Substitute for hard paper, ebonite, fibre, and the like, and its preparation. H. FRIEDLANDER and A. JANSER (U.S.P. 1,766,817, 24.6.30. Appl., 24.6.25. Austr., 26.6.24).—See B.P. 236,224; B., 1926, 986.

Manufacture of [non-curling] adhesive paper. J. MUIR, Assr. to G. H. WILKINSON (U.S.P. 1,766,302, 24.6.30. Appl., 15.6.28. U.K., 2.3.28).—See B.P. 308,444; B., 1929, 469.

Washing machine for raw materials for shoddy. H. WAFFENSCHMIDT (B.P. 331,158, 14.10.29).

Drying of sheets, plates, hanks, etc. SOC. EN NOM COLLECTIF ELECTRICITÉ GÉN. R. PONTIÈRE, and J. LE CLECH (B.P. 312,999, 4.6.29. Fr., 4.6.28).

Processes and devices for drying tubular bodies [composed of regenerated cellulose]. S. SOKAL. From KALLE & Co. A.-G. (B.P. 330,542, 8.3.29).

Scraper blade for detaching threads of cellulosic material from the winding-up drum of a spinning machine. M. DASSONVILLE (B.P. 330,753, 29.5.29. Fr., 30.10.28).

Blotting paper [having one face polished]. G. H. WILKINSON (B.P. 330,756, 31.5.29).

Machines for coating paper, fabric, etc. T. II. L. DIXON (B.P. 330,638, 15.3.29).

Rubberised fabric (B.P. 303,368).—See II. **Treatment of water-absorbent materials (B.P. 326,940).**—See VI. **Hypochlorites from waste lyes (B.P. 305,931).**—See VII. **Compound sheets of glass (B.P. 330,265).**—See VIII. **Plastics containing cellulose derivatives (B.P. 312,604).** **Moulded articles (B.P. 329,273).**—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Wool dyeing, with special reference to the hosiery and tweed trades. J. G. GRUNDY (J. Soc. Dyers and Col., 1930, 46, 157—162).—The principal dye groups which can be applied are the acid, Cloth Fast, Neolan, Chrome Fast, and indigoid vat colours. For dyeing knitted fabrics in fast-to-light fawn, grey, and similar shades it is recommended to use Kiton Fast Yellow 2G, a colour of the Alizarine Sapphire Blue series, and a 1:1 mixture of Kiton Red G and Kiton Fast Red GL or BL, since these dyes fade at equal rates. Cloth Fast dyes are not so level-dyeing as the acid dyes, but yield shades much faster to washing and perspiration; they are much used in the tweed trade. Neolan dyes, which contain chemically combined chromium residues, are applied similarly to acid dyes, but have the fastness properties of Chrome Fast dyes. Since Neolan dyes require a large proportion (5—8%) of sulphuric acid in the dye bath the dyed wool must be thoroughly washed (preferably with dilute ammonia); difficulties in the subsequent processes of milling and back-washing have been traced to residual acid retained by the wool. The fastness properties of a very large number of dyes are tabulated. Wool treated for $\frac{1}{2}$ hr. at the boil with 80% of tannic acid and 6% of acetic acid (40%), and then fixed in a bath containing 5% of stannous chloride and 5% of chromium acetate (*d* 1.16) for $\frac{3}{4}$ hr. at 60° loses its affinity for Acid, Cloth Fast, Neolan, and Chrome Fast dyes; wool thus treated can be used in materials required in two colours. A list of dyes unaffected by this reserving process is given.

A. J. HALL.

Standardisation of tests for fastness of dyed fabrics. S. G. BARKER (J. Soc. Dyers and Col., 1930, 46, 162—164).—Results of investigations previously carried out (cf. Hedges, B., 1928, 260; King, B., 1928, 2639, 707; Cunliffe, B., 1929, 280) are summarised.

A. J. HALL.

Dyeing of furs. DOHOGNE.—See XV.

PATENTS.

Bleaching sensitive vegetable and animal fibres and materials, more particularly feathers, skins, pelts, and hairs (bristles) by treatment with oxidising bleaching agents. V., J., and R. BÖHM (BRÜDER BÖHM), Assees. of H. GOLDARBEITER (B.P. 310,030, 11.4.29. Austr., 21.4.28).—Bleaching is carried out by means of alkaline hydrogen peroxide in the presence of an oil mordant (a sulphonated oil such as Turkey-red oil) and a decomposition catalyst for hydrogen peroxide (salts of copper, nickel, cobalt, or silver).

F. R. ENNOS.

Dyeing cotton and artificial silk with indigosols. L. LIBMANN (F.P. 637,833, 18.7.27).—Fabric is mordanted with a dioxide (*e.g.*, of manganese or lead), washed, and dyed in a solution of an indigosol also containing sulphuric, hydrochloric, or acetic acid. Pattern effects are obtained by discharging the mordant with reducing agents before dyeing. A. J. HALL.

Manufacture of coloured fabrics. [Application of vat dye resists to yarn before weaving.] J. MORTON, E. G. HARRIS, J. I. M. JONES, and MORTON SUNDOWN FABRICS, LTD. (B.P. 328,996, 6.11.28).—Sodium *m*-nitrobenzenesulphonate is applied in hot 25% aqueous solution, with or without size, to white or coloured yarn, which after weaving with untreated fibres is dyed or printed with vat dyes. C. HOLLINS.

Dyeing of piece goods and yarns with vat dyes. I. G. FARBERIND. A.-G. (B.P. 305,230, 1.2.29. Ger., 2.2.28).—Well penetrated dyeings are obtained by impregnating tightly beaten fabrics, or fabrics containing sized or tightly twisted yarns, with a liquor containing an unvat dye and an agent for promoting "bleeding," and then treating with an alkaline reducing liquor containing sodium hyposulphite. Suitable "bleeding" agents include an emulsion of an oil, or fat, or a free acid or water-soluble derivative thereof, or a cellulose derivative capable of forming in water a neutral or alkaline colloidal solution such as an alkali salt of carb-oxy-methylcellulose (*cf.* B.P. 317,117; B., 1929, 894).

A. J. HALL.

Dyeing of viscose. I. G. FARBERIND. A.-G. (B.P. 306,153, 15.2.29. Ger., 17.2.28).—Uniform shades on viscose silk are obtained by using couplings of tetra-azotised *m*- or *p*-azoxyanilines, which may also be produced by alkaline reduction of couplings of diazotised *m*- or *p*-nitroanilines. Examples are: *p*-nitroaniline-*o*-sulphonic acid \rightarrow β -naphthylamine-7-sulphonic acid, reduced to azoxy-compound (reddish-blue); *m*-nitroaniline \rightarrow β -naphthol-8-sulphonic acid, reduced to azoxy-compound (orange); *p*-nitroaniline-*o*-sulphonic acid \rightarrow phenol, methylated, reduced to azoxy-compound (golden-orange), etc. C. HOLLINS.

Dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 328,988 and 329,014, [A] 8.2.29, [B] 7.2.29).—Viscose silk is dyed in level shades (A) with monoazo couplings of an oxamic acid of the benzidine series with a naphthol- or naphthylamine-sulphonic acid, a 1:8-aminonaphthol- or dihydroxynaphthalene-sulphonic acid, or a *m*-diamine, or (B) with disazo dyes of the type: *p*-nitroaniline etc. (excluding nitroanisidines) \rightarrow a naphthol, a naphtholsulphonic acid, or an *N*-substituted 2:8-aminonaphtholsulphonic acid, reduced, \rightarrow 1:8- or 2:8-aminonaphthol-mono- or -di-sulphonic acid. Examples are: (A) benzidineoxamic acid \rightarrow N.W.-acid (bluish-scarlet), S-acid (brownish-violet), H-acid (violet), *m*-phenylenediamine (brown); dianisidineoxamic acid \rightarrow 2S-acid (bluish-violet), H-acid (bluish-violet), naphthionic acid (red-orange); (B) *p*-nitroaniline \rightarrow 8-hydroxy-2:2'-dinaphthylamine-3:6-disulphonic acid, reduced, \rightarrow H-acid (blue); *p*-nitroaniline \rightarrow N.W.-acid, reduced, \rightarrow 2S-acid (blue); 5-nitro-*o*-toluidine \rightarrow 2:7-naphtholsulphonic acid, reduced, \rightarrow H-acid (greenish-

blue); *p*-aminoacetanilide \rightarrow Schäffer acid, hydrolysed, \rightarrow S-acid (Prussian-blue). C. HOLLINS.

Production of coloured articles made from cellulose esters. I. G. FARBERIND. A.-G. (B.P. 308,780, 14.2.29. Ger., 29.3.28).—Threads and films manufactured from chlorinated organic acid esters of cellulose (*cf.* B.P. 306,132; B., 1930, 504), or mixtures of such derivatives with similar non-chlorinated esters (*e.g.*, cellulose acetate), are dyed with basic dyes; the chlorinated derivatives are more easily dyed than the non-chlorinated. A. J. HALL.

Coloration of fabrics [containing cellulose esters or ethers]. BRIT. CELANESE, LTD. (B.P. 306,534, 4.2.29. U.S., 23.2.28).—In printing fabrics or fibres containing cellulose derivatives with vat dyes which produce delustering of the silk, an addition of one or more of the following agents is made to the printing paste to prevent or inhibit the loss of lustre: methyl and ethyl ethers of ethylene glycol, diacetone alcohol, ethylene glycol with or without alcohol, benzyl alcohol, triacetin, cyclohexanone, potassium or ammonium thiocyanate, inorganic salts (*cf.* B.P. 246,879; B., 1926, 317), sugar (*cf.* B.P. 259,266; B., 1926, 977), phthalimide, and xylol monomethylsulphonamide. [Stat. ref.]

A. J. HALL.

Printing of materials made from or containing cellulose esters or ethers. IMPERIAL CHEM. INDUSTRIES, LTD., A. SHEPHERDSON, and L. SMITH (B.P. 330,652, 19.12.28).—The printing strength of aminoanthraquinone (*cf.* B.P. 211,720; B., 1924, 375) is much improved by the addition of up to 5% of a hydroxyethylamine, particularly triethanolamine, to the printing paste.

A. J. HALL.

Production of prints on cloth or like material. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 329,207, 22.6.29).—Cloth printed with vat or sulphide dyes to which has been added aqueous polyvinyl alcohol (with or without other thickening agent), but no alkali, may be subsequently slop-padded in an alkaline (reducing) bath and steamed without danger of the colour running. A mixture of 1 pt. of aqueous polyvinyl alcohol and 3 pts. of 10% starch paste is effective.

C. HOLLINS.

Discharge effects on cellulose acetate silk. I. G. FARBERIND. A.-G., Assees. of A. FISCHESSE (G.P. 461,753, 18.6.26).—Dyed cellulose acetate silk fabric is printed with zinc salts of formaldehyde-sulphoxylic acid, then steamed, and treated in the usual manner. Non-dischargeable basic dyes may be added to the discharge paste for the production of multi-coloured effects.

A. J. HALL.

Treatment of wool, fur, or like materials with organic liquids. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 330,598, 9.3.29).—The usual objectionable grey or white appearance produced by treating wool materials with an organic solvent and then evaporating this (as in moth-proofing) may be avoided by adding 1–2% of a second solvent or mixture of solvents having a high b.p., *e.g.*, butyl and benzyl alcohols, cyclohexanol, cyclohexanone, methyl phthalate. A. J. HALL.

Decolorising [cellulosic] film. E. B. MIDDLETON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P.

1,743,155, 14.1.30. Appl., 31.12.25).—Film is treated with a chlorine bleaching liquor or gas and then with a liquor containing activated charcoal and an organic solvent (ethyl alcohol) capable of dissolving the products of decomposition of the dyes originally present without attacking the film. A. J. HALL.

Obtaining effects on textile materials of vegetable, animal, or other origin. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,978, 5.11.28).—Preparations containing "mono- or di-methylolurea" are applied locally to textiles, or are applied generally and removed from portions by solvent. Such preparations in printing pastes give clean prints, or without dyes they lead to lustre and damask effects. On mercerisation of the printed goods the methylolureas protect the textile, and crêpe effects are obtained. C. HOLLINS.

Delustring of artificial filaments, yarns, or threads. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 328,247, 8.1.29).—All types of artificial silk threads are delustred to any desired degree by subjection to a process of abrasion in which the threads are led over abrasive surfaces, or over loose abrasive material, or in which a stream of the abrasive material dispersed in a liquid medium is projected on the moving threads. Suitable abrasives include kieselguhr (to pass 120-mesh for cellulose acetate silk), pumice powder, powdered glass, carborundum, and emery. Other abrasives which are soluble in solvents and can thus be completely removed from the delustred threads are sodium sulphate, alum, and sodium chloride. A. J. HALL.

Weighting of silk. A. FEUBEL (U.S.P. 1,749,699, 4.3.30. Appl., 17.11.26. Ger., 28.11.24).—Natural or artificial silk is impregnated with basic aluminium nitrate solution obtained by dissolving freshly precipitated aluminium hydroxide in about half the equivalent quantity of nitric acid or by dissolving highly basic chloride or sulphate of aluminium in the least possible quantity of nitric acid. The aluminium is finally fixed on the fabric as its silicate or other insoluble compound. D. J. NORMAN.

Sizing of fibrous materials. E. PINEL (F.P. 637,654, 18.11.26).—The fibres are treated with a size containing carob-bean gum (tragasol), glycerin, and water, and then with another prepared with flour, dextrin, and wax. A. J. HALL.

Treatment of fibrous materials. I. TUDOR and O. TUDOR-HART (B.P. 328,638, 2.11.28).—A resilient character resembling that of horse hair may be imparted to vegetable fibres such as coir by first boiling the fibre for about 1 hr. with 3–5% caustic soda solution, rinsing, drying, preferably at 80° with continuous teasing, and finally polishing the fibre. D. J. NORMAN.

Treatment of fibres and fabrics. H. LIVSEY, G. E. HOLDEN, and J. & J. M. WORRALL, LTD. (B.P. 328,831, 30.5.29. Addn. to B.P. 313,980; B., 1929, 679).—In the process of the prior patent, animal glues and sizes are employed instead of the gelatin. H. ROYAL-DAWSON.

Treatment of water-absorbing materials to render them non-absorbent, acid-resisting, and alkali-resisting. H. N. MORRIS (B.P. 326,940, 2.2.29).—Paper, textiles, building materials, etc. are

impregnated with a common solution of rubber and one or more salts of cellulose prepared as described in B.P. 274,968 (B., 1927, 789), and are then dried. A. J. HALL.

Imparting bacteria- [mildew]-resistant properties to textile materials. C. B. WHITE and E. SCHAEFER, ASSTS. to VIVATEX PROCESSES, INC. (U.S.P. 1,745,134, 28.1.30. Appl., 23.10.26).—Sized cellulosic yarns and fabrics are protected from attack by mildew by the previous addition to the size of soluble compounds of rare-earth elements such as cerium, lanthanum, didymium, uranium, zirconium, and particularly thorium; 0.5–2.0% (calc. on the weight of the size) of thorium fluoride gives practically complete resistance to mildew attack. A. J. HALL.

Cleaning of wool material stained with tar- and pitch-tips. I. G. FARBENIND. A.-G. (B.P. 309,871, 16.4.29. Ger., 16.4.28).—The wool is treated in two successive baths, the first being a clear concentrated emulsion of heavy benzol (etc.) and ammonia, the second an emulsion containing less benzol or merely a solution of an alkali salt of the emulsifying agent, which in each case is a mixed fatty aromatic sulphonic acid, such as dibutyl-naphthalenesulphonic acid. C. HOLLINS.

Carrying out washing operations [on textiles] with hard water. G. ULLMANN and W. SECK (U.S.P. 1,746,170, 4.2.30. Appl., 9.11.28. Austr., 5.8.27).—In addition to ordinary soap, certain sulphonic compounds having colloidal properties and forming water-soluble calcium soaps are added to the water in amount less than that equivalent to the substances causing the hardness; suitable compounds are the sulphonated (polynuclear) aromatic or hydroaromatic hydrocarbons with an alkyl side-chain, or condensation products of these with aliphatic sulphonic acids or with a fatty oil. E. LEWKOWITSCH.

Dyeing and printing cellulose esters and ethers. F. FISCHER and C. E. MÜLLER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,142, 17.6.30. Appl., 20.4.27. Ger., 23.4.26).—See B.P. 269,934; B., 1928, 121.

Production of fast-coloured resists under aniline black. W. COTTON, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,658, 24.6.30. Appl., 28.1.26. Ger., 5.2.25).—See B.P. 247,211; B., 1927, 329.

Process in which cellulose acetate artificial silk and like products are treated with hot liquors. A. J. HALL, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,765,581, 24.6.30. Appl., 22.7.25. U.K., 31.7.24).—See B.P. 246,879; B., 1926, 317.

Treatment [weighting] of artificial silks. R. CLAVEL (U.S.P. 1,761,707, 3.6.30. Appl., 27.5.27. Ger., 17.9.26).—See B.P. 277,602; B., 1928, 154.

Protection of wool, rugs, and the like against the attack of moths. E. HARTMANN, M. HARDTMANN, and P. KUMMEL, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,819, 24.6.30. Appl., 26.11.26. Ger., 27.11.25).—See B.P. 303,092; B., 1929, 241.

Method and apparatus for applying colours to fibrous textile fabrics. P. RAUER (B.P. 330,935, 18.3.29).

Machines for washing and similarly treating fabrics with liquids. H. WHITEHEAD (B.P. 330,915, 15.2.29).

Dyes from naphthazarin (B.P. 327,860). Application of disazo dyes (B.P. 329,056).—See IV. Waterproof fabrics (B.P. 314,015).—See V. Materials for marking animals (B.P. 329,408).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Automatically controlling acid effluents. H. K. RICHARDSON (Chem. Met. Eng., 1930, 37, 293—295).—To neutralise effluents containing free sulphuric acid with limestone it is necessary to keep the acid concentration less than 0.3%, otherwise a coating of calcium sulphate formed on the stone prevents further action. An electrolytic conductivity recorder records the strength of the effluent and rings a warning bell if the concentration exceeds 0.8%. A second conductivity recorder operates a valve through which water is added to dilute the acidity of the effluent to 0.3%. This recorder graphs the acid concentration of the diluted effluent. A third recorder records the p_H of the diluted effluent after neutralisation by limestone. It also opens a valve admitting a solution of soda ash, should the desired neutrality not have been effected, as sometimes happens owing to abnormal conditions, e.g., when an excessively large volume of effluent has flowed through the limestone so rapidly that the reduced time of contact prevents the neutralisation from being completed.

D. K. MOORE.

Magnesium in commercial calcium citrate. F. PERCIABOSCO (Annali Chim. Appl., 1930, 20, 211—217).—All the samples of commercial calcium citrate examined contained appreciable proportions of magnesium, which occurs in the crude juice as the double salt, $MgCa_2(C_6H_5O_7)_2 \cdot 5H_2O$, and may amount to 0.9% (MgO) in the recovery liquid. To prevent this contamination of the recovered citrate with magnesium, treatment with either calcium chloride or hydrochloric acid is recommended.

T. H. POPE.

Nitrophosphates. G. CALCAGNI (Annali Chim. Appl., 1930, 20, 218—222).—The action of nitrogen dioxide on various phosphates moistened with water and kept shaken during the action yields fertilisers containing proportions of soluble phosphoric acid and nitrogen varying with the conditions employed. When treated in the cold (hot) with excess of the nitrogen dioxide, (1) tricalcium phosphate (41.89% P_2O_5), (2) apatite (40.22), (3) pebble phosphate (34.24), and (4) Kosseir phosphate gave fertilisers containing the following percentages of soluble P_2O_5 and N: (1) 41.69, 29.27 (31.82, 14.2); (2) 40.08, 33.37 (40.30, 27.04); (3) 33.96, 42.15 (33.87, 29.63); (4) 29.92, 46.84 (30.16, 32.65). These nitrophosphates, which are much less heavy than the corresponding amounts of superphosphate and nitrates with the same fertilising constituents, may be obtained with any required ratio between the proportions of soluble phosphoric acid and nitrogen, by using the calculated quantities of crude phosphate and nitrogen dioxide.

T. H. POPE.

Modernised silica grinding. R. L. CAWOOD (Chem. Met. Eng., 1930, 37, 304—305).—Sand obtained from sandstone by crushing, washing, and grinding is pulverised in a ball-mill lined with siliceous. This sand is unloaded from wagons and by means of an elevator and enclosed belt conveyor is discharged into a steel bin. From the bottom of this bin the sand is fed by a motor-operated feed device of variable capacity into a pit. A scoop on the mill lifts it from the pit and feeds it into the mill. A current of air carries the fines to an elevator, which lifts them to a centrifugal air separator, which removes the fines while the oversizes are returned to the mill. A screen fastened to the discharge end of the mill prevents balls and pebbles from passing. The process is continuous and automatic. By slight adjustment a product of varying fineness from 97% through 100-mesh to 97% through 300-mesh can be obtained. The power requirements of the various items of plant are given.

D. K. MOORE.

Solubility of sulphur. D. HENVILLE (Analyst, 1930, 55, 385).—Precipitated sulphur dissolves readily in carbon disulphide and slowly but completely in methylated spirit and petroleum spirit, whereas sublimed sulphur (the sample used) was only soluble to about 83% in each of these solvents. The sulphur samples extracted contained only traces of mineral matter. Sulphur ointment containing 10.2% S yielded 3.75% of residual sulphur after extracting 2 g. for 2 hrs. with petroleum spirit in a continuous extractor.

D. G. HEWER.

Autoclaves. HARRISON.—See I. Triple fertiliser. PESTOV and KALABEKOVA. Apatite and nepheline rock as fertiliser. SOMOV. Ground sulphur for dusting etc. STREETER and RANKIN.—See XVI.

PATENTS.

Manufacture [synthesis] of hydrogen chloride or hydrogen bromide or their corresponding acid solutions. RÖHM & HAAS A.-G. (B.P. 312,908, 7.5.29. Ger., 2.6.28).—Hydrogen and the halogen are passed with a velocity sufficiently high to prevent reaction of the gases or products with the metal through a burner constructed (preferably) of copper, or of iron, lead, or an alloy, and thence into a combustion chamber constructed (preferably) of lead and provided with means for cooling the combustion gases to about 80°.

L. A. COLES.

Dehydration of materials [caustic alkali]. J. F. WAIT, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,749,455, 4.3.30. Appl., 31.12.24).—Caustic alkali which has had its moisture reduced to 10% by steam and direct fire heat, is, while molten, subjected to an electric current between electrodes. Either alternating or direct current may be used, i.e., the final moisture may be removed by evaporation or electrolysis.

B. M. VENABLES.

Catalytic oxidation of [impure] ammonia. SELDEN Co., Asses. of A. O. JAEGER (B.P. 309,583, 26.3.29. U.S., 14.4.28).—Impure ammonia produced, e.g., in the distillation of coal tar is oxidised in one or more stages with the use as catalyst, during at least one stage, of diluted multi-component zeolites produced

as described in B.P. 279,466 (B., 1928, 603); *e.g.*, the air-ammonia mixture is passed at 400–450° over a zeolite catalyst to burn away phenolic and other impurities and then, after washing with alkali solutions etc. if necessary, over platinum gauze or an iron-aluminium multi-component zeolite at 650–800° to effect oxidation of the ammonia. L. A. COLES.

Manufacture of alkali hypochlorites from [artificial-silk] waste alkali lyes. I. G. FARBENIND. A.-G. (B.P. 305,931, 9.2.29. Ger., 11.2.28).—Chlorine is passed at about 50° into waste lye containing organic impurities, whereby organic matter is oxidised to carbon dioxide, and alkali hypochlorite solution remains.

H. ROYAL-DAWSON.

Manufacture of alkaline-earth and alkali formates and chromium-green. I. G. FARBENIND. A.-G. (B.P. 305,588, 6.2.29. Ger., 7.2.28).—A suspension or solution of an alkaline-earth or alkali chromate is reduced at 150–350° under increased pressure by carbon monoxide.

H. ROYAL-DAWSON.

Manufacture of acid calcium phosphate [for use in baking powders, self-raising flour, etc.]. R. HADDAN. From FEDERAL PHOSPHORUS CO. (B.P. 330,777, 20.6.29).—Milk of lime, *d* about 1.2, is added slowly with stirring to 75% phosphoric acid until the suspension has *d* 1.4 and contains 1–3% excess of lime, and the suspension is dried by spraying in warm air.

L. A. COLES.

Production of barium sulphide. KALI-CHEMIE A.-G., Assees. of RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 316,966, 26.6.29. Ger., 7.8.28).—The reduction of heavy spar, especially that with poor agglomerating properties, is facilitated by precalcination at, *e.g.*, 600–700°, or by disintegration to particles not exceeding 1 mm. in diameter, or by the addition of small quantities of mineral acids or alkali or alkaline-earth salts, or by a combination of two or more of these processes.

L. A. COLES.

Reduction of metallic compounds and production of arsenates. G. N. KIRSEBOM (B.P. 315,811, 18.7.29. U.S., 18.7.28).—Material containing reducible metal compounds, such as flue dust, antimony ore, cadmium dust, slime, caustic lead slag, etc., is heated with sodium hydroxide and arsenic trioxide; the metals or alloys collecting at the bottom of the melt are run off and the slag is treated with water to extract sodium arsenate and excess sodium hydroxide. The sodium arsenate is recovered by crystallisation and treated with milk of lime to precipitate calcium arsenate and to yield sodium hydroxide solution which, after filtration, is returned, together with the mother-liquor from the crystallisation, to the process. L. A. COLES.

Reduction of metal oxides, anhydrides, or metalloid oxides [to lower oxidation stages]. J. CHABANNAIS (F.P. 637,403, 11.7.27).—The pulverised oxide is treated at a suitable temperature in a container or autoclave under continuous agitation with a liquid or viscous reducing agent (*e.g.*, hydrocarbons or anhydrides of not too low b.p.). The oxide produced has an improved reactivity towards acidic or basic reagents.

S. K. TWEEDY.

Treatment of siliceous minerals. A. J. MOXHAM, Assr. to ELECTRO CO. (U.S.P. 1,748,989, 4.3.30. Appl., 25.11.24).—Siliceous material containing potassium, aluminium, and ferric iron, such as greensand, is digested with not too concentrated sulphuric acid, *e.g.*, in 100% excess. Part of the potassium and aluminium sulphates formed is allowed to crystallise out as potassium alum; the remainder of these sulphates, together with the ferric sulphate, is precipitated out in concentrated sulphuric acid. This precipitate may be utilised in iron or steel manufacture or as a pigment. The mother-liquor is used in subsequent digestions. S. K. TWEEDY.

Production of oxides of titanium. J. BLUMENFELD (B.P. 307,881, 11.3.29. Ger., 15.3.28).—A titanium halide (*e.g.*, tetrachloride) distributed upon a soluble salt, *e.g.*, an alkali chloride or sulphate, is treated with steam and the mass is calcined, cooled, and extracted with water to remove the salt; the product is obtained in a finely-divided form suitable for use as a pigment.

L. A. COLES.

Preparation of oxygen-evolving preparations suitable for use in respiratory apparatus and the like. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 305,101, 30.1.29. Ger., 30.1.28. Addn. to B.P. 280,554; B., 1928, 447).—An alkali peroxide, together with catalysts and inert substances is hydrated in cooled rotating drums with 9–23% of water introduced in an atomised form, or as vapour or snow. The hydrated material is formed into shaped pieces and is heated, before or after shaping, to increase its stability.

W. G. CAREY.

Contact sulphuric acid process. SELDEN Co., Assees. of A. O. JAEGER (B.P. 314,858, 27.5.29. U.S., 3.7.28).—See U.S.P. 1,741,310; B., 1930, 660.

Production of hydrocyanic acid. P. T. DOLLEY, Assr. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,761,433, 3.6.30. Appl., 19.10.27).—See B.P. 299,019; B., 1929, 850.

Preparation of vanadic acid. A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP., and J. A. BERTSCH (U.S.P. 1,765,870, 24.6.30. Appl., 15.5.26).—See B.P. 287,401; B., 1928, 366.

Manufacture of ammonia. C. URFER, Assr. to SOC. D'ETUDES MINIERES ET INDUSTRIELLES (U.S.P. 1,767,780, 24.6.30. Appl., 2.6.27. Fr., 8.1.25).—See B.P. 253,540; B., 1927, 218.

[Production of] stable ammonium bicarbonate. E. DEHNEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,705, 24.6.30. Appl., 8.6.25. Ger., 10.6.24).—See B.P. 244,645; B., 1926, 155.

Electrolytic manufacture of [per-]compounds containing active oxygen. O. NITZSCHKE and E. NOACK, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,722, 24.6.30. Appl., 4.6.27. Fr., 26.4.26).—See B.P. 290,750; B., 1928, 523.

Production of calcium nitrate, alumina, and phosphorus. R. GRIESSBACH, O. SCHLIEPKE, and O. HEUSLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,785, 24.6.30. Appl., 17.1.28. Ger., 24.1.27).—See B.P. 296,832; B., 1928, 815.

Manufacture of calcium molybdate. A. KISSOCK (U.S.P. 1,763,712, 17.6.30. Appl., 6.11.26).—See B.P. 280,240; B., 1929, 393.

Dissociation of sulphur vapour. P. C. H. SIEDLER and E. SCHULTE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,767,319, 24.6.30. Appl., 1.10.28. Ger., 12.10.27).—See B.P. 298,599; B., 1929, 128.

Manufacture of sulphuryl chloride. R. H. McKEE and C. M. SALLS (U.S.P. 1,765,688, 24.6.30. Appl., 26.6.23).—See Can.P. 251,586; B., 1926, 406.

Carbon dioxide ice apparatus, process, and product. A. P. THURSTON. From DRYCE EQUIPMENT CORP. (B.P. 331,077, 13.6.29).

Ammonia and hydrogen sulphide from gases (B.P. 316,278).—See II. **Electric furnace** (B.P. 303,798).—See XI. **Satin-white** (B.P. 327,848).—See XIII. **Fertiliser** (B.P. 309,175).—See XVI.

VIII.—GLASS; CERAMICS.

Glass-house refractories. Study of corrosion-resisting properties. J. H. PARTRIDGE and H. C. BIGGS (J. Soc. Glass Tech., 1930, 14, 63—75 T).—The results of an examination of seven fireclay blocks, two samples of sillimanite blocks, and one of cast mullite by Bowmaker's method (B., 1929, 815) were correlated with those observed in practice. It is shown that under standard conditions the test gives results of value in conjunction with accurate porosity determinations in estimating probable behaviour in service. High alumina content (other things being equal), porosity of less than 20%, fine close uniform texture, and high firing temperature gave good resistance. The test extended to pot-clay mixtures indicated corrosion-resistances less than those of good tank blocks. Small crucibles, made from various mixtures of clay with ball clay and grog and with addition of alumina, were used for founding at 1270—1300° successive charges of a glass having the composition: SiO₂ 57.8, PbO 26.55, Na₂O 5.3, K₂O 6.92%; the corrosion was determined by visual inspection. It is concluded that addition of alumina to a pot-clay mixture improves the corrosion-resistance, provided that the alumina combines with some of the free silica to form mullite. This did not always occur at temperatures as low as 1350°. M. PARKIN.

Chemical reactions in the melting of potassium borosilicate glass. M. A. BESBORODOV and L. M. SILBERFARB (J. Soc. Glass Tech., 1930, 14, 39—51 T).—Using quartz ground to pass 3000-mesh/cm.², potassium carbonate, and boric acid, all chemically pure, a glass having SiO₂ 63, B₂O₃ 25, K₂O 12% was investigated. The batch was ground in a ball-mill and heated in lots of 20—25 g. to temperatures ranging in 100° steps from 500° to 1200°. Reaction, as detected by loss in weight, water-soluble and residual silica, was considered to set in below 500°. The amount insoluble in water increased rapidly between 700° and 900°, and at lower temperatures potash appeared to form soluble borates or silicates, no insoluble potassium borosilicates being found below 700°. At 500° some insoluble borosilicates were formed, the amount being small below 700°, and became significant only when potash also entered into

the composition of the "insoluble," which was found to range from SiO₂ 99, B₂O₃ 1.15%, K₂O nil at 500° to SiO₂ 68.58, B₂O₃ 21.07, K₂O 10.25% at 1200°. At 900° the K₂O rose to a maximum of 15.3% with SiO₂ 73.3 and B₂O₃ 11.4%. M. PARKIN.

Choice of annealing schedule for commercial glassware. J. B. MURGATROYD (J. Soc. Glass Tech., 1930, 14, 3—12 T).—The final strain in the article was determined by the temperature gradient across the strain-free glass when it "set," and the greatest value, θ , of the temperature difference between inner and outer walls was given by $\theta = Bx/50NaY$, where x is the percentage of the breaking stress which could safely be permitted in the annealed article at room temperature, B the breaking stress in kg./mm.², Y the Young's modulus, a the coefficient of expansion for the glass at room temperature, and N the ratio of expansion coefficient above the critical temperature to a . The rate of loss of temperature allowable to yield ware possessing $x\%$ of breaking stress at room temperature is $BKx/50NaYdSL^2$ degrees per sec., where d is the density in the annealing range, S the sp. heat, K the coefficient of thermal conductivity, and L the thickness of the glass wall in cm. Taking average values for a soda-lime-silica glass, the author arrived at a cooling rate of 1.3°/L² per min. to leave 5% of the breaking stress. For ware 1 cm. in thickness the annealing schedule was then 15 min. for ware to become uniform and strain-free at the annealing temperature, 11 min. to cool at the rate of 5°/min. through the annealing range (given as 56°), and 35 min. cooling at 10—15°/min., i.e., 61 min. in all. Thicker ware would take longer. Ware with up to 20% of the breaking stress present did not show any lessening of resistance to mechanical shock, whilst it withstood thermal shock better. If this figure were substituted for x in the expressions given, the rate of annealing would become 5.2°/L² per min. At 500° the loss of heat by radiation, ignoring the departure from black-body conditions, from glassware in a lehr was 0.013 g.-cal./sec., whilst that by conduction from one face to the other was 0.01 g.-cal./sec.

M. PARKIN.

Nature of minerals in artificial melts of 2CaO.SiO₂, of 3CaO.SiO₂, and of 8CaO.2SiO₂.Al₂O₃. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 161 B).—The solids deposited from melts of the above composition have been examined microscopically. In all three mixtures strongly birefringent, crystalline needles were obtained, most abundantly from the melt 2CaO.SiO₂, which probably have this composition. Crystals corresponding with alite from Portland cement clinker were obtained only from the melt 3CaO.SiO₂. Crystals of lime have also been observed in some of the mixtures. O. J. WALKER.

Velocity of crystallisation of soda-lime-silica glasses. K. TABATA (J. Soc. Chem. Ind., Japan, 1930, 33, 95—96 B).—The work of Zschimmer and Dietzel (B., 1926, 877; 1929, 472) is criticised. The crystallisation of silica may be observed during surface devitrification accompanied by surface contraction, and the importance of the latter phenomenon is emphasised.

H. F. GILLBE.

Beryllium glass. II. Potassium-beryllium series. C. F. LAI and A. SILVERMAN (J. Amer. Ceram. Soc., 1930, 13, 393—398; cf. B., 1928, 894).—A preliminary investigation of the properties of beryllium glasses is described, and results are given in the form of triaxial diagrams for K_2O - BeO - SiO_2 and Na_2O - BeO - SiO_2 . The region of high beryllia and low silica indicates better glasses than that of low beryllia and high silica. Density determinations result in lower values for potash than for soda glasses. Refractive index, composition, and properties of these glasses are also tabulated.

R. J. CARTLIDGE.

Determination of boric oxide in glass. V. DIMBLEBY (J. Soc. Glass Tech., 1930, 14, 51—60 r).—In tests on the methods of Wherry and Chapin (B., 1908, 1179) and of Sullivan and Taylor (B., 1914, 1154), the author found that good-quality resistance-glass flasks were superior, in general, to those of other types, even though containing boric oxide. After ageing, the amount of attack was practically constant. The amount of calcium carbonate in excess and the time of boiling caused variations in blank titration of the order of 0.65 c.c. of the sulphuric acid used. Serious loss of boric oxide could occur if the precipitate obtained with calcium carbonate were not dissolved in dilute hydrochloric acid and reprecipitated by the reagent, and use of more than 0.3 c.c. of phenolphthalein as indicator caused low values. Careful control of these points enabled good agreement to be obtained by independent analysts. Detailed analytical procedures embodying these findings are given (a) for simple soda-lime-boric oxide-silica glasses and (b) for those rich in boric oxide or containing lead or zinc.

M. PARKIN.

Thermal expansion of glass. I. General form of the expansion curve. II. Glasses of the series sodium metasilicate-silica. W. E. S. TURNER and F. WINKS (J. Soc. Glass Tech., 1930, 14, 84—109 r, 110—126 r).—I. Contrary to expansion curves described by other workers up to 1928, this curve was not substantially linear up to the critical or transformation temperature, C_t , but, as shown also by Klemm and Berger, possessed inflexion points between 0° and C_t . These in both silicate and borosilicate glasses occurred at temperatures closely alike, in spite of considerable differences in composition, viz., at 120 — 150° , at 250° , and a third, where it existed, at 350° . Certain glasses did not show these points, and two were encountered that gave practically linear curves right up to the upper annealing temperature A_t . Annealing influenced the form of the curve between A_t and C_t , the deviation from the straight portion occurring at a lower temperature on strained specimens. The increase in expansion in the range C_t - A_t was most marked in the case of glasses rich in boric and iron oxides.

II. Fifteen glasses of the system sodium metasilicate-silica, ranging from 51.15 to 91.6% SiO_2 , were prepared by melting in a platinum crucible in an electric furnace, precautions being taken to obtain homogeneous glass from which rods were drawn to give test pieces 10 cm. \times 0.5 cm. The glass with 51.15% SiO_2 gave an expansion curve in which the normal critical range was entirely absent, and was not induced

by re-annealing. Glasses with 51.15—85.04% SiO_2 behaved normally, whilst those richest in silica gave indications of the C_t point, but no A_t point, as shown by the bending over of the curve even up to 650° , the optically determined annealing point being much below this. The annealing temperatures rose from 440° for the glass having 51.15% SiO_2 to 530° for that with 85.04% SiO_2 , whilst the temperatures of the change points t_1 , t_2 , and t_3 (cf. preceding abstract) were very constant at 120 — 140° , 240 — 250° , and 350° , respectively. The C_t value rose with silica content up to 455° for 72.8% SiO_2 and then remained practically constant; A_t rose similarly, but irregularly, till 85% SiO_2 was reached, after which the rise was more rapid. The new measurements over the range 0 — 130° substantially confirmed those of English and Turner, and differed notably from those of Samsoen. The relationship between chemical composition and thermal expansion was strictly linear for all ranges of temperature below C_t for glasses with 60—85% SiO_2 and probably linear with a different slope for those with 85—100% SiO_2 .

M. PARKIN.

Sp. heat of pyrex glass from 25° to 175° . T. DE VRIES (Ind. Eng. Chem., 1930, 22, 617—618).—A thermocouple and a nichrome heating element were fused into a piece of pyrex glass silvered over and suspended in a highly evacuated container surrounded by an oil-bath. The energy input and rise of temperature for 3-min. periods of heating and for various initial temperatures were determined. The values found were in close agreement with the equation: sp. heat = $0.1839 + 0.0003134(t - 20^\circ)$.

C. IRWIN.

[Strength of glass containing cracks.] F. W. PRESTON (J. Soc. Glass Tech., 1930, 14, 37—38 r).—In a communicated discussion of Milligan's observations (B., 1930, 557) the present author suggests that Milligan's polariscope figures reveal "bearing stresses," and that hydrofluoric acid would probably notably increase the strength.

M. PARKIN.

Development of acid-resisting, white, sheet-steel enamels. A. I. ANDREWS (J. Amer. Ceram. Soc., 1930, 13, 411—426).—The acid-resistance of enamels has been studied with a view to control such variables as composition, smelting, milling, and firing. Simple acid-resisting enamels were made from the series Na_2O - B_2O_3 - SiO_2 and Na_2O - PbO - SiO_2 . The results of acid-resistance as shown by the spot test check well with those of the grain test. The data given show that silica and titania both improve the acid-resistance, whilst fluxes, unless properly proportioned, tend to decrease this property. Fineness of grinding and smelting conditions make no difference to acid-resistance, but increase of firing temperature increases the acid-resistance of most enamels. Addition of titania to an enamel does not increase the refractoriness as much as does silica.

R. J. CARTLIDGE.

Clays from Shdanka (Sysran-Viasma Rly.). V. PERMYAKOV (Trans. Ceram. Res. Inst., Russia, 1929, No. 17, 4—31).—The composition and ceramic properties of the grey or brown coarse-grained clays are recorded.

CHEMICAL ABSTRACTS.

Examination of china clay [for grit]. R. G. THIN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10,

388—393).—An elutriation method for the determination of grit in china clay is preferred to sedimentation methods. A modification of Schöne's apparatus is described. Higher percentages of grit are obtained by this method than by Strachan's method (unpublished), but it is pointed out that appreciable variation in results obtain if standard experimental conditions are not adhered to. A brief note on the moisture content of clays is given.
T. T. POTTS.

[Examination of china clay for grit.] J. STRACHAN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 395—397; cf. preceding abstract).—Thin's method is criticised, and it is held that a sedimentation method alone can correctly determine grit in a clay, elutriation serving only for grading the true clay according to particle size. The Schöne method is held to take too small a sample, to require too long a time, to be subject to disturbing convection currents in the elutriating tube, and to be difficult to wash out. The volume, temperature, and p_H of the water require careful control. By a sedimentation method it is possible to remove quantitatively all grit over 0.05 mm. in 1—1½ hrs. Mineralogical analysis of the grit should supplement the quantitative determination.
T. T. POTTS.

Technical analysis of clays with the aid of the microscope. H. MÖHL (Sprechsaal, 1929, 62, 731—734; Chem. Zentr., 1930, i, 885).—The methods employed are described, together with the microscopical characteristics of various clays.
A. A. ELDRIDGE.

Solubility of feldspars in water. C. W. PARMELEE and A. J. MONACK (J. Amer. Ceram. Soc., 1930, 13, 386—392).—Data are presented showing the relation between the rate of solubility and the time, using different feldspars. The alkalinity or solubility of the feldspar was measured by colorimetric determinations of the p_H values. Equations are given merely as indications of the true relations. The results obtained show that no definite relation exists between the ratio K_2O/Na_2O and the solubility of feldspar in water. High-soda and high-potash feldspars are stated to have equal solubilities, whilst intermediate feldspars possibly have the highest solubilities. The data also indicate that there may be sufficient increase in alkalinity to affect the viscosity of casting slips.
R. J. CARTLIDGE.

PATENTS.

Manufacture of coloured glassware. T. DAVIDSON (B.P. 329,022, 11.2.29).—Variegated, cloud, or streak effects are obtained in glass by the appropriate inter-fusion of two or more glasses of different shades or colours; the finished articles are then given a backing or surface coating of coloured enamel or paint.

H. ROYAL-DAWSON.

Manufacture of compound sheets of glass and cellulose derivative composition. ACETEX SAFETY GLASS, LTD., and W. JOHNSTON (B.P. 330,265, 4.3.29).—Two sheets of glass with an interposed sheet of, e.g., cellulose acetate, are cemented together by treating the sheet of cellulose acetate with a solvent (triacetin) of high b.p., pressing the sheets together by means of compressed air, and finally subjecting them to steam

under pressure and at a temperature below the b.p. of the solvent.
C. A. KING.

Enamelling of glass articles. M. JEANDIDIER (F.P. 633,277, 25.4.27).—Directly after or during moulding the articles are dusted over with an enamel which has a coefficient of expansion differing from that of the glass constituting the articles. The enamel fuses and on cooling adheres to the glass, to give a crazed effect.
S. K. TWEEDY.

Drying of ceramic products. J. KOTTERBA (G.P. 461,077, 1.8.25).—The articles are placed in chambers with perforated bottoms through which the hot drying air is introduced under pressure and the wet air is withdrawn. The hot air rising in the chambers is thus moistened by direct contact with the gravitating moist air leaving the same, so that the articles are not brought into contact with hot dry air, whereby drying of merely the outer layer, with consequent non-removal of the interior moisture, is prevented. The dried articles are free from cracks.
S. K. TWEEDY.

Drying of refractory materials. W. J. GARDNER (B.P. 330,447, 29.7.29).—A longitudinal drying chamber, through which articles of refractory material are conveyed on trucks, is divided into several zones of increasing temperature by means of baffle walls. Fans are provided in each zone to cause a turbulent motion of the atmosphere and ports are provided for renewing the air supply.
C. A. KING.

Terra-cotta firing system. A. H. SHEFFIELD (U.S.P. 1,749,988, 11.3.30. Appl., 15.4.27).—The interior of the kiln is provided with an inner wall which extends upwards for about a third of the height of the kiln and is spaced from the wall of the latter. The flames from gas jets projecting through apertures in the outer wall play upon the inner wall and pass up the space between the walls. The inner wall may also be mounted upon a separate base which is spaced from the true bottom of the furnace. The gas flames then pass under the base as well and up a flue provided in the middle of the latter.
S. K. TWEEDY.

Tunnel kiln. P. A. MEEHAN, Assr. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,764,460, 17.6.30. Appl., 3.4.28).—See B.P. 312,786; B., 1929, 645.

Forming abrasive [surfaces on] articles [by spraying]. E. GARNER, and CARBORUNDUM Co., LTD. (B.P. 330,322, 16.3.29).

[Segmental] grinding or abrading wheels. NORTON Co. (B.P. 308,991, 2.4.29. U.S., 2.4.28).

Light filter (U.S.P. 1,751,220).—See I. Nozzles for artificial silk (B.P. 314,381).—See V. Electric furnace for glass (B.P. 303,798). Tunnel kiln (U.S.P. 1,749,762). Insulating material (B.P. 330,268).—See XI.

IX.—BUILDING MATERIALS.

Mixed Portland cements. VI. S. NAGAI (J. Soc. Chem., Ind., Japan, 1930, 33, 82—84 B; cf. B., 1930, 284).—The strength of Portland cement mixtures containing various proportions of ground clinker and spent shale is found to be considerably greater than that of ordinary Portland cement.
H. F. GILLBE.

Draught, temperature, and combustion conditions in commercial kilns burning building brick. H. P. KIMBELL (Univ. Toronto Fac. Appl. Sci. Eng., Eng. Res., Bull. No. 8, 1928, 1—34).—Temperatures, draughts, and gas analyses of the burning of four different charges of bricks in four kilns of two different types are given, and calculations are made both of the efficiency of burning and also of the principal loss, viz., the heat in the products of combustion. Of the useful heat, the sensible heat in the hot bricks is by far the largest item, being usually more than ten times that required to drive off moisture. The efficiency of burning appears to be 20—25%, though since the sp. heat of the material is not known with any accuracy this figure is in doubt. There are too many variables for any conclusions to be drawn. B. M. VENABLES.

High iron oxide Portland cement. II. S. NAGAI and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 85—87 B; cf. B., 1930, 462).—Further data are given relative to the chemical composition, density, and compressive strengths of Portland cements rich in iron.

H. F. GILLBE.

High iron oxide Portland cements. III. S. NAGAI and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 137—140 B; cf. preceding abstract).—The compressive strengths and other physical properties of some 1:3 high iron oxide Portland cements (made from "Colloidal Jodo," a Japanese natural product containing soluble silica and alumina) were compared with the properties of similar cements prepared from pure chemicals.

S. K. TWEEDY.

Calcium ferrites and iron cements. I. S. NAGAI and K. ASAKA (J. Soc. Chem. Ind., Japan, 1930, 33, 130—133 B).—The ferrite of highest lime percentage in clinker of high lime content is $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$ (cf. Sosman and Mervin, B., 1916, 1108). This ferrite is readily obtained by heating a mixture of 5 pts. of quicklime and 1 pt. of ferric oxide above 1200° ; it is soluble in 0.5*N*-hydrochloric acid, whereas monocalcium ferrite is insoluble.

S. K. TWEEDY.

Calcium ferrites and iron cements. II. S. NAGAI and K. ASAKA (J. Soc. Chem. Ind., Japan, 1930, 33, 161—164 B; cf. preceding abstract).—Dicalcium ferrite, $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$, has been obtained by extracting the free lime from heated mixtures of lime and ferric oxide by means of a glycerin-alcoholic solution of ammonium acetate. By heating an equimolecular mixture of the two oxides between 1000° and 1200° , monocalcium ferrite, $\text{CaO} \cdot \text{Fe}_2\text{O}_3$, was obtained. The two ferrites can be separated from lime-ferric oxide mixtures by treating firstly with 0.4*N*-hydrochloric acid and then with 0.8—2.0*N*-acid, which dissolves the di- and mono-ferrite, respectively.

O. J. WALKER.

Hydration of Portland cement. Influence of monocalcium phosphate [? gypsum]. I—V. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 147—153 B).—The hydration of efficiently burnt Portland cement always commences with the formation of needles and plates of calcium aluminate, which brings about the binding of the cement; in the course of time the needles are completely converted into plates. When gypsum is

added to the cement calcium sulphoaluminate, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2.5\text{CaSO}_4 \cdot 30\text{H}_2\text{O}$ is formed instead; this compound prolongs the binding time. The hardening process depends on the formation of a gel mass of calcium hydrosilicate which occurs much later. Finally, large hexagonal crystals of hydrated lime are deposited. The hydration of inferiorly burned cement commences with calcium hydrosilicate gel formation; both the binding and hardening depend on the formation of this compound. Only when the water addition exceeds a certain limit, which is higher the greater is the proportion of free lime, are the short, thick needles of calcium aluminate produced. The large hexagonal plates of hydrated lime separate out earlier than in the case of properly burned cement; the separation is earlier the greater is the quantity of free lime present. Very much later (e.g., after several weeks) there is a slight formation of well-defined hexagonal plates of calcium aluminate, which promotes the hardening process. No evidence was found for the formation of needles of calcium silicate during the hydration of cement. The optical properties of the calcium sulphoaluminate crystals are briefly mentioned; in order to obtain these crystals, properly burned clinker, containing not more than 0.4% of free lime and as free as possible from sulphuric acid and sulphides, must be treated for 8—14 hrs. with 30 times its weight of water. After filtration and addition of saturated gypsum solution to the filtrate, the mixture is allowed to crystallise.

S. K. TWEEDY.

Lime-alumina cement. I. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 167—169 B).—The compositions and compressive strengths of lime-alumina cements containing about 30% Al_2O_3 and 50% CaO have been determined.

O. J. WALKER.

Small-piece testing on strength of cement mortars. IV. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 87—89 B; cf. B., 1930, 462).—Further data are given of variations of the compressive strength produced by varying the method of curing. For mixed Portland cements, e.g., cements containing blast-furnace slag, the strength resulting from curing for 1 day in moist air, 6 days in water, and 21 days in air is lower than that attained by curing for 1 day in moist air and 27 days in water. Cements rich in alumina, e.g., "ciment fondu" and lumnite cement, diminish in strength if cured in water for long periods, on account of the dissolution of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$.

H. F. GILLBE.

Hydration of calcined gypsum. W. C. HANSEN (Ind. Eng. Chem., 1930, 22, 611—613).—Earlier theories of the acceleration or retarding of the setting of calcined gypsum by salts in solution are criticised. The progress of the reaction was recorded by means of time-temperature curves. It was found that neither the solubility of gypsum in the salt solution, nor the action of the positive ion in forming an adsorption complex has anything to do with the matter. Results also show that normally the gypsum does not precipitate so fast as the hemihydrate dissolves. By seeding the mixture with newly formed gypsum nuclei the setting rate was increased. It was found that ammonium acetate and citrate, which retard the setting, also

retard the precipitation of gypsum from supersaturated solutions. Gypsum pastes examined microscopically showed the formation of crystals from the solution and not around the particles of hemihydrate. There is no evidence of gel foundation or colloidal action. It is simply a case of dissolution and precipitation, which latter may be accelerated or retarded. C. IRWIN.

Synthesis of calcium aluminates and their hydration. V. S. NAGAI and R. NAITO (J. Soc. Chem. Ind., Japan, 1930, 33, 133—137 B).—The aluminates $3\text{CaO}, 5\text{Al}_2\text{O}_3$ and $3\text{CaO}, \text{Al}_2\text{O}_3$ were prepared from the oxides; the latter aluminate always contains free lime. Comparative strength tests (cf. B., 1930, 241) showed that $\text{CaO}, \text{Al}_2\text{O}_3$ has the highest initial strength, but $3\text{CaO}, 5\text{Al}_2\text{O}_3$, having a lower initial strength, overtakes it after seven days. The other aluminates, $5\text{CaO}, 3\text{Al}_2\text{O}_3$ and $3\text{CaO}, \text{Al}_2\text{O}_3$, have lower initial strengths, which increase slightly with time. Solubility tests on the four aluminates showed that $3\text{CaO}, \text{Al}_2\text{O}_3$ and $5\text{CaO}, 3\text{Al}_2\text{O}_3$ form the hydrate $3\text{CaO}, \text{Al}_2\text{O}_3, n\text{H}_2\text{O}$. S. K. TWEEDY.

Synthesis of calcium aluminates and their hydration. VI. S. NAGAI and R. NAITO (J. Soc. Chem. Ind., Japan, 1930, 33, 164—166 B).—The effect of heating eutectic mixtures of lime and alumina has been studied. The supposed aluminate $2\text{CaO}, \text{Al}_2\text{O}_3$ is now believed to be a mixture of $3\text{CaO}, \text{Al}_2\text{O}_3$ and $5\text{CaO}, 3\text{Al}_2\text{O}_3$. The compressive strengths of the products obtained by heating the eutectic mixtures have been measured. O. J. WALKER.

Preparation of a new road binder. C. F. BROADHEAD (Gas World, 1930, 92, 621—627).—Tar obtained by carbonisation of Australian coal in vertical retorts was found unsuitable for roadmaking, even after distillation or blending. Polymerisation of unsaturated components of the tar could, however, be achieved by heating it with small amounts of formalin and aqueous ammonia in the presence of air. The properties of the substance produced are similar to those of bitumen, and it has been successfully used for road dressing. A plant for the preparation of one million gallons per year is to be constructed, and particulars are given of its design and operation.

R. H. GRIFFITH.

Colour lakes. CLAYTON.—See XIII.

PATENTS.

Hydraulic binding agent. S. L. A. ODÉN, D. R. E. WERNER, and S. GIERTZ-HEDSTRÖM (F.P. 633,197, 29.3.27).—For the purpose of regulating the binding time, cements, gypsum, hydraulic lines, etc., preferably after winnowing into portions of various granule sizes, are treated with gaseous media (sulphur dioxide or trioxide, hydrogen chloride or fluoride, aluminium chloride, carbon dioxide, etc.) which form difficultly soluble, but hydrolysable, compounds on the surface of the granules of the binding agent. The gaseous media may be used for effecting the winnowing or may be admixed with the winnowing gas. Cement may be produced from the products by mixing them with alloys of aluminium (with iron, zinc, magnesium, etc.) which evolve heat, and sometimes gases, when

treated with water. Calcium carbide, which may be impregnated with organic or inorganic substances, may also be added with the alloys. S. K. TWEEDY.

Production of aluminiferous cement. E. MOYAT (G.P. 457,463, 2.2.27).—The raw materials (essentially bauxite and lime) are heated separately to any desired temperature and then passed to a large common chamber where they fuse in consequence of mutual interaction. Slag formation is avoided.

S. K. TWEEDY.

Cement mixture of high mechanical strength. SOC. COMMERCIALE DES CEMENTS DE LA CROISSETTE SOC. ANON. (Swiss P. 124,715, 18.12.26).—The finely-ground mixture contains 30—70% of one or more cements (Portland or aluminiferous cement), 20—30% of a natural silicate which crystallises well (e.g., feldspar), together with at least one mineral phosphate (apatite), and, if desired, 10—40% of a filler such as sand or marble waste.

S. K. TWEEDY.

Manufacture of porous [cementitious] products. J. A. RICE, Assr. to BUBBLESTONE CO. (U.S.P. 1,749,508, 4.3.30. Appl., 29.5.24).—A honeycombed mass of hard cementitious material is produced by mixing granules of a jelly-like material, preferably containing a large percentage of readily volatile liquid such as water, with any suitable mortar or plastic mixture capable of setting either during or subsequent to the preparation of the latter, and allowing the mixture to harden in the usual manner. A suitable jelly material is obtained by hardening glue with formalin or chromium compounds, but agar-agar, cellulose derivatives, etc. may also be employed.

S. K. TWEEDY.

Production of cementitious material. G. O. CASE, E. M. ELLIS, and L. H. MONTIGUE (B.P. 330,278, 1.3.29).—For use as a plaster or mortar a mixture of 10—25% of calcium (or magnesium) oxide and 90—75% of raw calcium (or magnesium) carbonate is ground together to the degree of fineness of Portland cement. The mixture may be used neat or mixed with sand.

C. A. KING.

Artificial stone compositions, cements, and the like. J. H. THOMPSON (Austral.P. 6171, 28.2.27).—Magnesium hydroxide or oxide is mixed with a solution of chloride of magnesium, iron (ferrous or ferric), zinc, or ammonium (which may contain magnesium nitrate or sulphate or other metal salts), or else is mixed with dehydrated magnesium chloride and then moistened with water, and then mineral or inorganic material, which is inert towards the magnesium or other salts and chosen according to the kind of stone it is desired to imitate, is incorporated with the mixture (e.g., sand, pumice, asbestos, kaolin). Colouring matter may be added, as well as metal oxides or filings. When the material is to be moulded to solid articles, solid vegetable material (timber or cane) may be added for reinforcing purposes.

S. K. TWEEDY.

Production of well-adhering coatings on asbestos-cement-slate plates. HAWENTA-PLATTEN-GES.M.B.H., and A. FRICKE (B.P. 327,871, 24.4.29).—The plates are superficially ground, heated if desired, and then wire-brushed, before being coated with paint etc.

S. S. WOOLF.

Making of roads, pavings, foundations, etc. BERRY, WIGGINS & Co., LTD., and H. H. HOLMES (B.P. 330,072, 22.4.29).—Stone or road metal which is damp or dirty is rendered suitable for coating with bitumen by an initial treatment in a mixer with a small quantity of cold creosote before adding heated bitumen thereto.

W. G. CAREY.

Materials for use in construction of roads, roofs, etc. SOUTH METROPOLITAN GAS Co., H. PICKARD, and C. M. C. HUGHES (B.P. 330,440, 23.7.29).—A dispersion of fine coal in tar is prepared at 250–300°, the proportions being such that the mixture has m.p. 97–103° (ring-and-ball) and a penetration of 12–20 at 25°. A heated mixture of 60% of stone ($\frac{1}{4}$ in. to dust), 10% of peat moss, coconut fibre, or similar fibrous material, and 30% of the bituminous binder may be used for roadmaking or may be pressed into blocks.

C. A. KING.

Covering roads and tiles and manufacturing tiles, plates, blocks, and the like. N.V. MIJNBOWEN CULTUURMAATS. "BOETON" (B.P. 310,922, 8.3.29. Holl., 3.5.28).—A mixture prepared at the ordinary temperature of a filler, solid bitumen, a bitumen solvent (e.g., carbon disulphide), and water is applied, after heating, if desired, at not above 100°, to road surfaces, concrete tiles, etc.; the water is then expelled and the mass bound by rolling or compression. (Cf. B.P. 310,923; B., 1930, 666.)

L. A. COLES.

Coating wood and other materials with a hard covering. J. A. GUSTAFSSON (B.P. 330,736, 17.5.29. Swed., 8.3.29).—The wood etc. is successively provided with a priming layer of thin and a grounding layer of thicker cement insoluble in water, preferably casein-lime cement, strewn with fairly fine sand, emery, or glass powder, and coated with an emulsion comprising a drying oil, an aqueous paste or glue solution, and finely-pulverised quartz, emery, or glass and, if desired, pigments; further coatings of nitrocellulose lacquers and decorative material may also be applied.

L. A. COLES.

Manufacture of a cement or plaster from gypsum. F. MULLIGAN (U.S.P. 1,766,448, 24.6.30. Appl., 20.2.25. U.K., 28.5.24).—See B.P. 238,949; B., 1925, 851.

Manufacture of cellular building materials. L. DESMARQUEST (U.S.P. 1,761,108, 3.6.30. Appl., 16.4.29. U.K., 18.4.28).—See B.P. 312,764; B., 1929, 599.

Pavement. L. KIRSCHBRAUN, Assr. to FLINTKOTE Co. (U.S.P. 1,767,533, 24.6.30. Appl., 8.10.28).—See B.P. 322,835; B., 1930, 192.

Gypsum fireproofing blocks. F. S. BRIDGES (B.P. 331,001, 3.4.29).

Construction of roads or pavements. UNAPHALT (ROADS), LTD., and H. E. G. RICHARDS (B.P. 330,271, 4.3.29. Addn. to B.P. 316,108).

[Photogravure process for] reproduction of the natural appearance of articles [e.g., wood] on other surfaces. OXFORD VARNISH CORP., Assecs. of L. V. CASTO (B.P. 308,303, 27.2.29. U.S., 21.3.28).

[Linoleum] floor coverings. ARMSTRONG CORK Co., Assecs. of S. H. HARTMAN (B.P. 317,004, 15.7.29. U.S., 8.8.28).

Surfacing for concrete floors [with linoleum etc.]. F. B. DEHN. From PARAFFINE COMPANIES, INC. (B.P. 330,951, 14.2.29).

Determining moisture in aggregate (U.S.P. 1,749,606).—See I. **Treatment of water-absorbent materials** (B.P. 326,940).—See VI. **Electric furnace** (B.P. 303,798).—See XI. **Freezing of soils** (B.P. 308,310).—See XVI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Substitution of limestone by sodium chloride in fluxes of blast-furnace smelting. E. V. BRITZKE, A. N. KRESTOVNIKOV, and I. B. CHMANENKOV (Min. Suir. Tsvet. Met., 1929, 359–375).—In experiments in a laboratory electric furnace the most favourable composition of the slag was: SiO_2 42.72, FeO 2.25, Al_2O_3 15.46, CaO 33.76, MgO 2.56, Na_2O 50.4. Substitution of 10.67% CaO by 5.04% Na_2O lowers the m.p. of the slag by 300°. Sulphur combines with the slag according to the equation: $\text{Na}_2\text{CO}_3 + \text{FeS} = \text{Na}_2\text{S} + \text{FeO} + \text{CO}_2$; $\text{Na}_2\text{S} + 2\text{CaO} \cdot \text{SiO}_2 = \text{CaS} + \text{Na}_2\text{O} \cdot \text{SiO}_2 + \text{CaO} \cdot \text{SiO}_2$.

CHEMICAL ABSTRACTS.

Preservation of highly-polished surfaces [of steel]. A. C. HANSON (Ind. Eng. Chem., 1930, 22, 603–604).—Experiments in which samples of steel plate were stored for a period in atmospheres of varying humidity showed that rusting commences when the temperature falls below the dew point. However high the humidity may be, there is no rusting unless condensation occurs.

C. IRWIN.

Protective coatings for iron and steel. I. Metallic coatings. H. N. BASSETT (Ind. Chemist, 1930, 6, 241–243).—The processes of galvanising, metal spraying, and tin plating are shortly described. Calorisation is the formation of a surface alloy, Al_3Fe , with a layer of solid solution of aluminium in iron below. The Meker process, which produces solid solution with practically no Al_3Fe , gives more satisfactory results. The articles to be coated are heat-treated with powdered Al_3Fe with about 0.7% of aluminium chloride. The life of steel so treated is increased tenfold at 1100°. Other processes for the protection of iron and steel at high temperatures produce an artificial coating of triferrous tetroxide, of ferric phosphate, or of nitride. The Coslett (phosphate) method is largely used in the cycle industry.

C. IRWIN.

Determination of iron oxide in liquid steel. C. H. HERTY, JUN., J. M. GAINES, JUN., H. FREEMAN, and M. W. LIGHTNER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 311, 3–13).—Aluminium is added to the molten steel, in which alumina is then determined. The alumina tends to segregate in the small ingots.

CHEMICAL ABSTRACTS.

Electrolytic determination of non-metallic inclusions in steel. C. H. HERTY, JUN., G. R. FRITTERER, and W. E. MARSHALL, JUN. (Min. Met. Invest., U.S.A.,

Co-op. Bull., 1929, No. 44, 25 pp.).—None of the aqueous extraction methods hitherto proposed completely removes inclusions containing manganous oxide. Inclusions extracted by electrolytic means are likely to be contaminated by atmospheric oxidation, liberation of hydrogen, metallic iron, and porous cup material.

CHEMICAL ABSTRACTS.

Loss of silica in the determination of silicon in technical iron. V. LINDT (Chem.-Ztg., 1930, 54, 327—328).—Six types of iron of high, medium, and low silicon content, principally such materials as are used for transformer stampings, have been analysed by four different methods. The results showed that the loss of silica was proportional to the amount and the concentration of the hydrochloric acid used for the dissolution of the iron and for washing the precipitates. The best results were obtained by dissolving the iron in nitric acid, evaporating down with sufficient concentrated sulphuric acid to leave an excess after $\frac{1}{2}$ hr., cooling, taking up with water, boiling, filtering, and washing the residue with water very slightly acidified with hydrochloric acid. Repeated evaporation of the silica with concentrated hydrochloric acid to render the precipitate more easily filterable was found undesirable and unnecessary. The brisk heating with sulphuric acid decomposes the resistant carbides, leaving pure silica.

H. J. DOWDEN.

Physical properties of the iron-nickel-chromium system. W. A. DEAN (Rensselaer Poly. Inst., Eng. & Sci. Series, 1930, No. 26, 29—55).—The resistance to oxidation at 1000°, sp. resistance, crystal structure, hardness, and magnetostriction have been investigated. Iron-chromium alloys containing 20—40% Cr with varying amounts (0—55%) of nickel resist corrosion as well as the more expensive commercial alloys. A 50:50 nickel-chromium alloy had the maximum resistivity, and a 20:60:20 iron-nickel-chromium alloy the minimum temperature coefficient ($0.65 \times 10^{-1}/^{\circ}\text{C}.$). The predominant lattice is face-centred, but in some regions a body-centred lattice is found, and in others the two co-exist. Hardness increases as chromium is substituted for iron and nickel, reaching a maximum in alloys containing both lattices. The 80:5:15 iron-nickel-chromium alloy has a low coefficient of expansion, as have also the invar alloys. Magnetostriction is confined to iron-chromium and iron-nickel alloys, and to adjacent ternary alloys containing not more than 10% of the third component.

C. W. GIBBY.

Determination of the hardness-resistance to bending diagram of grey cast iron. J. NAVARRO (Anal. Fis. Quím., 1930, 28, 501—506).—The relation between the Brinell hardness and the resistance to bending (as measured on a Frémont machine) of grey cast iron can be expressed by the straight line $y = 3.23x + 42$, where y is the hardness and x the coefficient of bending resistance, i.e., the transverse breaking stress in kg./mm.² The irons used in the tests contained 1.88—4.35% Si, 0.03—0.17% S, 0.05—0.47% P, and 2.92—3.58% C, and their microstructure resembled that of a mild steel with graphite inclusions. The hardness of the cast irons in all cases was about 30 Brinell numbers lower than that of the

steel having a similar structure to that of the ground mass of the cast iron.

A. R. POWELL.

Method and apparatus giving the extension coefficient and breaking load of metallurgical products in thin sheets. C. JOVIGNOT (Compt. rend., 1930, 190, 1299—1302).—The test-piece (thickness e) is pressed tightly between two circular jaws, and a measured hydraulic pressure is applied through a reservoir below it till rupture of a leather sphere (radius r) occurs. Both the pressure required (P) and the deformation (f) of the metal are recorded, and $x = f^2 \div$ constant gives the coefficient of deformation and $Pr/2e$ the breaking load. The nature of the break furnishes an indication of the homogeneity of the test-piece.

J. GRANT.

Electrolytic refining of copper, using complex salts of cuprous chloride. VI. N. KAMEYAMA and K. ONODA (J. Soc. Chem. Ind., Japan, 1930, 33, 101—102 B; cf. B., 1930, 331, 195).—The presence in the electrolyte of nickel, zinc, iron, or lead in proportions up to 10% of the copper present yields on electrolysis at 50° for about 50 hrs. with a current density of 2 amp./dm.² and a copper concentration of 42—52 g./kg. a cathode deposit containing not more than 0.007% of the foreign metal.

H. F. GILLBE.

Rapid analysis of manganese-brasses. HABERLAND (Chem.-Ztg., 1930, 54, 346).—The method consists in separating the manganese as manganese dioxide by treating the evaporated nitric acid solution with solid potassium chlorate, followed by separation of the copper and lead by electrolysis, removal of the iron and aluminium by treatment with ammonia, separation of nickel as "nickel oxime," and finally calculation of zinc by difference.

H. J. DOWDEN.

System nickel-iron-copper. P. R. KOSTING (Rensselaer Poly. Inst., Eng. & Sci. Series, 1930, No. 26, 1—27).—The following properties of iron-nickel-copper alloys have been investigated: temperature coefficients of electrical resistance between 20° and 100°, sp. resistances at 20°, thermoelectric forces against copper, and coefficients of thermal expansion. The boundary found for the immiscible area agrees with previous determinations. Additions to constantan of iron up to 15%, the limit of solubility, decrease the sp. resistance and thermoelectric force and increase the temperature coefficient of resistance. Addition of copper to invar up to the solubility limit of 22% decreases the resistance and increases the temperature coefficient of resistance, the thermoelectric force, the coefficient of expansion, and the temperature at which the expansion suddenly increases. Small additions of iron to non-magnetic copper-nickel alloys make them magnetic.

C. W. GIBBY.

Corrosion of monel metal and acid-resisting bronze. M. G. BINEK (Korrosion u. Met., 1929, 5, 247—248; Chem. Zentr., 1930, i, 1045).—The monel metal (Ni 62.2, Cu 34.7, Al 3.0%, lead, and iron) and the bronze (Cu 81.6, Pb 10.0, Sn 7.9%, iron, zinc, nickel, and phosphorus) were attacked in diminishing degree (the monel metal less than the bronze) by 10% hydrochloric acid, chamber sulphuric acid (19.5%)

containing nitric acid, contact sulphuric acid, and sulphuric acid (19.5%).

A. A. ELDRIDGE.

Rapid determination of bismuth and copper in lead bullion by internal electrolysis. E. M. COLLIN (Analyst, 1930, 55, 312—318).—The method as carried out by Sand's internal-electrolysis apparatus (cf. A., 1930, 880) was devised for the determination of amounts of bismuth less than 0.01 g. in the presence of up to 10 g. of lead. To 5 g. of sample dissolved in 50 c.c. of 20% nitric acid containing 1 g. of added tartaric acid are added 2 c.c. of 2% hydrochloric acid, the silver chloride and any insoluble residue from the lead are filtered off, the precipitate is washed with hot water, the filtrate diluted to 100 c.c. and cooled, and a 2% potassium permanganate solution added to convert any tervalent into quinquevalent antimony. After addition of 5 c.c. of a 5% solution of hydroxylamine hydrochloride and dilution to 200 c.c., the liquid is electrolysed at 85—90° for 15 min. The bismuth and copper are separated from the combined deposit by means of ammonia solution and ammonium carbonate. Amounts of copper and bismuth found agreed very closely with those added to a pure lead with unknown amounts of antimony and silver.

D. G. HEWER.

Influence of cyclic stress on corrosion. D. J. MCADAM, JUN. (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 329).—The results are expressed diagrammatically. Even low stresses accelerate the damage due to corrosion.

CHEMICAL ABSTRACTS.

Medium for flotation cells. DYER and McCLELLAND.—See I. **Functions of coke ovens.** STEWART.—See II. **Sheet-steel enamels.** ANDREWS.—See VIII. **Aircraft finishes.** GARDNER.—See XIII.

PATENTS.

Multiple-hearth furnace. E. J. FOWLER and D. BAIRD, ASSRS. to NICHOLS COPPER CO. (U.S.P. 1,751,142, 18.3.30. Appl., 8.3.28).—In a multiple-hearth roaster, the air which cools the rabbles is used also to withdraw additional heat from some of the intermediate hearths by means of pipe coils or other devices attached to the central shaft; the heat so withdrawn may be transferred to other hearths, preferably by blowing the air into contact with the ore.

B. M. VENABLES.

Hardening of copper. N. O. HEDMAN, N. R. NÄSLUND, and J. D. LARSSON (B.P. 330,408, 17.6.29).—The copper or an article made thereof is heated to about 600° and then cooled by dipping in a bath containing 14 vols. of sulphuric acid and 1 vol. of nitric acid, to which are added 5—10 g. of potash per litre.

H. ROYAL-DAWSON.

Separation of minerals by flotation. F. G. MOSES, R. W. HESS, and R. L. PERKINS, ASSRS. to BARRETT CO. (U.S.P. 1,739,369, 10.12.29. Appl., 20.5.27).—2-Thiopyridine is used as flotation agent.

C. HOLLINS.

Manufacture of fluxes [for low-temperature welding]. J. A. ROGNON (B.P. 316,196, 24.7.29. Fr., 24.7.28).—A flux suitable for welding aluminium and its alloys, without preparatory cleaning and without a metal solder, consists of a powdered mixture of borax

18%, zinc chloride 40%, sodium bromide 34%, and aluminium or ammonium bromide 8%.

W. G. CAREY.

Metallurgical furnace. D. CUSHING (U.S.P. 1,766,110, 24.6.30. Appl., 12.12.28. U.K., 26.9.27).—See B.P. 303,042; B., 1929, 175.

Apparatus for producing metals from their oxide ores. W. E. TRENT, ASSR. to TRENT PROCESS CORP. (U.S.P. 1,767,779, 24.6.30. Appl., 11.5.28).—See B.P. 316,986; B., 1930, 669.

Lead alloy. S. BECKINSALE and H. WATERHOUSE (U.S.P. 1,766,871, 24.6.30. Appl., 11.4.27. U.K., 16.4.26).—See B.P. 272,320; B., 1927, 606.

Composition for soldering metals. K. GEISEL, ASSR. to ALUMINUM SOLDER CORP. OF AMERICA (U.S.P. 1,761,116, 3.6.30. Appl., 28.5.28. Ger., 20.2.28).—See B.P. 315,010; B., 1929, 726.

Coating, impregnating, or alloying metals and other materials with aluminium and aluminium alloys. W. SMITH, ASSR. to EXPANDED METAL CO., LTD. (U.S.P. 1,761,850, 3.6.30. Appl., 2.3.27. U.K., 17.3.26).—See B.P. 279,273; B., 1927, 943.

Production of [metallic] acid-resisting wrapping material. K. HUMPERT, ASSR. to STANIOLFABR. BURG-DORF A.-G. (U.S.P. 1,765,920, 24.6.30. Appl., 16.8.27. Switz., 17.6.27).—See B.P. 292,162; B., 1929, 61.

[Jarring mould device for] production of sound ingots. A. G. EGLER (B.P. 303,157, 21.12.28. U.S., 30.12.27).

Apparatus for intercepting furnace dust in blast furnaces. J. STOECKER (B.P. 330,928, 19.3.29).

Method and apparatus for producing a metallic spray. E. BRIDGER (B.P. 308,355, 19.3.29. N. Zealand, 22.3.28).

Lead[—antimony] alloy tanks. W. T. BUTCHER, W. CARROTT, and LOCKE, LANCASTER and W. W. & R. JOHNSON & SONS, LTD. (B.P. 330,924, 18.3.29).

Utilising [by re-compression] the lost and residual gases produced when liquefied gases of low b.p. are drawn off or transferred from one vessel to another, particularly for welding and cutting purposes. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 306,130, 31.1.29. Ger., 16.2.28).

Heating furnace (B.P. 302,652 and 330,160). **Heating of metal sheets** (U.S.P. 1,750,884).—See I. **Reduction of metallic compounds** (B.P. 315,811). **Siliceous minerals** (U.S.P. 1,748,989).—See VII. **Zinc-white pigments** (B.P. 312,648).—See XIII.

XI.—ELECTROTECHNICS.

Copper oxide rectifier. W. OGAWA, C. NEMOTO, and S. KANEKO (J. Soc. Chem. Ind., Japan, 1930, 33, 125 B).—The contact between the cuprous oxide and copper is imperfect; the oxide has a larger electron affinity than the copper, so that the number of electrons emitted from the copper by an alternating electric impulse exceeds the number emitted from the oxide, and consequently rectification results in the direction of oxide to copper. Microscopical examination of the

contact and investigation of the relation between the form of the copper and the rectifying action support this explanation. An efficient rectifying suboxide of copper (? cuprous oxide) is prepared by heating copper embedded in magnesium oxide. S. K. TWEEDY.

Influence of the shape of carbon electrodes on the properties of carbon-zinc elements. N. S. KRIVOLUTSKAYA and G. G. MOROZOV (J. Appl. Chem., Russia, 1929, 2, 703—718).—Better utilisation of manganese dioxide and improved depolarising properties of the elements are obtained by decreasing to an optimal value the current density through the carbon-manganese dioxide agglomerate. The use of flat carbon electrodes and square agglomerates increases the capacity of the element by 25%. The thickness of the layer of agglomerate is not important.

CHEMICAL ABSTRACTS.

Corrosion of the zinc electrode in a dry cell during open circuit. S. MAKINO (J. Soc. Chem. Ind., Japan, 1930, 33, 176 B).—The presence of positive manganese dioxide electrodes in dry cells increases the chemical corrosion of the negative electrodes during open circuit. O. J. WALKER.

Sludge of transformer oils. YAMADA.—See II. **Measurement of colour and gloss.** DESBLEDS.—See V. **Controlling acid effluents.** RICHARDSON.—See VII. **Coatings for iron and steel.** BASSETT. **Determination of inclusions in steel.** HERTY and others. **Copper refining.** KAMEYAMA and ONODA. **Analysis of manganese-brasses.** HABERLAND. **System nickel-iron-copper.** KOSTING. **Determination of bismuth and copper.** COLLIN.—See X. **Alkalinity of fruit produce.** HIRSCH and RICHTER.—See XIX.

PATENTS.

Electric [muffle] furnace. H. G. CAMPBELL (B.P. 329,830, 15.5.29).—Electric heating elements are arranged in series in the furnace casing and door, and current flows through both elements whether the door be open or shut. J. S. G. THOMAS.

Electric furnace. C. E. CORNELIUS (B.P. 303,798, 8.1.29. Swed., 9.1.28).—In furnaces suitable for the manufacture of glass, water-glass, aluminous cement, etc., the electrode bodies are fixed on the furnace bottom wholly within the furnace and are spaced from all the furnace walls. J. S. G. THOMAS.

Carbon electrodes for electric furnaces. GEBR. SIEMENS & Co. (G.P. 461,094, 6.5.25).—The electrode contains a metal insertion, e.g., a network of expanded metal, which is connected, e.g., by spot-welding, to a metal casing which embraces the exterior of the electrode. S. K. TWEEDY.

[Heating elements for crucible] electric furnaces. L. D. J. TISSEYRE (B.P. 305,631, 7.2.29. Fr., 8.2.28).—A continuous graphite resistance, formed of rigid elements placed end to end and preferably of rectilinear or curvilinear shape, surrounds the crucible, so that current flows through the resistance in a plane or planes at right angles to the longitudinal axis of the crucible. [Stat. ref.] J. S. G. THOMAS.

Electric furnace [for dental purposes]. R. SCHMITZ (B.P. 302,594, 17.12.28. Ger., 16.12.27).—An

auxiliary furnace with independent heating elements is inserted into a member, which is suspended in the shaft above the principal heating chamber, and can be easily replaced by a simple insulating member of the same external shape. Means are provided for connecting the muffles hermetically, for observing the melting, alloying, and casting processes, and for introducing compressed air. J. S. G. THOMAS.

[Electric] tunnel kiln and its operation. F. A. J. FITZGERALD and J. KELLEHER, Assrs. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,749,762, 11.3.30. Appl., 10.6.25).—Material moved through the furnace is heated to an intermediate temperature by radiation from a metallic resistor exposed to the furnace atmosphere, and to its final temperature by radiation and conduction from a carbonaceous resistor surrounded by an inert atmosphere within a silicon carbide enclosure. J. S. G. THOMAS.

Flux-coated [iron] electrodes for electric welding. J. MEHL (B.P. 310,341, 23.4.29. Ger., 23.4.28).—The coating surrounding an iron core contains substances which, at the m.p. of the electrode, evolve oxygen which consumes a portion of the core. Thus, e.g., the coating may consist of 5% of asbestos, 90% of manganese dioxide, 3% of chromium oxide, and 2% of vanadium oxide, worked up into a paste with sodium silicate. [Stat. ref.] J. S. G. THOMAS.

Incandescence-cathode device [rectifier]. H. C. RENTSCHLER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,749,780, 11.3.30. Appl., 31.1.22).—A large iron anode, and a small thorium cathode in series with and heated by a tungsten heating coil, are arranged in a sealed envelope. J. S. G. THOMAS.

Thermionic cathodes. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. F. PERROTT and J. J. FITZPATRICK (B.P. 303,381, 31.12.28. U.S., 31.12.27).—A nickel core coated with the carbonate of an alkaline-earth metal and preferably with a binder, e.g., a cellulose compound dissolved in amyl acetate, is heated in a protective environment, e.g., *in vacuo*, to a temperature above 1050° until a material of higher electron-emissive power than that of the oxide of the alkaline-earth metal is produced. [Stat. ref.] J. S. G. THOMAS.

Manufacture of oxide, sulphide, and haloid cathodes for electric-discharge devices. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 313,151, 1.5.29. Ger., 8.6.28).—A cathode core, coated with the oxide, sulphide, or halide of a refractory metal, e.g., tungsten or molybdenum, or, alternatively, with one or more compounds yielding such oxide, sulphide, or halide when heated, e.g., more especially ammonium tungstate or sulphotungstate, is coated with a deposit of an alkaline-earth metal and then heated for a short time at 1500°. J. S. G. THOMAS.

Electron-discharge tubes or thermionic valves [with incandescence cathodes]. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 329,854, 29.5.29. Holl., 15.12.28).—A conductor of high sp. electric resistance, e.g., of zirconium, capable of absorbing residual gases or impurities at high temperatures, but which does not emit electrons, is connected in parallel with the cathode. J. S. G. THOMAS.

Incandescence bodies, e.g., filaments, for electric incandescence lamps, vacuum tubes, etc. SIEMENS & HALSKE A.-G. (B.P. 309,536, 2.3.29. Ger., 12.4.28).—A core of tungsten is coated with eka-manganese (atomic number 75) or with an alloy of these two metals.

J. S. G. THOMAS.

Electric filaments and resistances. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 330,280, 4.3.29).—The core of an incandescence cathode consists of zirconium together with a small proportion, e.g., 1.5–2%, of aluminium. Such cathodes are characterised by comparatively small variations of voltage being produced by relatively large variations of current flowing in the cathode.

J. S. G. THOMAS.

Luminous electric-discharge tubes. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 316,611, 5.7.29. Ger., 1.8.28).—Material for replenishing the gas filling of discharge tubes is provided in the form of a compact mass; e.g., magnesium carbonate, sodium nitride, or barium azide, mixed with powdered glass or enamel, is heated by an electric heater, which is normally out of circuit, but which is automatically connected in circuit when the current loading on the tube attains a predetermined value.

J. S. G. THOMAS.

Electric lamp. J. RISLER, Asst. to RISLER CORP. OF AMERICA (U.S.P. 1,748,396, 25.2.30. Appl., 8.8.27. Fr., 2.11.26).—An electric discharge tube filled with a mixture containing 70% of neon and 30% of helium at a total pressure of between 10^{-4} and 5 mm. Hg is claimed.

H. ROYAL-DAWSON.

Photoelectric cells. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 319,650, 26.2.29. Holl., 25.9.28).—A layer of, e.g., calcium fluoride or magnesia is sandwiched between a layer of the photoelectric material, preferably an alkali metal, e.g., caesium, and a substratum which is usually the support for the photoelectric material.

J. S. G. THOMAS.

Photoelectric cells. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of V. K. ZWORYKIN (B.P. 307,082, 2.3.29. U.S., 3.3.28).—A very thin, approximately uni-molecular, photosensitive film, e.g., of caesium, produced by heating caesium trinitride, is deposited upon the conducting surface, composed, e.g., of magnesium or an alkaline-earth metal, of the cell.

J. S. G. THOMAS.

Light-[photo]electric cells. F. ROTHER (B.P. 311,701, 10.4.29. Ger., 14.5.28).—The anode or cathode, or both, or an auxiliary electrode consist(s) of cold radioactive material (e.g., thorium or thorium alloy), the α -particles from which penetrate the photo-cathode, whereby the photoelectric current is increased.

J. S. G. THOMAS.

Selenium cell. "SELENOPHON" LICHT- u. TONBILDGES. M.B.H. (B.P. 330,258, 1.3.29. Austr., 9.2.29).—In a selenium cell of the condenser type, metal plates which cannot be wetted by molten selenium constitute the electrodes and are connected with a source of current, whilst plates capable of being wetted by molten selenium are arranged at the sides or between these plates and are not in direct connexion with a source of current.

J. S. G. THOMAS.

Dry metal rectifier cells. SIEMENS-SCHUCKERT-

WERKE A.-G. (B.P. 308,306, 8.3.29. Ger., 21.3.28).—A metal plate, e.g., of copper, coated on each side with a compound (cuprous oxide) and a superposed layer of sprayed metal (zinc), is provided with spring-metal strips, e.g., of copper or brass, bearing on the metal compound layers.

J. S. G. THOMAS.

Electrode for electrolytic cells. E. F. LUNDEEN, Asst. to WILLARD STORAGE BATTERY CO. (U.S.P. 1,749,145, 4.3.30. Appl., 31.1.27).—An electrolytic cell comprises an electrolyte containing citric acid, a film-forming electrode, and an iron-chromium electrode containing more than 10% by wt., preferably 13%, of chromium.

J. S. G. THOMAS.

Electrolyte for rectifiers, condensers, and the like. W. D. DOOLEY (U.S.P. 1,748,011, 18.2.30. Appl., 18.7.27).—The solution consists of sodium silicate 30%, sodium hydroxide 5%, gelatin 5%, and water 60%.

H. ROYAL-DAWSON.

Conserving storage-battery solution. C. B. DILTZ (U.S.P. 1,749,665, 4.3.30. Appl., 4.2.27).—A storage-battery electrolyte consists of an aqueous solution of sulphuric acid (d 1.25) containing $\frac{1}{2}$ lb. of sodium carbonate, $1\frac{1}{2}$ oz. of boric acid, $1\frac{1}{2}$ oz. of tartaric acid, 1 oz. of carmine, and 2 oz. of sodium silicate per gal.

J. S. G. THOMAS.

Manufacture of electric insulating material [from glass]. R. HADDAN. From CORNING GLASS WORKS (B.P. 330,268, 4.3.29).—Insulating sheets of thickness less than 0.015 in. are made from glass having a softening point below 700°.

J. S. G. THOMAS.

Electric insulating material. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 310,894, 2.5.29. Ger., 2.5.28).—Artificial material made from a glass flux containing mica or asbestos is coated with mica or mica flakes.

J. S. G. THOMAS.

Insulating materials [for under-water cables etc.]. WESTERN ELECTRIC CO., LTD. From BELL TELEPHONE LABS., INC. (B.P. 329,683, 23.2.29).—Deproteinised rubber (cf. B.P. 307,966; B., 1929, 401) and deresinated gutta-percha are combined, preferably in about equal proportions, and, if desired, montan wax or pitch is added.

J. S. G. THOMAS.

Insulation of electric wires and cables. BRIT. INSULATED CABLES, LTD., and F. J. BRISLEE (B.P. 330,552, 9.2. and 9.3.29).—Insulation embodying an esterified cellulose composition, applied, under approximately atmospheric pressure, in layers round a conductor with a textile support and substantially freed from solvent, is consolidated by heat-treatment applied simultaneously or otherwise to all the layers, so that adjacent layers unite and the composition impregnates the textile support.

J. S. G. THOMAS.

Insulating oil for electrotechnical purposes. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 301,876, 6.12.28. Ger., 7.12.27).—Raw oil obtained by a process of non-cracking distillation, e.g., of Texas crude oil, is mixed with resin.

J. S. G. THOMAS.

Electrolytic apparatus. A. E. KNOWLES (U.S.P. 1,767,375, 24.6.30. Appl., 13.4.29. U.K., 14.4.28).—See B.P. 320,388; B., 1930, 21.

Gas washer for electrolytic apparatus. A. E. KNOWLES (U.S.P. 1,767,292, 24.6.30. Appl., 15.10.26. U.K., 19.10.25).—See B.P. 261,164; B., 1927, 81.

[Multi-cellular, high-voltage, dry] electric batteries. BURGESS BATTERY Co., Assees. of W. B. SCHULTE (B.P. 312,605, 5.4.29. U.S., 28.5.28).

[Sealing the covers of] electric accumulators. W. and W. HADDON (B.P. 330,698, 18.4.29).

[Pressure] electrolyser with collecting block. J. E. NOEGGERATH (B.P. 307,934, 15.3.29. Ger., 16.3.28).

[Mica-glass] gastight [anode] seals [for mercury-arc rectifiers] and their manufacture. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. BUECHNER (B.P. 318,555, 5.9.29. U.S., 5.9.28).

Thermionic cathodes. E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 330,331, 26.3.29. Addn. to B.P. 278,787).

Electric-discharge tubes [emitting positive-column light]. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 315,699, 17.6.29. Holl., 16.7.28).

[Cathodes for] thermionic valves. B. LOEWE (B.P. 307,028, 1.3.29. Ger., 1.3.28).

Metallic-vapour and gaseous arc-discharge devices. P. FREEDMAN (B.P. 329,627, 16.2. and 16.12.29).

Photoelectric cell. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 329,887, 5.7.29. Austr., 2.1.29).

Manufacture of light-sensitive cells. W. ENGELKE (B.P. 330,807, 18.7.29).

X-Ray tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 331,052, 17.5.29).

Electrolytic deposition of metal on metal tubes. J. STONE & Co., LTD., W. LAMBERT, and R. W. WILD (B.P. 330,639, 15.3.29).

Gas-analysis apparatus (U.S.P. 1,751,715). Light filter (U.S.P. 1,751,220).—See I. Purification of used mineral oils (U.S.P. 1,747,161).—See II. Degreasing wool etc. (B.P. 304,295).—See V. Dehydration (U.S.P. 1,749,455).—See VII. Coating composition (B.P. 303,169). Resinous products (U.S.P. 1,737,916—8).—See XIII. Sterilisation etc. of fruit juices (B.P. 330,368).—See XIX. Photographic etching (U.S.P. 1,751,909).—See XXI.

XII.—FATS; OILS; WAXES.

Antioxidants of fats and oils. I. Antioxygenic powers of α - and β -naphthols. Y. TANAKA and M. NAKAMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 107—109 B).—At room temperature both naphthols have strong antioxidising activity (linseed oil containing 0.5—5% of α -naphthol and exposed to air retained its iodine value unchanged for 78 days), α -naphthol being rather the more active, and especially at higher temperatures; at 100—150° the antioxidising power is retained, but loss occurs through volatility. It is suggested that these substances may be added to semi-drying oils to be used as lubricants. E. LEWKOWITSCH.

Antioxidants of fats and oils. II. Action of phenols and some substitution products of mono-

hydric phenol. III. Combined effects of anti-oxidants and driers on the oxidation of linseed oil. IV. Action of antioxidants of rubber and some organic compounds on the oxidation of drying oils. Y. TANAKA and M. NAKAMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 126—127B, 127—128B, 129—130B; cf. preceding abstract).—II. The action of various phenols (concentration 0.01 mol.-%) on the oxidation of linseed oil exposed to sunlight for about 39 hrs. has been examined. All phenols possess an antioxygenic effect toward linseed oil; the activity sequence is pyrogallol > quinol > pyrocatechol > phloroglucinol (anhydrous) > resorcinol > phloroglucinol ($2H_2O$) > phenol. It is stated that the antioxygenic activity varies, not with the number of hydroxyl groups, but according to their nuclear position. The cresols and mononitrophenols are more active than is phenol, whilst the monochlorophenols possess very weak antioxygenic activity. For the nitro- and chloro-phenols, the activity is not always proportional to the number of substituents, but depends on their nuclear positions. The antioxygenic power decreases in the order $o > p > m$ for the cresols, but the activities of the three isomeric mononitrophenols are almost equal.

III. Experiments using lead and manganese salts of the fatty acids of linseed oil or lead and manganese resins as driers (0.2%, calc. as the metal content) and pyrocatechol, α -naphthol, and β -naphthylamine as antioxidants (0.5%) indicate that in the case of co-existence of antioxidant and drier, the former does not entirely destroy the activity of the latter, but prolongs the induction period of drying oil; the retarding action of the antioxidant on the activity of the drier is selective. The activity sequence for antioxidants in the presence of an equal amount of a certain drier corresponds with that for the antioxidants alone (cf. Long and Egge, B., 1928, 718).

IV. The antioxidant activity of 9 commercial preparations (used in the rubber industry) in 1% concentration on "insolated" linseed oil has been examined, together with 12 organic compounds in varying concentrations. Antioxidants of rubber all possess antioxidant activity toward linseed oil; the experiments were performed at the same time as those described in II (above), and the efficiencies of the various preparations are given in terms of the activity sequence (above). 1-Methylnaphthalene, anthracene, and alizarin (although possessing phenolic properties) accelerate oxidation of linseed oil, whilst *ac*-tetrahydro- β -naphthol (resembling an aliphatic alcohol) acts as an antioxidant. The greatest antioxidant activity was exhibited by β -naphthylamine, which was followed in order by pyrocatechol and α -naphthol. C. W. SHOPPEE.

Bromometry of fats and oils. S. YUSHKEVICH (Masloboino Zhir. Delo, 1929, 45, 39—44).—Rosenmund and Kuhn's method is recommended for technical purposes; the reaction time should be prolonged to 10—15 min. Results obtained by Winkler's method are higher and not concordant.

CHEMICAL ABSTRACTS.

Losses of nickel in hydrogenation plants. A. KALYUZHNIN (Masloboino Zhir. Delo, 1929, 45, 10—13).—Loss of nickel in the filtrate after precipitation of the

sludge (treated with sulphuric acid) with sodium carbonate is avoided by precipitation at the b.p. Hydrolysis of fat, resulting in combination of nickel with fatty acids, is avoided by using dry hydrogen. The consumption of nickel sulphate was reduced from 2.17 to 1.81 kg. per ton of fat.

CHEMICAL ABSTRACTS.

Determination of acetyl value of oils and fats by thermometric titration. T. SOMIYA (J. Soc. Chem. Ind., Japan, 1930, 33, 140—142 B).—The dry oil is heated with an equal weight of acetic anhydride in a vapour-tight pressure bottle at 140° for 2 hrs., the excess of acetic anhydride in an aliquot part being determined in the manner already described (B., 1929, 274). The method gives accurate results. C. W. SHOPPEE.

Deodorisation of fish oil fatty acid or its soap by Varrentrapp's reaction. M. HIROSE (J. Soc. Chem. Ind., Japan, 1930, 33, 90—91 B).—Odourless soap can be obtained from fish oils by Varrentrapp's reaction in the presence of wax alcohols from sperm oil, e.g., by heating herring oil with an equal weight of the alcohols and 35—42% of caustic soda at 210—240°. As the wax alcohols themselves are oxidised to fatty acids the yield of insoluble fatty acid is over 100% (iodine value 40—65 compared with 105—127 for original fatty acids). The wax alcohols treated alone gave 20—30% of fatty acids and a loss of 50—30% by volatilisation (mostly cetyl alcohol). The soap obtained by this method possesses lathering and detergent properties comparable with those of commercial laundry soap and can be used in combination with other soaps. Soap free from wax alcohol is obtained by distilling under reduced pressure.

E. LEWKOWITSCH.

Physico-chemical principles of washing materials [soaps]. L. ZAKARIAS (Chem.-Ztg., 1930, 54, 328—329).—Methods and apparatus for the measurement of the protective colloid action and foam-producing capacity of soap solutions are briefly described. Measurement of p_H of a dilute solution of soap containing an excess (0.05—0.1%) of alkali showed that when the foam is gradually removed the solution has an acid reaction; part of the soap is therefore wasted unless additional alkali be added. Reference is made to the author's proposal to use alkaline polysaccharide solutions as detergents (cf. Leipziger Monatsschr. Textilind., March, 1930). The explanation of the washing action is that the alkali peptises the dirt, which then forms a mixed colloid with the colloidal component and is thus not easily reprecipitated on the fabric. The alkaline component also enlarges the pores of the fabric and facilitates rinsing.

H. J. DOWDEN.

Composition and polymerisation of Chinese wood (tung) oil. E. R. BOLTON and K. A. WILLIAMS (Analyst, 1930, 55, 360—367).—Figures obtained by the authors' method for polymerisable matter (B., 1926, 712) and for the glyceride of elæostearic acid in the same samples of tung oil showed such close agreement that it is assumed that the polymerisable matter consists entirely of the glyceride of elæostearic acid; hence it follows that the original method of determining the polymerisable matter determines also the glyceride. The separate determination of the glyceride of elæostearic acid in tung oil was made by dividing the differ-

ence between the bromine value and the true or instantaneous iodine value by 87.2 (the difference between the bromine value, in terms of iodine, corresponding to saturation of all three double linkings, and the true iodine value corresponding to absorption of four atoms of hydrogen per mol. of acid) and multiplying by 100, since the amount of iodine monochloride absorbed by all other constituents of the oil is exactly equivalent to that of the bromine. The true iodine value ($T.I.V.$; method described in detail) may be determined graphically from the iodine values ($I.V.$) obtained after absorption has proceeded for 30 min. and for 3 hrs., or from the expression: $T.I.V. = I.V._{30 \text{ min.}} - \frac{1}{5}(I.V._{3 \text{ hrs.}} - I.V._{30 \text{ min.}})$. If, however, the iodine value of elæostearic acid is defined as the percentage of halogen in terms of iodine absorbed by four of the unsaturated atoms, then this figure may be obtained with sufficient accuracy for practical purposes by an absorption limited to 20 min.

D. G. HEWER.

Apparent iodine value of tung oil. J. VAN LOON (Chem. Umschau, 1930, 37, 175—177).—It is emphasised that the "iodine value" of tung oil depends on the type of reagent used, the time of reaction, and the halogen excess, and therefore cannot be considered as a criterion of purity or quality of the oil. True iodine values can only be obtained after some 7 days' contact with considerable excess of iodine chloride or bromine solutions.

E. LEWKOWITSCH.

Oil of cucumber seeds. G. EINHORN, A. MILSKI, and E. KALASHNIKOV (Masloboino Zhir. Delo, 1929, 45, 44—48).—The ether-extracted oil had d_{25}^{25} 0.9251, n_D^{20} 1.4750, f.p. —3.5°, saponif. value 191.1, Hübl value 115.3, Reichert-Meißl value 1.05, Polenske value 0.87, acetyl value 16.6, free acids 1.76%, unsaponifiable matter 1.91%; the acids consisted of oleic (58.49), linoleic (22.29), palmitic (6.79), and stearic (3.72% of oil) acids. The oil was richer in vitamin-A than sunflower oil. The seed contained moisture 8.0, oil 31.47, protein 29.69, carbohydrates 1.88, cellulose 13.89, pentosans 4.67, pectins 0.59, ash 3.92, P_2O_5 (in ash) 2.25, phytin 1.1, lecithin 2.60%.

CHEMICAL ABSTRACTS.

Effect of dissociated water vapour on vegetable oils. G. I. LAVIN and E. E. REID (J. Amer. Chem. Soc., 1930, 52, 2454—2455).—When water vapour is passed through an electric-discharge tube and then through cottonseed, olive, linseed, or castor oil, a white fibrous substance, insoluble in organic solvents, is produced.

H. BURTON.

Causes of turbidity in processed drying oils. V. KISELEV and N. SUKHANOV (Masloboino Zhir. Delo, 1929, 46, 24—30).—Samples of raw, bleached, and cold-treated linseed oil were processed with cobalt, manganese, and lead driers. Since the cold-treated oil gave the best results, the mucilaginous substances cannot act as a protective colloid. Cobalt-manganese driers did not cause turbidity in 5 months, whilst the lead-treated oils became turbid during the first 2 weeks. Air-blown linseed oil keeps better than unblown oil when cobalt or manganese is used, whilst the reverse holds for lead. Light has only a slight effect. Moisture accelerates the appearance of turbidity.

CHEMICAL ABSTRACTS.

Homogenisers. MÖLLERING. Medium for flotation cells. DYER and McCLELLAND.—See I. Utilising soya beans. TAKAYAMA.—See III. Colour lakes. CLAYTON.—See XIII. Fat in wheaten products. HERD and AMOS. Fatty acids of food fats. GROSSFELD and SIMMER.—See XIX.

PATENTS.

Extraction of oils from vegetable matter [cacao bean, ground-nut, etc.]. I. S. HOCKER, Assr. to HOCKER CORP. (U.S.P. 1,751,331, 18.3.30. Appl., 10.3.26).—The material is roasted, finely-divided, and mixed with water at a temperature above the m.p. of the fatty constituent; after heating the mixture to about the b.p. and cooling, a saccharifying enzyme and sugar are added (in order to solubilise undissolved starch) and the whole is then heated at about 65° for 5–10 min. before separating the oil by centrifuging.

E. LEWKOWITSCH.

Purification of vegetable oils. J. C. HEBDEN, Assr. to HEBDEN SUGAR PROCESS CORP. (U.S.P. 1,745,367, 4.2.30. Appl., 2.4.23).—The oil is mixed with about 10% of its volume of an aqueous suspension (0.01%) of the basic tannate of aluminium, iron, or titanium (cf. U.S.P. 1,545,318–9; B., 1925, 819) at ordinary temperatures. The non-oleaginous matter, foots, etc. are coagulated and removed with the water by centrifuging.

E. LEWKOWITSCH.

Treatment of cod-liver oil. J. C. HAMILTON, B. H. THURMAN, and L. G. COPES, Assrs. to BEST FOODS, INC. (U.S.P. 1,745,851, 4.2.30. Appl., 6.8.26).—The oil is drawn off from the foots after agitation with alkali and settling, and is allowed to percolate, at room temperature, through a mass of granular active carbon through which a countercurrent of inert non-toxic gas (e.g., carbon dioxide) passes. The oil may be heated with a clarifying agent and filtered before percolation, if desired.

E. LEWKOWITSCH.

Preservation of oils [e.g., cod-liver oil]. W. G. CHRISTIANSEN, F. R. CHAPPELL, and A. E. BRIOD, Assrs. to E. R. SQUIBB & SONS (U.S.P. 1,745,604, 4.2.30. Appl., 5.11.27).—The return of unpleasant flavour to a deodorised cod-liver oil is inhibited by the addition of small amounts (e.g., 0.1%) of certain compounds of the class of phenols, aromatic amines, and aminophenols, before storage.

E. LEWKOWITSCH.

Treatment of soap [during cooling]. A. JACOBI A.-G. (B.P. 308,985, 2.4.29. Ger., 30.3.28).—Soap is shaken during the whole or part of the cooling operation.

H. ROYAL-DAWSON.

Separation of substances of dissimilar volatilities [e.g., refining fats]. E. WECKER (U.S.P. 1,766,863, 24.6.30. Appl., 23.7.26. Ger., 17.3.26).—See B.P. 277,085; B., 1927, 883.

Production of soap threads. A. H. CHARLTON, Assr. to T. B. ROWE & CO., LTD. (U.S.P. 1,764,866, 17.6.30. Appl., 1.11.26. U.K., 24.11.25).—See B.P. 267,999; B., 1927, 417.

Washing of textiles (U.S.P. 1,746,170).—See VI. **Fatty products** (B.P. 324,276). **Artificial cream** (B.P. 330,307). **Bleaching foodstuffs** (B.P. 327,670). See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Mixing, grinding, and refining of paints and enamels. S. SMITH (J. Oil Col. Chem. Assoc., 1930, 13, 163–167).—A lecture. Progress in the industry during the last 30 years is reviewed. The method of mixing recommended is to place some dry colour in the machine, to add sufficient medium to form a paste, and then gradually to feed in sufficient colour and liquid to keep the material in the machine in the paste form. For “grinding,” flat stone mills are considered best, though these have been largely replaced by roller mills. A two-roll mill with differential speeds not exceeding 4:1 will give a product superior to that obtained from a triple-roll mill. In the refining of paint the single-roll mill is very satisfactory. The recently introduced disc-bar mill not only refines but machines paste, paints, and enamels to any consistency, giving an output of ready-mixed paints and enamels of 60–80 gals./hr.

F. C. HARWOOD.

Influence of drying conditions on the resistance to weathering of paints. H. WOLFF (Farben-Ztg., 1930, 35, 1933–1934).—Three blue paints of different composition were allowed to dry under entirely different conditions of temperature, humidity, and light. After 1½ years' weathering under natural conditions the general conclusion is drawn that the conditions of drying of a paint film profoundly modify the film, and hence its resistance to weathering. Specifically, films dried under the influence of ultra-violet light showed the greatest durability.

J. O. CUTTER.

Colour lakes—their manufacture and uses. H. CLAYTON (J. Soc. Dyers and Col., 1930, 46, 154–157).—Colour lakes (largely red) usually consist of the calcium or barium salts of red dyes slightly soluble or insoluble in water. The barium lake of Lithol Red is brighter and yellower than the calcium lake, and neither is suitable as a pulp lake since each requires dry-grinding for full development of its tinctorial power. Lithol Red lakes require a certain minimum period to assume stable properties between drying and grinding with oil. The calcium lakes of Permanent Red 4B and 2B are important bright bluish-reds which give blue undertones and are thus popular with lithographic printers. Helio Fast Red RL, the Hansa Yellows, and Pigment Green B, being insoluble, are not precipitated as lakes, but are simply ground with an extender. Lakes from acid dyes are chiefly precipitated with barium chloride, and the higher the temperature of precipitation the brighter is the resulting lake. Lakes made from basic dyes by precipitation with tannic acid or Katanol are generally fugitive to light, spirit, and alkali, and after drying are difficult to grind. Tungstic acid lakes are slightly faster to light than those prepared with tannic acid, but much less fast than the Fanal and Brillfast colour lakes. Although direct-cotton dyes may be easily converted into lakes by precipitation with barium chloride or aluminium hydroxide, such lakes are generally dull and have low colouring power. With the exception of red alizarin lakes, which are among the fastest to light of lake reds, the mordant dyes are little used for the manufacture of lakes. Lakes made from vat dyes are very expensive and dull in shade; a vat colour lake

ground in oil with zinc oxide fades after a few days' exposure, whereas the same lake ground with white lead requires 3 months to fade to the same extent. The degree of grinding of a lake can largely affect the rate of drying of the paint made from it. Lime Green, made by grinding a solution of Brilliant Green Crystals or Malachite Green with dry green earth, whereby the dye is absorbed to the extent of 5%, is very fast to light, whereas Brilliant Green itself is very fugitive. A cellulose lacquer should contain not more than 10% of pigment, so that lakes for this purpose should be very concentrated and finely divided. The presence of a colour lake considerably prolongs the life of the cellulose lacquer film. The Fanal lakes are produced from basic dyes by the formation of a complex salt of phosphomolybdotungstic acid; the higher the mol. wt. of the lake the faster it is to light. Other applications of lakes in industry are: for paints and enamels, inks etc., paper coating or dyeing, distempers etc., leather, wallpaper, calico printing, rubber. A. J. HALL.

Effect of the addition of chalk on bright pigments. H. WAGNER and I. KESSELRING (Farben-Ztg., 1930, 35, 1982—1984).—Chalk when used as a substrate for pigments has the following advantages: small particle size, low sp. gr., soft texture, surface activity, and slight transparency to ultra-violet light; the pastes also have high plasticity. The external weathering of chalk paints is poor, but for internal use chalk gives durability and an excellent finish. J. O. CUTTER.

Polish turpentine. S. KRAUZE (Rocz. Chem., 1930, 10, 384—396).—The following substances have been found in Polish wood turpentine oil (b.p. 152—180°): α -pinene, isopinene, $\Delta^{1,2}$ - and $\Delta^{5,6}$ -carene, and sylvestrene. Satisfactory purification of the oil cannot be effected by the action of 3% sulphuric acid or of phosphoric acid; the use of the latter reagent, moreover, raises the b.p. and destroys the optical activity of the oil. Potassium hydroxide solutions destroy the α -pinene fractions and induce polymerisation. The addition of metallic sodium affords a convenient method of purifying the oil. The presence of degradation products in the oil is best detected by addition of calcium hypophosphite in hydrochloric acid solution, when a brown to black coloration is obtained. The iodine value of crude wood turpentine oil is 315.1; after purification with sodium 355, as compared with the value 349.4 for crude German oil of turpentine and 357.2 for commercial α -pinene. The α -pinene fraction, b.p. below 160°, yields 23% of crude borneol on treatment with tetrachlorophthalic acid, whilst the fraction of b.p. 161—163°, which does not contain α -pinene, yields under analogous conditions 7—14% of borneol, which is also obtained in still smaller yields from all fractions of b.p. 163—167°. The borneol obtained from fractions of b.p. above 160° originates probably from isopinene, as β -pinene was not detected in the oil.

R. TRUSZKOWSKI.

Aircraft finishes. H. A. GARDNER (Proc. Amer. Soc. Testing Materials, June, 1930, Preprint, 9 pp.).—Various schemes for doping and finishing fabric wings etc. and exposure tests thereon are detailed. In view of the relative expense of acetate dope and the satis-

factory results obtained with nitrate dope, the latter is considered likely to replace the former. The use of pigmented dopes and top varnishes and the protective coatings particularly suitable for steel and for duralumin parts (the latter having a high coefficient of thermal expansion as compared with the former) are also discussed. S. S. WOOLF.

Resins. VI. Gutta-percha resins. E. STOCK (Farben-Ztg., 1930, 35, 1981—1982; cf. B., 1930, 623).—The usual chemical and physical constants of two gutta-percha resins, a treated resin called "Astrocopal," and of a Borneo fossil copal are given. The gutta-percha resins may be suitable for nitrocellulose, and satisfactory varnishes were made from the fossil copal. J. O. CUTTER.

Practical brush-out test for hiding power of paints. H. A. GARDNER, G. G. SWARD, and S. A. LEVY (Proc. Amer. Soc. Testing Materials, 1930, 30, ii. Preprint, 4 pp.).—An extract from a paper already abstracted (cf. B., 1930, 569). S. S. WOOLF.

Homogenisers. MÖLLERING.—See I. **Determination of lead and copper in beverages.** RICHARDSON.—See XIX.

PATENTS.

[Titanium] paints. H. WADE. From TITANIUM PIGMENT CO., INC. (B.P. 329,333, 12.11.28).—Paints comprising titanium pigments, a vehicle consisting principally of a drying oil, and a small proportion (e.g., 5% on the wt. of the paint) of cellulose nitrate as hardening agent are claimed. S. S. WOOLF.

Making a washable paint [distemper]. N.V. INDUSTRIEEL MAATS. "AMSTELLIN" (B.P. 309,553, 11.4.29. Holl., 12.4.28).—Part of the water is boiled with a mixture of sugar and starch; a mixture of linseed (stand) oil and a drying-oil varnish, with or without thinners, is stirred into this dispersion; more linseed oil is added, and, after keeping, the rest of the water and finally the dry pigment are mixed in. E. LEWKOWITSCH.

Preparation of coating compositions. BRIT. CELANESE, LTD. (B.P. 303,169, 28.12.28. U.S., 29.12.27).—Normally light-sensitive synthetic resins, or coatings containing them, are darkened by exposure to ultra-violet light before use; or the resin may be darkened during manufacture in the presence of benzoic or salicylic acid as catalyst. Coatings containing such darkened resins with cellulose derivatives, especially cellulose acetate, do not change colour on exposure to light. E. LEWKOWITSCH.

Materials for marking animals. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 329,408, 26.2.29).—Such materials comprise a suspension or solution of a colouring matter in an aqueous emulsion of a free resin acid, e.g., rosin, and, if desired, an ammonium salt of a water-insoluble fatty acid, e.g., oleic acid, with or without the addition of a water-soluble organic solvent, a plasticiser, e.g., castor oil, and a disinfectant or healing agent, e.g., cresol. These products dry readily and become resistant to weathering, but are readily removable from the wool by the use of sodium carbonate solution etc. S. S. WOOLF.

Production of zinc-white pigments. C. R. BERINGER (B.P. 312,648, 29.5.29. Hung., 29.5.28).—Metallic zinc, or a metal or alloy containing zinc, is heated, at the outset by external firing and subsequently by the heat of reaction alone, in a current of oxidising gas in a continuously or intermittently revolving, cylindrical, reverberatory furnace. Improved colour of the final zinc-white due to absence of violent bubbling in the zinc bath, and increased life of fireclay furnace linings are claimed.

S. S. WOOLF.

Manufacture of "satin white." RILEY'S CHEMICALS & COLOURS, LTD., and C. H. RILEY (B.P. 327,848, 28.3.29).—The violence of the reaction between lime and aluminium sulphate is retarded by addition of sodium sulphate to either ingredient before mixing or during the admixture, a flocculent precipitate of "satin white" being formed. In a preferred process, 1 pt. of anhydrous sodium sulphate in the form of salt cake is added to 3–25 pts. of molten aluminium sulphate containing 17–18% Al_2O_3 , and lime is subsequently added.

S. S. WOOLF.

Cellulose acetate [plastic] composition. W. GUMP, Assr. to J. C. DEHLS and L. STEIN (U.S.P. 1,740,854, 24.12.29. Appl., 30.9.27).—*p*-Hydroxy-*tert*-butylbenzene, m.p. 98° , b.p. 236 – 238° , its 3-bromo-derivative, m.p. 50° , and 3:5-dibromo-derivative, m.p. 78° , are used as plasticisers for cellulose acetate.

C. HOLLINS.

Plastic compositions containing cellulose derivatives. E. I. DU PONT DE NEMOURS & Co. (B.P. 312,604, 3.4.29. U.S., 28.5.28).—Such compositions containing unsymmetrical diarylethanes, e.g., *as*-diphenylethane, as plasticisers are claimed.

S. S. WOOLF.

Manufacture of cellulose ether-oxyn lacquers and application of the products obtained. I. G. FARBENIND. A.-G. (B.P. 307,361, 5.3.29. Ger., 5.3.28).—Lacquers produced by incorporating solutions of an oxyn and a cellulose ether (plasticisers, resins, etc. being added as required) show a high stability to heat and low inflammability.

E. LEWKOWITSCH.

Synthetic resin varnishes and synthetic resins. IMPERIAL CHEM. INDUSTRIES, LTD., H. H. MORGAN, and A. A. DRUMMOND (B.P. 329,313, 9.11.28).—Phenolic compounds, formaldehyde, and a fatty acid ester, e.g., raw or treated tung oil, linseed oil, fish oil, or olive oil, are caused to react under heat in the presence of a volatile organic solvent and a catalyst, driers being added at any stage. The solvent is then sufficiently removed to yield a substantially solid body, which may be dissolved in a suitable solvent to produce varnishes. The phenol may first be condensed with the formaldehyde in the presence of the solvent and an alkaline catalyst, and the product then acidified with a weak acid, e.g., boric acid, and distilled until the b.p. indicates that all the water has been removed; the fatty acid ester is then added and distillation continued until a drop of the solution dries clear. Dyes or pigments may be incorporated.

S. S. WOOLF.

Manufacture of [resinous] condensation products (A, B) of phenols [and aldehydes], (C) of urea and formaldehyde. (A) O. A. CHERRY and F. KURATH,

(B, C) O. A. CHERRY, Assrs. to ECONOMY FUSE & MANUF. Co. (U.S.P. 1,737,916—8, 3.12.29. Appl., [A] 19.2.28, [B] 21.4.26, [C] 23.5.27).—(A) Furfuramide or hydrobenzamide is used as condensing agent for production of phenol-formaldehyde resins by a one- or two-stage process, intended for insulating compositions. (B) Phenol and formaldehyde are condensed in presence of 2–12% of formic acid. (C) "Mono- and di-methylolureas" [hydroxymethylcarbamides] are obtained from carbamide and formaldehyde by maintaining a p_H of about 5 by means of buffer salts.

C. HOLLINS.

Hardening of condensation products from phenols and aldehydes. F. SCHMIDT (B.P. 316,858, 4.12.28. Ger., 3.8.28. Addn. to B.P. 304,648; B., 1930, 69).—The products of the prior patent are improved by the addition of multivalent alcohols or their esters (especially those containing free hydroxyl groups), which retard hardening and remove the turbidity from phenol-formaldehyde resins.

E. LEWKOWITSCH.

Manufacture of synthetic resin. E. E. NOVOTNY and C. J. ROMIEUX, Assrs. to J. S. STOKES (U.S.P. 1,738,310, 3.12.29. Appl., 20.9.23).—Resin-forming mixtures containing phenol, e.g., equimolecular proportions of phenol and benzaldehyde, are caused to react, if desired in presence of acid, basic, or neutral catalysts under a fractionating column so adjusted as to remove water of condensation as fast as it is formed. The reaction mixture is thereby maintained at a higher temperature than would be possible under an ordinary reflux condenser and the reaction proceeds further. The catalysts are removed from the fusible condensation product, which is then rendered infusible by addition of methylene-containing hardening agents, e.g., hexamethylenetetramine.

S. S. WOOLF.

Resinous condensation product from lignin and furfuraldehyde. M. PHILLIPS (U.S.P. 1,750,903, 18.3.30. Appl., 6.4.29).—Lignin and furfuraldehyde heated at 100 – 150° in the presence of a small amount of hydrochloric acid condense to form a brittle, black, hard resin.

E. LEWKOWITSCH.

Manufacture of artificial masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,713, 29.9.28. Cf. B.P. 327,417; B., 1930, 625).—Bleached montan wax or oxidation products thereof consisting mainly of free fatty acids of high mol. wt., e.g., montanic acids, are esterified alone or in admixture with water or high-boiling solvents, e.g., naphthalene, which are subsequently removed, with resinous organic condensation products containing hydroxyl groups, but free from hydroaromatic or aromatic sulphur compounds. The esterification may take place simultaneously with the production of the resinous material. Accelerators, e.g., sodium bisulphate, boric acid, or anhydrous zinc chloride may be used. Fillers, dyes, gums, plasticisers, etc. may be incorporated, and the product is pressed and heated, preferably after comminution. [Stat. ref.]

S. S. WOOLF.

Manufacture of moulded articles. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, C. G. GARTON, and A. E. SMITH (B.P. 329,273, 11.2.29).—The felted, sheeted, fibrous mass which is fed to the final presses

or calendars in the manufacture of pressboard is used as basic material for the blank in the moulding of thin-walled articles. The wet mass is soaked in alcohol, removed, and immediately immersed in a bonding material, *e.g.*, a synthetic resin. The impregnated mass is allowed to dry and moulded under heat and pressure.

S. S. WOOLF.

Production of an ornamental metallic surface on a resinous moulded composition or fibre. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. EATON (B.P. 301,432, 29.11.28. U.S., 29.11.27).—The inner surface of the mould is sprinkled with powdered metal, *e.g.*, bronze powder, before the composition is filled in and hot-pressed.

E. LEWKOWITSCH.

Bituminous paint. R. MEZGER, Assr. to P. LECHLER (U.S.P. 1,762,069, 3.6.30. Appl., 6.3.25. Ger., 26.4.24).—See B.P. 231,411; B., 1925, 461.

Method and apparatus for making ink [from hydrocarbons]. A. L. MILLER, Assr. to AULT & WIBORG Co. (U.S.P. 1,765,991, 24.6.30. Appl., 28.2.27).—See B.P. 286,207; B., 1929, 196.

Apparatus for preparation of Indian ink and the like. R. H. M. GREEN (B.P. 330,830, 19.8.29).

Atomisers (B.P. 330,022).—See I. **Fabric coating composition** (B.P. 303,368).—See II. **Binding agent** (B.P. 328,908).—See III. **Azo pigments** (B.P. 303,838). **Azo lakes** (B.P. 328,292).—See IV. **Cellulose esters or ethers** (B.P. 328,934).—See V. **Chromium-green** (B.P. 305,588). **Siliceous minerals** (U.S.P. 1,748,989). **Titanium oxides** (B.P. 307,881).—See VII. **Coating asbestos-cement plates** (B.P. 327,871).—See IX. **Insulation of wires etc.** (B.P. 330,552).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Inner properties of estate rubber from Java and South Sumatra. R. RIEBL (Arch. Rubbercultuur, 1930, 14, [5]; Med. Proefstat. Rubber, 1930, [57], 223—250).—As the routine tests at the Buitenzorg laboratory relate to rubber prepared on a limited number of estates, three samples taken at random from the product of different days have been drawn from 293 estates, the total number of samples being 963. The proportion of the samples showing a vulcanisation time within 10 and 20 min. of the average (110 min.) ranged from 72% to 93%, and from 93% to 100%, respectively, for various districts. No instance was found of abnormally low tensile strength, *viz.*, more than 10% below the average, the average itself being 1.5 kg./mm.² Slope, viscosity, and plasticity were also measured; the first-named was very constant, whilst the last was distinctly higher, both for crêpe and smoked sheet rubber, than the average observed in 1925. The results of the investigation generally corresponded very closely with the average figures from the routine tests during 1928 and 1929; the restricted number of estates submitting samples for routine testing may consequently be regarded as fairly representative for Java plantation rubber. No sign was detected of deterioration in any respect. (Cf. De Vries and Riebl, B. 1930, 470.)

D. F. TWISS.

Volume increase in vulcanisation [of rubber].

R. DITMAR and C. H. PREUSSE (Chem.-Ztg., 1930, 54, 501—502).—Raw rubber submitted in a mould to the temperature conditions of vulcanisation softens, but does not increase in volume. In the presence of sulphur, however, the volume increases with consequent extrusion of the surplus. The common assumption that the density of the vulcanisate is the same as that of the original mixture is incorrect. Results are given of experiments showing the increase in volume when vulcanised in a spherical mould 2 cm. in diam. of mixtures containing varying proportions of carbon black, whiting, and talc.

D. F. TWISS.

Compressibility of rubber. L. H. ADAMS and R. E. GIBSON (J. Wash. Acad. Sci., 1930, 20, 213—223).—Using a cylinder of rubber, vol. about 20 c.c., immersed in a liquid inside a steel cylinder, applying pressure by means of a piston, and making frequent calibrations with a substance of known compressibility, *e.g.*, steel, measurement was made of the compressibility of rubber at intervals of 1000 bars from 12,000 bars downwards. The compressibility curve for soft rubber resembles that of a liquid such as amyl alcohol and shows little in common with that of a solid; the curve for vulcanite, although different from that for a solid such as sodium chloride, is more like that of a compressible solid than of a liquid and is similar to that of potassium. Soft rubber is almost twice as compressible as vulcanite at atmospheric pressure, but the difference is greatly reduced at pressures above 5000 bars. The large proportion of sulphur in vulcanite has a decided influence on its compressibility, lowering it at the lower pressures and keeping it large at higher pressures. A piece of vulcanite of normal volume 100 c.c. (containing 27.4% by wt. of sulphur) would become 85.4 c.c. at 12,000 bars, whereas a piece of soft rubber containing 10% of sulphur would decrease from 100 c.c. to 82.8 c.c.

D. F. TWISS.

Determination of traces of copper in [rubber] materials. A. RUTHING (Chem.-Ztg., 1930, 54, 403).—Methods for the colorimetric determination of copper (less than 0.005%) in unrubbed and rubbered materials prepared by cold vulcanisation with sulphur chloride are described.

E. H. SHARPLES.

Colour lakes. CLAYTON. Gutta-percha resins. STOCK.—See XIII.

PATENTS.

Manufacture of rubber or of polymerisation products of diolefines, which are plastic and/or elastic. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 329,969, 27.2.29).—Plastic and/or elastic polymerisation products with exceptional stability on storage are obtained from diolefines, such as isoprene or butadiene, by adding a dissolved or emulsified anti-ager to the latex or latex-like products from which the polymerised material is to be separated. The anti-ager may be introduced before or during coagulation of the latex-like suspension and in such quantity as to give 0.5—2% in the coagulated product. Compounding ingredients also may be introduced into the latex or latex-like products prior to coagulation.

D. F. TWISS.

Emulsions containing india-rubber. COLAS PRODUCTS, LTD., L. G. GABRIEL, and J. F. BLOTT (B.P.

329,965, 27.2.29).—A mixture of oil, *e.g.*, linseed oil, or molten or liquid bituminous material and rubber latex is subjected to agitation in a mixing apparatus preferably of the colloid-mill type. A preferred mixture is one of bitumen, artificially produced from petroleum, with sufficient latex to introduce 25% by wt. of rubber, calculated on the emulsion. No additional emulsifying agent is necessary, emulsification being effected solely by the latex, but a small proportion of stabilising agent, such as alkali caseinate, may be added to the finished emulsion (cf. B.P. 254,004; B., 1926, 748).

D. F. TWISS.

Manufacture of rubber goods. SOC. ITAL. PIRELLI (B.P. 301,476, 30.11.28. Italy, 30.11.27).—A cylindrical core, heated internally by hot water or electrically, is caused to travel through latex, which may have been preserved and/or compounded, and emerge therefrom coated with a layer of compact coagulated rubber. The thickness of the deposit may be regulated by adjusting the temperature and surface speed of the core and the depth of the latex. In order to prevent thickening or coagulation except at the surface of the core, a coiled pipe through which cold water is circulated may be provided in the latex vessel, the bottom of which may be formed by a resilient membrane with an aperture permitting the liquid-tight passage of the core (cf. B.P. 284,608 and 292,964; B., 1929, 612; 1930, 205).

D. F. TWISS.

Cementing methods and substances. [Attaching rubber to other surfaces.] RUBBER LATEX RES. CORP. (B.P. 309,168, 18.3.29. U.S., 6.4.28).—Laminated articles comprising a base, *e.g.*, metal, Bakelite, wood, and a mass of vulcanised rubber united thereto by means of an intermediate film consisting of a hardened or coagulated water-resistant proteid and rubber are claimed. Heat-coagulable proteins, *e.g.*, egg-albumin, blood, hæmoglobin, are preferred, but dextrin, glue, etc., subsequently treated with formaldehyde, or gelatin or isinglass in dichromate solution, subsequently exposed to light, may be used. After the protein has been rendered water-resistant *in situ*, the vulcanisable rubber mass is superimposed and the assembled article is vulcanised, whereby the rubber of the mass and the film are simultaneously vulcanised and mutually integrated.

S. S. WOOLF.

Oxidisable organic compounds. [Anti-agers for rubber.] GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 316,251, 19.4.29. U.S., 26.7.28).—Alkyl-naphthylamines are used as anti-agers for rubber; examples are *n*-butyl- β -naphthylamine, b.p. 178—183°/4—5 mm., the α -isomeride, heptylnaphthylamines, etc.

C. HOLLINS.

Drying apparatus (U.S.P. 1,750,813).—See I. Rubberised fabric (B.P. 303,368).—See II. Artificial silk products (B.P. 328,627).—See V. Treatment of water-absorbent materials (B.P. 326,940).—See VI. Insulating materials (B.P. 329,683).—See XI.

XV.—LEATHER; GLUE.

Acido-proteolytic enzymes in the tanning industry. C. GORINI (Zymologica, 1930, 5, 41—46).—The use of dung or glandular (pancreatic) enzymes for

the treatment of hides after unhairing may be advantageously replaced by that of the author's acido-proteolytic enzymes, which are capable of carrying out digestion in an acid medium.

T. H. POPE.

Tanning and dyeing of furs. A. DOHOGNE (Boll. Off. Staz. Sperim. Ind. Pelli, 1930, 8, 168—171).—After steeping for 1—2 days, with several changes of the water, rabbit skins are drained, centrifuged, defleshed, and, tanned. In the Ghent district non-sulphited quebracho is mostly used for tanning, but in Leipzig the skins are pickled and treated on the flesh side with fish oil. Use is sometimes made of formaldehyde, "chrome," alum, oil, synthetic, or combined tanning. The tanned skins are greased, dried at 35°, subjected to fulling etc., defatted, mordanted with potassium dichromate, copper sulphate, ferrous sulphate, or chrome or potash alum, and decolorised by alkaline hydrogen peroxide, hyposulphite, etc., often in presence of a catalyst. Dyeing, either of the whole skins or of the tips only, is mostly effected by means of phenols, amines, phenylamines, such as *p*-phenylenediamine etc., which are capable of giving, on oxidation, colouring matters absorbed by the skins. For dyeing to imitate beaver, use is made of a dye bath containing various Ursol dyes, ammonia, and hydrogen peroxide. The dyed skins are washed in running water, centrifuged, and dressed on the flesh side with a solution of sodium chloride, glycerin, and yolk of egg.

T. H. POPE.

Comparison of the quinhydrone and hydrogen electrodes in solutions containing tannin. E. L. WALLACE and J. BEEK, JUN. (Bur. Stand. J. Res., 1930, 4, 737—745).—Measurements of the p_H of solutions containing chestnut and quebracho tanning extracts and of systems consisting of leather and water in equilibrium have been made with the hydrogen and quinhydrone electrodes and the results are compared graphically. In the leather-water systems the value of $p_{H(H)}$ is slightly less than that of $p_{H(Q)}$, and although no general expression can be formulated for converting one series of results into the other, corrections can be applied to the quinhydrone electrode readings to obtain the corresponding hydrogen electrode readings in certain cases where the nature of the solution is known. In solutions of powdered chestnut-wood extract the values of $p_{H(H)}$ decreased in a smooth curve convex to the axis of composition with increasing tannin content, whereas the values of $p_{H(Q)}$ increased along a curve concave to the same axis under the same conditions. Similar results were obtained for solutions of solid, ordinary quebracho extract, but the differences were more marked, $p_{H(H)}$ decreasing from 4.95 with 0.25% of tannin to 4.75 with 7% of tannin and $p_{H(Q)}$ increasing from 4.1 to 5.25 in the same range.

A. R. POWELL.

Improved Kubelka-Němec sedimentation method of determining the insoluble matter in tannin analysis. V. NĚMEC and E. KUDLAČEK (Collegium, 1930, 72—74; cf. B., 1929, 991).—An inverted bottle (diam. 9 cm.) from which the bottom has been cut away is fitted with a rubber stopper through which pass a wide and a narrow glass tube. The upper end of the narrow tube is bent through 180°, and its lower end is fitted with a rubber connexion, clip, and delivery tube.

The upper end of the wide tube is 1.0—1.1 cm. above the open end of the other tube. The analytical tannin solution is poured into the inverted bottle (any excess runs out through the wide tube), and the bottle is covered with a glass; after being set aside for 4—5 min. particles $>1\mu$ will have settled below the mouth of the narrow tube. The supernatant liquor is then withdrawn through the narrow tube and an aliquot portion evaporated to dryness.

D. WOODROFFE.

Action of water on vegetable-tanned leather.
II. Resistance of the collagen-tannin compound to hydrolysis on long washing. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1930, 25, 173—182; cf. B., 1929, 567).—Vegetable-tanned calfskin samples were extracted first with chloroform, then in a Wilson-Kern extractor with distilled water for 1—121 days. Water, hide substance, and ash were determined in the original and the dried, extracted leathers, and from these results and the weight of the leathers before and after extraction the amount of tannin combined with 100 g. of hide substance was calculated. The amount of residual tannin calculated by means of the residual weight of leather was less than that obtained by the analysis of the residue. After 65 days' washing, the amount of material removed thereby was so small that the residue must have been a tannin-collagen compound which was hydrolysed only to a negligible extent. In similar tests with vegetable-tanned hide powder similar results were obtained, but equilibrium was reached more quickly. The quebracho-collagen compound was more resistant to hydrolysis than the oak-collagen, whilst the gambier-collagen was much less resistant. The same initial tannin content of the leather was obtained when the pelt or powder was tanned with a 2% solution as with a 4% solution of tannin, but less resistance to hydrolysis was shown by the product tanned in the stronger solution. The initial ratio of tannin to hide substance was increased by increasing the time of tanning. Hide substance combines irreversibly with a limited amount of tannin, after which it combines with more tannin, which is removable by washing, the amount depending on the amount originally fixed by the leather and on the kind of tanning material used.

D. WOODROFFE.

Skin- and bone-glues. H. MAIER-BODE (Kunst-dünger- u. Leim-Ind., 1929, 26, 327—329, 339—341, 351—354; Chem. Zentr., 1930, i, 927).—It is confirmed that the viscosity of fresh glue liquor is smaller than that of a sol of equal concentration prepared from the solid material; it is considered that, on drying, reaggregation of the glutin takes place. Skin and bone glues are conveniently tested by determining the viscosity of a 17.75 or 20% solution at 30° and 40°. A modification of Ostwald and Köhler's test differentiates skin glue from bone glue.

A. A. ELDRIDGE.

Colour lakes. CLAYTON.—See XIII.

PATENTS.

Imparting a blue colour to leather polish. S. H. DOWNINGTON (B.P. 330,014, 7.3.29).—Ferric ferrocyanide is well stirred into the usual mixture of oils and fats (except linseed oil) in the liquid or semi-liquid state. [Stat. ref.]

H. ROYAL-DAWSON.

Depilating hides and skins. M. BERGMANN (U.S.P. 1,765,199, 17.6.30. Appl., 27.7.25. Ger., 12.2.25).—See B.P. 247,826; B., 1926, 377.

Dehairing hides and skins and preparing them for tanning. C. J. M. M. LE PETIT, Assr. to RÖHM & HAAS Co. (U.S.P. 1,767,536, 24.6.30. Appl., 21.5.26. Fr., 18.4.25).—See B.P. 250,907; B., 1926, 1022.

Preparation of gelatin. A. PANSKY (U.S.P. 1,761,362, 3.6.30. Appl., 14.12.23. Fr., 24.11.23).—See B.P. 221,702; B., 1924, 956.

Adhesive agent (B.P. 328,908).—See III. **Bleaching of skins etc.** (B.P. 310,030). **Treatment of fur** (B.P. 330,598).—See VI.

XVI.—AGRICULTURE.

"Build-up and break-down" of soil zeolites as influenced by reaction. P. S. BURGESS (Arizona Agric. Exp. Sta. Tech. Bull., 1929, No. 28, 101—135).—The base-exchange capacity of many arable soils can be increased by treatment with solutions of alkalis or alkali salts, the increase being closely proportional to the hydroxyl-ion concentration of the solution. The zeolitic complex can be partly (1.2—18.4%) broken down by leaching with relatively large amounts of dilute solutions of organic acids (including carbonic). Appreciable amounts of hydrogen zeolite were formed. The break-down was more rapid and complete if solutions of aluminium chloride or sulphate were used. It is probable that active build-up of soil zeolites takes place largely in the calcareous soils of arid and semi-arid regions where alkaline soil solutions prevail, and that disintegration is more common in the more or less acid soils of humid regions.

CHEMICAL ABSTRACTS.

Comparison of soil-liming materials. A. T. WIANCKO, G. P. WALKER, and S. D. CONNER (Indiana Agric. Exp. Sta. Bull., 1929, No. 329, 3—24).—Ground limestone and marl are preferred for Indiana sandy loam and silt loam. The coarse limestone particles have a definite, although delayed, action. Moderate applications affect the acidity of the surface layer only.

CHEMICAL ABSTRACTS.

Impoverishment of forest soils by use of the litter. A. NĚMEC (Forstarchiv, 1929, 497—503; Bied. Zentr., 1930, 59, 257—258).—Low proportions of nutrients in many forest soils are attributed to the annual removal of mineral matter in the forest litter. In this respect the available potash is least affected.

A. G. POLLARD.

Phosphoric acid content of forest humus soils. A. NĚMEC (Forstwiss. Zentr., 1929, 721—732; Bied. Zentr., 1930, 59, 247—248).—The citric-soluble phosphate content of forest soils is generally smaller than that of similar arable soils. The humus layer of forest soils is the most abundant source of easily soluble phosphate, and in lower layers there is a general parallelism between the citric solubility of phosphates and the humus content. The humus layer under pines has a relatively low citric-soluble phosphate content. The impoverishment of forest soils by removal of the *débris* of leaves etc. for litter is emphasised.

A. G. POLLARD.

Nutrient contents of Rhineland soils. G. HAGER, (Landw. z. Rheinprovinz, 1929, No. 47; Bied. Zentr. 1930, 59, 256—257).—A survey of Rhineland soils is recorded. The productivity of soils is not governed by the nutrient content alone, but is dependent on many other factors, notably the proportions of clay and humus, tilth, soil depth, and climatic conditions. With due consideration of these factors, the Neubauer test gives valuable information as to soil fertility.

A. G. POLLARD.

Determination of the nutrient requirement and inoculability of soils by Niklas' *Azotobacter* method. L. KREYBIG (Forts. landw., 1929, 4, 12; Bied. Zentr., 1930, 59, 253—256).—Growth of *Azotobacter* in soils leads to increased citric solubility of the phosphates. In soils deficient in phosphate Rhenania phosphate increased the growth of *Azotobacter* to a greater extent than did superphosphate, particularly in humus soils with low exchange acidity. The application of phosphatic fertilisers increased the nitrogen fixation in phosphate-deficient soils. The *Azotobacter* method is considered satisfactory for the practical determination of the phosphate requirement of soils.

A. G. POLLARD.

Determination of nitrogen in soils by the Mitscherlich vegetation method. A. Y. SAMOILOVA (Udobr. Urozhai, 1929, 355—358).—Mitscherlich's equation, $\log(A - Y) = \log A - cx$, where Y is the yield, A the possible maximum yield obtained with an optimum quantity of the factor studied, c the activity coefficient of the nutrient factor, and x the amount of nutrient added, was employed. In determining the nitrogen resources of the soil the use of a coefficient calculated from experimental data gives more trustworthy results than that of Mitscherlich's coefficient 0.122. The establishment of the working formulæ is described.

CHEMICAL ABSTRACTS.

"Kalkammonsalpeter." O. NOLTE and H. MÜNZBERG (Mitt. Deut. landw. Ges., 1930, 45, 37; Bied. Zentr., 1930, 59, 258—259).—Comparison is made of the efficiency of "Kalkammonsalpeter," ammonium sulphate, and sodium nitrate in field trials. In dry seasons sodium nitrate was particularly effective, but in wet summers, when leaching losses were considerable, ammonium sulphate proved the more profitable. "Kalkammonsalpeter" is preferable to either of the above for cereals and root crops.

A. G. POLLARD.

Changes in soil reaction produced by ammonium, potash, and potash ammonium superphosphate GERLACH (Superphosphat, 1929, 5, 282; Bied. Zentr., 1930, 59, 248—249).—The increasing acidity of many German soils is more definitely attributable to the normal processes of cropping and leaching than to the use of physiologically acid fertilisers. Systematic liming or marling is preferable to the withholding of valuable, if slightly acid, fertilisers.

A. G. POLLARD.

Influence of roasting raw phosphates on the availability of phosphoric acid. N. D. SMIRNOV (Udobr. Urozhai, 1929, 359—363).—High-grade phosphate (23—30% P_2O_5) showed a slight increase of citrate-soluble phosphorus on roasting. Pot experiments with

oats on podsol and degraded chernozem revealed no superiority.

CHEMICAL ABSTRACTS.

Obtaining a concentrated triple fertiliser. N. E. PESTOV and E. A. KALABEKOVA (Udobr. Urozhai, 1929, No. 7, 421—425).—For the preparation of a fertiliser containing nitrogen, phosphorus, and potassium, but little chlorine, the optimal mol. ratio of phosphoric acid:potassium chloride:ammonia is 1:1.25:1. This gives a salt of composition KH_2PO_4 78.63, $NH_4H_2PO_4$ 19.73, KCl 1.64%.

CHEMICAL ABSTRACTS.

Apatite and nepheline rock of the Khibin Mts. as a direct fertiliser. I. P. SOMOV (Udobr. Urozhai, 1929, 412—418).—Soils of pH 4.4—4.1 responded, and of pH 5.3—5.2 did not respond well, to treatment with apatite. Addition of ammonium sulphate rendered the apatite more effective. The nepheline is a satisfactory source of potassium.

CHEMICAL ABSTRACTS.

Fertilising action of bone meal. E. TRUNINGER and F. KELLER (Landw. Jahrb. Schweiz, 1929, 43, 931—945; Chem. Zentr., 1930, i, 1028).—Bone meal, preferably defatted, gives good results only on acid soil. With one application raw bone meal gave 0.33, or with a double quantity 0.5, of the increased yield afforded by superphosphate. The activity depends largely on the fineness of division. The value of bone-meal nitrogen is estimated to be 50% of that of nitrate nitrogen.

A. A. ELDRIDGE.

Conditions of application and the effect of phosphate on chernozem. VIII. Reversion. M. A. EGOROV (Udobr. Urozhai, 1929, 401—412).—The sedimentation of soil extracts on treatment with calcium carbonate followed by carbon dioxide, with sodium phosphate and calcium salts, was studied. Various types of lime do not act on soils in the same way.

CHEMICAL ABSTRACTS.

Effect of fertilisers on the quality of the juices of sugar cane. R. F. GARCIA (Ann. Rep. Insular Exp. Sta. Porto Rico, 1929, 68—88).—In general, nitrogenous fertilisers gave the highest yields. Soluble manganese salts exercised a negligible effect.

CHEMICAL ABSTRACTS.

Effect of nitrogenous fertilisers on pastures. O. NOLTE, H. MÜNZBERG, and H. KOCH (Mitt. Deut. landw. Ges., 1929, 44, 385; Bied. Zentr., 1930, 59, 263).—Results of meat and milk trials on fertilised pastures are recorded.

A. G. POLLARD.

Comparison of the fertilising value of manure and mineral fertilisers. S. V. SHTSCHERBA (Udobr. Urozhai, 1929, 418—420).—Experiments with sugar beet and potatoes are recorded. Values for the starch content of potatoes are: without manure or mineral fertilisers 14.4, with manure 14.6, with mineral fertilisers 18.45, with both 18.8%.

CHEMICAL ABSTRACTS.

Composting barnyard manure with sulphur and rock phosphate. W. G. FRIEDMANN (Georgia Agric. Exp. Sta. Bull., 1929, No. 154, 1—14).—Untreated composts suffered a greater loss of nitrogen than did those containing sulphur; with 2% S only 10% or less of the nitrogen was lost. Superphosphate was more effective than sulphur in preventing loss of nitrogen from liquid

manures. Part of the phosphorus of a manure-rock phosphate-sulphur mixture becomes available during composting.

CHEMICAL ABSTRACTS.

Manuring and crop quality in root crops. KLEE-BERGER (Superphosphat, 1929, 5, 74—76; Bied. Zentr., 1930, 59, 259—260).—The effect of unbalanced fertilisation on the quality of sugar beet and potatoes is examined. In this respect nitrogenous fertilisers are of first importance. The use of phosphate fertilisers in amounts based on Neubauer trials may be unsatisfactory, since this method of examination allows of no consideration of quality. There is no foundation for the opinion that phosphate fertilisers are of minor importance for potatoes.

A. G. POLLARD.

Manurial trials with potassium ammonium superphosphate on root crops. DENSCH (Superphosphat, 1929, 5, 53; Bied. Zentr., 1930, 59, 259).—The efficiency of the nitrogen of "Kali-ammon-superphosphat" is similar to that of the simpler nitrogenous materials, and the fertiliser proved suitable for use on acid soils and for acid-sensitive plants.

A. G. POLLARD.

Fineness of ground sulphur sold for dusting and spraying. L. R. STREETER and W. H. RANKIN (New York State Agric. Exp. Sta. Tech. Bull. No. 160, 1930, 16 pp.).—A consideration of methods, including a description of a micro-projection method, of determining the degree of subdivision of ground sulphur, together with details of an examination of five commercial brands.

E. HOLMES.

Supplements for copper fungicides. E. B. HOLLAND, C. O. DUNBAR, and G. M. GILLIGAN (Mass. Agric. Exp. Sta. Bull., 1929, No. 252, 94—112).—The efficiency of insoluble copper fungicides depends largely on the degree of dispersion and other physical characteristics. "Soluble copper" is not a criterion, since various organic compounds produce filterable copper of low activity. The efficiency of the spray is increased by wheat flour, gelatin, soap, or tannic acid.

CHEMICAL ABSTRACTS.

Effect on plants of cyanide fumigation following spraying with Bordeaux mixture. O. BUTLER and R. R. JENKINS (Phytopath., 1930, 20, 419—429).—Injury to foliage following the fumigation with hydrocyanic acid of plants previously sprayed with Bordeaux mixture depends on the proportions of lime and copper sulphate used in preparing the latter. Exposure of various Bordeaux mixtures to hydrocyanic acid showed that no cupric cyanide was formed when the ratio of copper sulphate to quicklime used was 1:0.2 and in negligible amounts only when the ratio was 1:>4. In a 1:0.2 mixture insoluble cuprous cyanide was formed, and in a 1:6 mixture a soluble double cyanide. In mixtures between 1:1 and 1:6 the amount of cupric cyanide formed decreased and that of the double cyanide increased with increasing proportions of lime used. The double cyanide is injurious to foliage. A neutral Bordeaux mixture is the only type suitable for use when cyanide fumigation is to follow.

A. G. POLLARD.

Tank-mixture method of using oil spray. R. H. SMITH (J. Econ. Entom., 1930, 23, 376—382).—Uniform pine oil-water mixtures may be maintained in spray tanks and in the spray by the use of suitable paddle

agitators revolving at 200—250 r.p.m. More oil is retained by sprayed surfaces at a distance from the nozzle than close to it. The quantity of oil deposited is largely determined by the nature and quantity of the emulsifier used, soap causing a smaller deposit than other commonly used materials. The effect of an emulsifier in oil-water mixtures cannot be judged by its effect in other spray mixtures.

A. G. POLLARD.

Borax as an insecticide for protecting seed. H. H. SCHWARDT (J. Econ. Entom., 1930, 23, 401—404).—Powdered borax controlled the rice weevil when used at the rate of 10 oz. per bushel of grain, and the four-spotted bean weevil when 20 oz. per bushel of cow peas were used. Treated grain cannot be fed to cattle.

A. G. POLLARD.

Differentiation between good and poor germinative capacity of seeds by chemical means. H. O. PAECH (Diss. Agric.-Chem. Inst. Univ. Breslau, 1929; Bied. Zentr., 1930, 59, 268—270).—The germinative activity of seeds is closely related to their dehydrogenase activity. Of numerous colorimetric methods for the determination of the latter, the most satisfactory is that using *m*-dinitrobenzene. Comparative tests should be made on the same day with freshly-ground material.

A. G. POLLARD.

Seed treatments for the control of barley stripe. C. S. REDDY and L. C. BURNETT (Phytopath, 1930, 20, 367—390).—Results of trials with a number of mercurial fungicides are recorded and discussed.

A. G. POLLARD.

Nitrophosphates. CALCAINI.—See VII. **Drinking water for cattle.** McLACHLAN.—See XXIII.

PATENTS.

Fertilisers. A. A. J. and C. A. A. VILAIN (VILAIN FRÈRES) B.P. 309,175, 2.4.29. Fr., 7.4.28).—The product obtained by treating dolomite with nitric acid is claimed.

H. ROYAL-DAWSON.

Product for promoting plant growth. R. A. BAKER and C. C. CARPENTER (U.S.P. 1,747,281, 18.2.30. Appl., 30.6.27).—The product consists of a mixture of an alkaline-earth carbonate (*e.g.*, limestone or dolomite) and aluminium sulphate.

H. ROYAL-DAWSON.

Freezing soils by means of carbonic acid. H. DEHOTAY (B.P. 308,310, 20.3.29. Belg., 21.3.28).

Production of mixed manure containing phosphoric acid and nitrogen. F. G. LILJENROTH (U.S.P. 1,761,400, 3.6.30. Appl., 23.7.27. Swed., 13.8.26).—See B.P. 275,843; B., 1927, 826.

XVII.—SUGARS; STARCHES; GUMS.

Absorption of atmospheric oxygen by limed cane juice. J. A. AMBLER (Ind. Eng. Chem., 1930, 22, 357—362).—Quantitative studies of the rates of absorption of oxygen by cane and beet juices and pure sugar solutions containing up to 0.06% CaO, aerated under constant conditions at the ordinary temperature, showed that cane tannin and invert sugar undergo oxidation under these conditions, with formation of calcium salts of organic acids and consequent lowering of alkalinity. The rate of oxidation is greater at high than at low alkalinities. It is concluded that under technical conditions of liming and defecation of cane juices considerable

oxidation must occur in view of the high temperatures employed, the common use of air for mixing the limed juices, and the high alkalinities which occur locally before the lime is uniformly mixed with the juice.

J. H. LANE.

Comparison of some white-sugar methods.

H. A. FABER (Ind. Eng. Chem., 1930, 22, 366—367).—In American beet-sugar factories fully 85% of the available sugar in the thick juice is usually obtained as standard white granulated without remelting. This is due to the use of live steam in the vacuum-pan coils. In Javan and Continental European white-sugar factories the vacuum pans are heated by exhaust steam or vapour from the first effect of the evaporators, and in consequence circulation of the massecuite is sluggish, crystal growth is retarded, and water has often to be drawn into the pan to remedy supersaturation and false grain formation. A soft type of crystal is produced, centrifugal work is inefficient, and the mother-liquors are highly viscous. Only about 15% of the total available sugar is obtained as white sugar without remelting; to recover the whole, four boilings are necessary, as compared with two in American beet-sugar factories. The author strongly advocates the use of live steam for pan boiling in cane-sugar factories, for the production of white sugar.

J. H. LANE.

Determination of polarisation, ash, alkalinity, p_H value, and colour of molasses with one weighing. V. KONN (Z. Zuckerind. Czechoslov., 1930, 54, 325—331).—For factory analyses of beet molasses the author proposes to weigh out 26 g., make up to 200 c.c. with neutral distilled water, and use aliquot portions for the various determinations, viz., 20 c.c. for alkalinity by titration, 10 c.c. for p_H value by Clark's method, 20 c.c. diluted to 200 c.c. for colour degrees (Stammer), 10.4 c.c. diluted to 100 c.c. for carbonate ash by Sandera's conductimetric method, and 100 c.c. clarified with 3.5 g. of dry basic lead acetate for polarisation. With ordinary beet-factory or refinery molasses the results obtained agree satisfactorily with those found by the conventional Czechoslovakian methods, according to which the carbonate ash is determined by incineration. Rather wide discrepancies in ash values, up to 1.13%, were found, however, with molasses 40 years old and also with molasses containing much raffinose, and with a sample of Javan cane molasses. In general, with all these molasses the carbonate ash was only about four fifths of the sulphated ash, even after the usual subtraction of one tenth from the latter.

J. H. LANE.

Influence of the reaction on the technique of the preparation of lactose, condensed milk, and ice-cream. P. PARISI (Giorn. Chim. Ind. Appl., 1930, 12, 225—235).—The "sandiness" of condensed milk and of ice-cream and the low yield in lactose from concentrated whey depend essentially on crystallisation of the lactose, which is closely connected with the phenomenon of mutarotation and this with the temperature and hydrogen- and hydroxyl-ion concentrations of the solution. The velocity of transformation of the β -stereoisomeride of lactose into the α -form at a given temperature and a given p_H is the resultant of the separate velocities due to the hydrogen and hydroxyl ions. The actions

of these ions are represented by geometrical progressions having as exponents the p_H and p_{OH} values, the velocity coefficient k_2 being given by $ax^{p_H} + by^{p_{OH}}$. The logarithm of the common ratio of the geometrical progression as a function of the hydroxyl ions is seven times as great as that of the corresponding ratio for the hydrogen ions. The two curves intersect at p_H 6.7—6.8 and the minimum value of k_2 occurs at about p_H 5.0.

T. H. POPE.

Fertilisers and sugar cane. GARCIA.—See XVI.
Fermentation of sugar. TOMODA and KOCHI. **Fermentation of molasses.** TOMODA.—See XVIII.
Lactose from skim milk. NABENHAUER. **Test for sugars in jams.** LEWIS. **Dextrins in food materials.** GROSSFELD and HOLLATZ.—See XIX.

PATENTS.

Manufacture of sugars and polyhydric alcohols. J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 327,193, 3.12.28).—In the synthetic production of sugars from formaldehyde or its polymerides by heating with oxide catalysts, greatly increased yields of sugars are obtained by carrying out the reaction in presence of aliphatic alcohols, such as methyl or ethyl alcohol. Thus 500 pts. of a 30% formaldehyde solution, boiled under reflux for 10 hrs. with 500 pts. of methyl alcohol and 10 pts. of lead oxide, yield 140 pts. of syrupy sugars. Mixtures of polyhydric alcohols may be obtained from the synthetic sugar mixtures by catalytic reduction (e.g., with hydrogen in the presence of a platinum catalyst).

J. H. LANE.

Manufacture of sugar solutions suitable for fermentation purposes. J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 330,275, 25.2. and 22.3.29).—Peat is hydrolysed by acids and the separated solution treated, before or after neutralisation, with steam, or aerated while hot. [Stat. ref.] C. RANKEN.

Crystallisation (U.S.P. 1,749,588).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Water for brewing. G. CAPRINO (Zymologica, 1930, 5, 47—55).—In carrying out the decarbonatation of water for brewing, the nature of the raw materials used and of the beer to be produced must be considered. With brown beers, for which long-grown, well-kilned malt is used, a harder water is required than for pale beers. Substitutes such as rice give an alkaline reaction, softer water being hence required. For making beer of the Pilsen type, the liquor should be treated with lime water, this being followed, when necessary, by addition of sufficient hydrochloric or sulphuric acid to neutralise 1 or 2 degrees of hardness. The optimum p_H is 5.2, this corresponding with the most rapid and complete saccharification, the highest extract, and the greatest attenuation. Decarbonatation of the brewing water necessitates considerable increase in the amount of hops used.

T. H. POPE.

Efficiency of alcoholic fermentation of sugar. Y. TOMODA and H. KOCHI (J. Soc. Chem. Ind., Japan, 1930, 33, 75—76 B).—The relation between yeast reproduction and the efficiency of the alcoholic fermenta-

tion of sugar has been studied. The possibility of reducing rather than increasing the yield of alcohol by adding ammonium sulphate to the molasses mash is demonstrated, as more sugar is expended for yeast multiplication in the presence of the salt. The highest fermentation efficiency may be obtained by restricting the yeast multiplication during fermentation as far as possible: a high pitching rate of yeast usually increases the efficiency on account of the reduced reproduction.

E. LEWKOWITSCH.

Production of glycerin by fermentation. IX. Separation of glycerin from fermented waste molasses. Y. TOMODA (J. Soc. Chem. Ind., Japan, 1929, 32, 271—272 B).—The residue from the fermentation of molasses is evaporated to a thick paste from which the glycerin is extracted with 95% alcohol. In this way 70% of the glycerin is recovered. A. A. GOLDBERG.

[Detection of fruit wines in grape wines by means of the] conversion of dibenzylidenesorbitol into sorbitol hexa-acetate. H. JAHR (Z. Unters. Lebensm., 1930, 59, 285—288).—Werder's method (cf. B., 1929, 619) for the detection of cider in wine depends on the isolation of sorbitol as its dibenzylidene derivative. This alcohol is said only to occur in cider. As the hexa-acetyl compound is better crystallised and has a sharp m.p., it is preferred as a means of identification. The benzylidene derivative is decomposed by heating on a water-bath with hydrochloric acid in the presence of benzaldehyde. The benzaldehyde is extracted with ether and the acid removed with zinc oxide. The neutralised solution is then heated with acetic anhydride and zinc chloride. On taking up in water and keeping, the hexa-acetate crystallises out, seeding being sometimes necessary. Sorbitol hexa-acetate has m.p. 98—99°; mannitol hexa-acetate has m.p. 120°. B. W. TOWN.

Removal of cream of tartar from grape juice by freezing storage. M. A. JOSLYN and D. A. TUCKER (Ind. Eng. Chem., 1930, 22, 614—615).—Separation of excess cream of tartar from fresh grape juice by freezing at -18° and subsequent thawing is much more rapid than, and as thorough as, storage at 0°, but in the case of juice previously stored at room temperature and consequently containing less cream of tartar the small crystals produced by freezing redissolve to a considerable extent during thawing. W. J. BOYD.

Evaluation of potable spirits, especially "Kirschwasser" and rum, by determination of the higher oxidation potential by means of chloramine-T. A. MIERMEISTER (Z. Unters. Lebensm., 1930, 59, 289—300).—A method is described for the evaluation of the aromatic constituents to which the bouquet is due. A standard quantity of the spirits is carefully distilled, using a double fraction-head, into seven fractions of equal volume, and each fraction is treated with chloramine-T and set aside. Excess chloramine is back-titrated iodometrically, the chloramine value of the fraction being expressed as the number of c.c. of 0.01N-chloramine-T used up. The sums of the values for the fractions give characteristic numbers which can be used to differentiate genuine rum from the artificial product. The behaviour of alcohol-water mixtures con-

taining artificial aroma constituents, such as benzaldehyde and bitter-almond water, has been investigated.

B. W. TOWN.

The extract question in the liqueur industry. C. LUCKOW (Z. Unters. Lebensm., 1930, 59, 301—304).—It is suggested that only such sweet spirits as contain at least 22 g. of extract per 100 c.c. should be considered as liqueurs.

B. W. TOWN.

Improvement of the quality of alcoholic liquids by treatment with charcoal. C. LUCKOW (Z. Spiritus-ind., 1930, 53, 174—175).—The undesirable flavours and aromas which brandy, rum, arrack, and other alcoholic liquids had acquired by contact with oil, rubber, inferior pitch, etc. were removed by treatment of the liquids with 0.1—1% of charcoal for periods varying from 1 to 6 hrs. If this treatment diminished the characteristic flavour of the liquid, the treated liquid was blended with a further quantity which had not been contaminated. Where the liquid had no characteristic aroma or flavour, the amount of charcoal was increased to 3% and the period extended to 2—3 days.

C. RANKEN.

Acido-proteolytic enzymes and tanning. GORINI.—See XV.

PATENTS.

Treatment of top-fermentation pale ale, mild ale, and stout after the primary fermentation for bottling and draught purposes. H. E. DRYDEN (B.P. 330,327, 19.3.29).—After the conclusion of the primary fermentation an extract of fresh hops is added to the chilled ale or stout, and the liquid filtered to remove primary yeast, proteins, resins, etc. A quantity of priming sugar solution is added and the cold, bright, fermented liquor pitched with a small amount of pure yeast sufficient to ensure the generation of carbon dioxide to produce suitable condition at the end of two weeks.

C. RANKEN.

Dehydration of impure ethyl alcohol. E. RICARD, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,763,722, 17.6.30. Appl., 20.11.25. Belg., 20.11.24).—See F.P. 614,913; B., 1927, 857.

Ketone fuels (F.P. 637,993).—See II. **Sugar solutions** (B.P. 330,275).—See XVII.

XIX.—FOODS.

Atypical corrosion of the starch of American winter barley attacked by *Fusarium roseum* and *Gibberella Saubinetii*. D. STRILCIUC (Z. Unters. Lebensm., 1930, 59, 258—265).—It is established that the starch of the attacked barley is corroded atypically. The manner of the corrosion can be distinguished from that in germinating grain and that produced by moulds. The reason for this atypical corrosion is the local ferment produced by the fungal hyphae. This atypical corrosion can be used for diagnosis of the fungus in groats and flour.

B. W. TOWN.

Colloid chemistry of gluten. II. H. L. B. DE JONG and W. J. KLAAR (Cereal Chem., 1930, 7, 222—244; cf. B., 1930, 31).—The effect of varying alcohol concentration on the viscosity of positive and

negative gliadin sols is examined. The relative viscosity increases up to a concentration of alcohol of 48% by wt., but in higher alcohol concentrations a rapid fall in these values occurs with both positive and negative gliadin. It is suggested that the maximum in the relative viscosity is caused by the formation of an alcohol hydrate layer around the particles, and this is gradually removed in higher alcohol concentrations, causing a fall in the viscosity. The effect of changing hydrogen-ion concentration is to cause separation on both sides of the point of maximum hydration of the aqueous sol, and this separation is a gradual process. Whether an emulsoid will flocculate, separate, or remain in solution by discharging is dependent only on the isoelectric hydration. B. W. TOWN.

Comparative study of the non-gluten constituents of soft and hard wheat flours. H. HALL (Cereal Chem., 1930, 7, 270—279).—In bread-baking the gluten is the "frame-work" of the loaf, but this does not apply in cake-making. A bread batch contains nearly three times the amount of dry gluten that a normal cake batch contains. In cakes the starches are more completely gelatinised than in bread, and the texture of cakes is intimately bound up with this gelatinisation. High viscosities of the gelatinised non-gluten constituents are associated with high swelling powers and high water-imbibing powers. Soft wheat flours contain a greater proportion of non-gluten constituents than hard wheat flours, and have a proportionately higher swelling power. B. W. TOWN.

Comparison between commercially and experimentally milled flours. T. A. PASCOE, R. A. GORTNER, and R. C. SHERWOOD (Cereal Chem., 1930, 7, 195—221).—The saccharogenic activity of experimentally milled flour does not represent the potential activity of the same flour milled commercially, for the commercial product was found to have an activity from one to four times that of the experimental product. This is due to the finer state of division of the commercial article, for the saccharogenic activity of a commercial flour was increased by 35% by grinding for 20 hrs. in a ball-mill. In the baking test the only significant difference was the consistently lower "absorption" of the experimental flours. There was found a linear relation between the total protein content and the loaf volume when the sample of wheat was milled from several different lots of wheat, but not apparently when milled from one sample of wheat. A lyotropic series was demonstrated in the peptising effect of potassium halide salts on flour proteins, potassium bromide showing the highest peptisation, with the least variability. B. W. TOWN.

Possibilities of standardising the granulation test for flour. J. MICKA and K. VRANA (Cereal Chem., 1930, 7, 280—306).—To promote standardisation, it is recommended (a) that the flours should be treated so as to have the same percentage (13.00—13.50%) of moisture; (b) that the method of continuous sifting should be used, one lot of 100 g. sufficing for the whole experiment; (c) to use 20 g. of wheat as a cleaner; (d) to perform the test under standard conditions of temperature and humidity (21°, and 70% R.H.);

(e) that the flour and wheat be at the same temperature as the room (21°); and (f) that the percentage of flour passing through the silks be recorded in a standard manner. B. W. TOWN.

Fat: its determination in wheaten products. C. W. HERD and A. J. AMOS (Cereal Chem., 1930, 7, 251—269).—It is suggested that direct ether extraction gives a measure of the free fat existing in the product, whilst acid hydrolysis gives the total fat content, i.e., free fat + combined fat after its liberation. The major portion of the lipid material is obtained unhydrolysed by the alkaline and alcoholic hydrolysis methods. B. W. TOWN.

Effect of acid potassium tartrate as an ingredient in angel cake. E. GREWE and A. M. CHILD (Cereal Chem., 1930, 7, 245—250).—The change in colour and grain of angel cake by using acid potassium tartrate is largely due to acidity. This ingredient gives a fine-grained, white cake, which otherwise is yellow and coarse-grained. The same effect is produced by using citric, malic, or tartaric acid in place of the tartrate. B. W. TOWN.

Hydrocyanic acid in Lima beans. II. Influence of heat on the toxicity of the beans. S. K. HAGEN (Z. Unters. Lebensm., 1930, 59, 211—216; cf. B., 1928, 689).—Long heating at 125° does not influence the toxicity, i.e., the glucosidic content, of the beans, although it destroys the enzyme present in the bean, which is very specific in its action. B. W. TOWN.

Effect of mineral oil treatment on the composition of milk. M. B. MACDONALD, E. C. ANDES, and F. A. BRIGGS (J. Home Econ., 1930, 22, 213—218).—On addition of mineral oil to remove "onion flavour" the butter fat absorbs less than 1% of oil, and less than 0.5% of butter fat is lost. There is no detectable loss of vitamin-A. CHEMICAL ABSTRACTS.

α - and β -Lactose in milk products. H. C. TROY and P. F. SHARP (J. Dairy Sci., 1930, 13, 140—157).—The rate of change of α - to β -lactose is minimal at p_H 2.0—7.0, but approaches infinity at p_H 0 and 9; the rate of attainment of final solubility or of precipitation of α -lactose is also affected by the p_H . High concentrations of sucrose, as in condensed milk or ice-cream, have no appreciable effect on the equilibrium rate. Skim milk prepared by pressure or centrifugal spray, or by atmospheric or vacuum roll showed a non-crystalline equilibrium mixture of α - and β -forms, whilst skim-milk flakes contained much crystallised α -hydrate. The caking of milk powder and the sandiness of ice-cream are discussed. In the determination of total solids in milk products the residue contains an equilibrium mixture of non-crystalline α - and β -lactose. CHEMICAL ABSTRACTS.

Manufacture of casein and lactose from skim milk. F. P. NABENHAUER (Ind. Eng. Chem., 1930, 22, 54—56).—Methods of manufacture of rennet casein, self-soured casein, artificially-soured casein, and lactose are described. Rennet casein is prepared by adding rennet to the milk and maintaining the mixture at 36° for 15—20 min. The curd is allowed to settle, whey drained off, the curd washed with water, pressed, and

dried. This type of casein requires fresh skim milk, thorough washing and pressing, and rapid drying at a low temperature. Self-soured casein is prepared by allowing spontaneous curdling at 38°. Soft curd forms, and is clumped by direct heating with steam jets. The curd is strained off, washed with cold water, pressed, shredded, dried on trays or in a tunnel drier, and then ground. "Muriatic" casein is prepared by adding sufficient sulphuric or hydrochloric acid just to curdle milk at 41–49°. Curd separates as tough lumps, which are drained off, heated with steam to form one large doughy lump, kneaded, cooled, cut into small chunks, dried, and ground. This gives a hard, flinty product which does not "wet" rapidly. The grain-curd method of producing casein is to add slowly, with stirring, dilute hydrochloric acid to give p_H 4.6–4.8. The curd, precipitated in fine grains, is drained off. The more acid is added the lower are the ash content of the resulting casein and the viscosity of its solution. This treatment is limited by the fact that a very acid curd is difficult to handle. Sulphuric acid yields the hardest curd and lactic acid the softest. An outline of the removal of the albumin from the whey, its concentration, and crystallisation of the lactose produced is also given.

E. B. HUGHES.

Solubility method of classifying acid caseins.

W. R. MUMMERY and F. BISHOP (Analyst, 1930, 55, 367–369).—Caseins may be classified by their solubility in borax. The casein (2 g.), ground if necessary to pass 30-mesh (opening 0.0223 in.) is placed in a $\frac{3}{4}$ -in. test-tube, 12 c.c. of borax solution (20.833 g. made up to 1 litre) are added, and the solution is frequently stirred while kept at 70° for $\frac{3}{4}$ hr. Incomplete solubility is then demonstrated if the casein is not fully dissolved. When dissolved, the solution is made up to 100 c.c., 1 c.c. of which with 10 c.c. of water are placed in another tube, to this are added 5 drops of bromothymol-blue indicator (0.1 g. macerated with 3.2 c.c. of 0.05N-sodium hydroxide and made up to 250 c.c.), and the colour is compared with that of the standard buffer solution (Clark and Lubs' formula; B., 1916, 980). The p_H of the solution after dilution (solubility index) indicates the solubility of the casein, which above 6.8 (blue) is termed very good; from 6.8 to 6.4 (green), good; and below 6.4 (yellow), passable. If the casein has not completely dissolved, the p_H is 6 or less, provided that the undissolved particles are casein. Determinations on 230 samples of lactic acid caseins showed that 5.2% had solubility indices of 6.5, 37.8%, of 6.6; 41.7% of 6.7; 13.5% of 6.8; 1.3% of 6.9; and 0.5% of 7.0.

D. G. HEWER.

Substances causing heating in pears. E. BOTTINI (Annali Chim. Appl., 1930, 20, 181–187).—The so-called heating of pears may be reproduced artificially by storing the fruit in an atmosphere containing the vapour of ether, amyl or ethyl acetate, acetaldehyde, light petroleum, toluene, amyl or ethyl alcohol, chloroform, carbon tetrachloride, or carbon disulphide. These compounds kill the epidermal tissues and lead to the appearance of an enzymic action, which destructively oxidises aromatic compounds such as phenols and glucosides and causes browning of the vegetable tissues. Death of the epidermal tissues, accompanied by entry

of air, allows of the rapid diffusion of the heating throughout the interior of the fruit and of rapid invasion by moulds. The enzymic oxidising action is greatly hindered by the acidity of the fruit. Obviously the phenomenon cannot be combatted effectively by wrapping each pear in paper, as this maintains a stagnant atmosphere round the fruit. Adequate ventilation of the cold store and the use of a means of packing which favours rapid elimination of the ethereal metabolic products should reduce to a minimum losses due to this cause.

T. H. POPE.

Hydrogen-ion concentration of the juice of the pear during ripening. D. CORTESE (Annali Chim. Appl., 1930, 20, 197–204).—During the growth period of pears the p_H value for the juice falls gradually from 3.26 to the minimum 2.07, corresponding with the maximum size of the fruit. During the subsequent ripening period the value increases to about 4.5.

T. H. POPE.

F.p. of pears and apples. M. AGGRADI (Annali Chim. Appl., 1930, 20, 188–196).—The mean f.p., which represent the lowest temperatures to which fruit may be exposed without danger from alteration, vary from -1.4° to -3.3° for different varieties of pears and from -1.6° to -2.58° for different varieties of apples.

T. H. POPE.

Acidity and corrosion in canned fruit. E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1930, 22, 615–617; cf. B., 1928, 159).—Additional data are given to show that tin becomes less noble compared with iron as the p_H decreases, although the effect of such decrease varies according to the particular acid present. The greater tendency of enamelled cans containing fruit to perforate and to swell owing to development of hydrogen is due to the protective action of the large area of less noble tin on the small area of exposed iron in the plain can, whereas in the enamelled can the small area of exposed surface is rapidly detinned, thereby increasing the area of exposed iron and reducing its protection by the anodic tin. Addition of citric acid to sweet black cherries inhibits the tendency of these to perforate the can. The greater corrosive effect of dehydrated prunes over that of fresh prunes is similarly due to the dipping of the prunes into lye before dehydration in order to permit rapid evaporation through the skin, the p_H being thereby increased. Citric acid had a greater effect in rendering tin anodic to iron than a commercial grade of malic acid examined, although the range of p_H was the same in both cases. The presence of anthocyanin pigments increases corrosion in enamelled cans.

W. J. BOYD.

Microscopical diagnosis of marmalades. W. OETKER (Z. Unters. Lebensm., 1930, 59, 132–184).—After making an extensive anatomical and histological examination of the seeds and fruit of the species *Ananas*, *Ribes*, and *Vaccinium*, the author deals with the principles of determination and methods for the investigation of marmalades. It is considered that especial value should be attached to the detection of starch, large quantities of which were found in unripe fruit, in stone fruits, and in tomato, rhubarb, swedes, and carrots. Only apple marmalade contains large quantities of

starch that is of diagnostic value. But diagnosis merely on the presence of starch is not possible. The vascular bundles are of value in diagnosis. Coarse adulterants such as turnips are easily shown by their large vascular bundles, but the differentiation of fruits from one another is difficult. The absence of sclerenchymatous elements, as well as the kind and quantity of vessels present, supply important hints in diagnosis. The various fruits give fluorescent colours in ultra-violet light, but this is not yet of any value.

B. W. TOWN.

Simple polarimetric test for sugars in jams. S. J. LEWIS (Analyst, 1930, 55, 384).—The test first introduced by Juckenack and Pasternack (Z. Unters. Nahr. Genussm., 1904, 8, 10), depending on the fact that the specific rotation of the inverted extract of all fruit juices is approx. -20° , is used for a rapid sorting test for jams. The jam (25 g.) is carefully stirred with 120 c.c. of water for 1 min. without breaking the fruit, strained, 2 g. of nitric acid are added, and, after keeping in boiling water for 30–35 min., the mixture is cooled and just sufficient (about 12 c.c.) of a concentrated solution of lead subacetate added; after shaking, leaving for 5 min., and filtering, the inverted sugar solution, lead-free and nearly citric acid-free, is polarised at 20° . The total solids are determined in 5 c.c. of solution, their acidity is found and calculated as citric acid, and this weight is deducted from the solids, giving the weight of "saccharine solids" (w). The sp. rotation is calculated from: $[\alpha] = 100\alpha/20 \cdot 1w$, i.e., $5\alpha/w$ for a 100-mm. tube and $2 \cdot 5\alpha/w$ for a 200-mm. tube. Results obtained for various jams are: raspberry, from $-20 \cdot 8^\circ$ to $-22 \cdot 9^\circ$; for strawberry, $-20 \cdot 5^\circ$, $-20 \cdot 8^\circ$; stoneless plum, $-22 \cdot 4^\circ$, $-21 \cdot 9^\circ$; strawberry with 10% of added glucose, $+1 \cdot 21^\circ$. D. G. HEWER.

Detection of dried plums in plum jam. G. KAPPELLER, G. PRANGE, and W. REIDEMEISTER (Z. Unters. Lebensm., 1930, 59, 191–197).—The method employed depends on the differing colours of the precipitate obtained with lead acetate, and also with acid. Jam made with dried plums sometimes has a low acid content. A microscopical examination also helps in the differentiation, as does the occasional presence of mites in the jam made with dried fruit.

B. W. TOWN.

Determination of the alkalinity of fruit produce by conductivity measurements. P. HIRSCH and K. RICHTER (Z. Unters. Lebensm., 1930, 59, 184–191).—From apple juice, cider, etc. values were found for the alkalinity that harmonised with those found by titration of the ash. The method is more rapid and the values are more sharply defined, and 4–5 determinations can be carried out per hour. The solution to be investigated is mixed with an equal volume of standard hydrochloric acid and the conductivity measured. The conductivity of the mixture is denoted by x_m , that of a 1:1 mixture of standard hydrochloric acid and water by x_a , and of a 1:1 mixture of solution and water by x_s . Then the decrease in the conductivity $dx = (x_a + x_s) - x_m$. The value for the alkalinity is then determined by reference to a curve showing the value of dx for known mixtures of the fruit acids and their sodium salts.

B. W. TOWN.

Caffeine content of natural and treated coffee. A. RINCK and E. KAEMPF (Z. Unters. Lebensm., 1930, 59, 281–285).—The caffeine from treated coffee does not differ in its solubility or physiological action from that from natural coffee.

B. W. TOWN.

Technical aids in the manufacture of foods. Preservatives. A. BEHRE (Chem.-Ztg., 1930, 54, 325–327, 346–347).—The literature of food preservation by chemical methods is reviewed and discussed. A table of some 18 materials has been prepared showing compositions, proprietary names, applications, concentrations, physiological effects, and action on bacteria, moulds, etc. A further table gives the doses which produce symptoms of poisoning or death in dogs and puppies. Such materials as sugar, alcohol, oils, nitrates, nitrites, phosphates, acetates, and tartrates are considered unobjectionable, and are not included in the discussion. Mention is made of the difficulty of selecting for each and every food a material that is efficient but harmless. The use of 0.2% of benzoic acid in margarine and of sulphurous acid for preserved fruits appears to be satisfactory.

H. J. DOWDEN.

Detection and determination of dextrans in food materials. J. GROSSFELD and G. HOLLATZ (Z. Unters. Lebensm., 1930, 59, 216–236).—It was found that the colloids and colloidal starch could be precipitated from food materials by treatment with potassium ferrocyanide and zinc acetate without precipitating any of the dextrin. The dextrin was precipitated with alcohol, and it was found that considerably more dextrin was precipitated from a concentrated than from a dilute solution. The quantity of dextrin precipitated (x) is related to the quantity of syrupy starch (y) by the equation $y = ax^n$, where a and n are constants. The mean value of a was found to be 2.50, and of n was 2/3. A table is set out from these values from which x can be calculated.

B. W. TOWN.

Iodometry of food products. I. K. FUCHS, W. RUZICZKA, and E. KOHN (Z. Unters. Lebensm., 1930, 59, 266–281).—The material is treated with standard alcoholic solution of iodine, following the procedure of Margosches and co-workers (B., 1925, 639), and the excess of iodine titrated with thiosulphate. In the case of food products containing starch, the usual direct titration is difficult to carry out because the starch grains absorb iodine so strongly that the emulsion shows blue long after the end-point is reached. This difficulty is removed by adding excess of thiosulphate and back-titrating with standard iodine, when a sharp end-point is obtained.

B. W. TOWN.

Separation and determination of the solid fatty acids in food fats. J. GROSSFELD and A. SIMMER (Z. Unters. Lebensm., 1930, 59, 237–258).—A new procedure is given for the separation of the solid from the liquid fatty acids, depending on the crystallisation of the lead salts from dilute alcohol. The method gives a complete separation of oleic, linoleic, and linolenic acid. It was further found that hardened fats contained different isooleic acids, with different solubilities of their lead salts, of which one is probably n -elaidic acid. By adsorption on lead palmitate one can detect down to 2% of rape oil in olive oil. An addition of 5–10%

of rape oil was detected in linseed oil, peanut oil, and in sesamé oil by the raising of the iodine value of the solid fatty acids. B. W. TOWN.

Determination of minute amounts of lead and copper in food, beverages, etc. F. W. RICHARDSON (Analyst, 1930, 55, 323—325).—If the material yields very little ash 0.2 g. of calcium hydroxide is added as fixative. For liquids, 100 c.c. are evaporated in a silica dish, incinerated by an iron burner from the top and later also from below. The ash is heated almost to dryness with 2 c.c. of hydrochloric acid (d 1.16), then 1 g. of citric acid, 2 g. of ammonium acetate, and 5 c.c. of water are added, and, after boiling for $\frac{1}{2}$ min. and filtering, the filtrate is made slightly alkaline to alcoholic phenolphthalein with 10% ammonia, and then decolorised by acetic acid, allowing 0.03 c.c. excess of acid. Hydrogen sulphide solution is added and, after keeping warm for 1 hr., any sulphides and sulphur are collected on a small filter, washed, and the whole is transferred to a test tube to which are added 2 c.c. of a mixture of 50 c.c. of 10 vol. hydrogen peroxide, 10 c.c. of dilute sulphuric acid (1:1), and 60 c.c. of methylated spirit (64 O.P.). The tube is kept warm till the contents are bleached, and, after 1 hr., the filter paper is removed and washed first with the contents of the tube and then with 1 c.c. of peroxide mixture. After addition of 5 c.c. of water the hydrogen peroxide is expelled, the solution cooled, and the process continued as with the first filtrate (but leaving only 0.1 c.c. of excess acetic acid). The filtrate is made up to 10 c.c. and compared in miniature Nessler glasses with a standard containing 0.1 mg. Cu per c.c. Lead is determined in the contents of the small filter by pouring 2 c.c. of a 40% solution of ammonium acetate over the filter and washing with 3 c.c. hot water, whereby the usual procedure with phenolphthalein and ammonia solution gives a liquid with a small excess of acetic acid. The solution is made up to 10 c.c., hydrogen sulphide water added to 5 c.c., and a comparison made with a lead nitrate solution containing 0.1 mg. Pb per c.c. Modifications are necessary in treating materials leaving much phosphatic ash. D. G. HEWER.

Homogenisers. MÖLLERING.—See I. **Preparation of condensed milk and ice-cream.** PARISI.—See XVII. **Air-free water for food analysis.** FREELAND.—See XXIII.

PATENTS.

Preparation or substance for use in the manufacture of bread and such like. A. ARCHIBALD (B.P. 330,779, 22.6. and 26.7.29).—Cotton oil for use in bread baking is replaced by an emulsion of water, cottonseed oil, rice, saponin, and glycerin. The saponin-glycerin mixture may be replaced by an infusion of Irish moss. E. B. HUGHES.

Treatment of flour, meal, milling products, and the like. N.V. INTERNAT. OXYGENIUM MAATS. "NOVADEL" (B.P. 312,182, 21.5.29. Holl., 19.5.28).—Flour etc. is improved by treatment with small quantities of mono- or di-chloroamine or a mixture of the two. Air is passed through a mixture of sodium hypochlorite (or hypochlorous acid) and ammonia, thus becoming

charged with monochloroamine, and is brought into intimate contact with the substances to be treated. The use also of nitrogen trichloride or of per-compounds is not excluded. E. B. HUGHES.

Preparation of cereal food. W. D. and B. A. SMITH (B.P. 327,811, 14.2.29).—Wheat grains are cooked by heating under pressure with water, heating the product by steam and hot air, immersing it in vegetable oil heated at about 200°, and then centrifuging. E. B. HUGHES.

Butter-composition test. C. C. BOMBERGER (U.S.P. 1,748,668, 25.2.30. Appl., 14.4.27).—A rough method of determining moisture, fat, curd, and ash of butter by the application of heat only is described. The butter (10 g.) is weighed between the various heat-treatments which are calculated to remove (a) moisture; (b) fat by burning; (c) curd, by heating until nearly white, leaving the ash. J. H. BUSHILL.

Manufacture of cream and like fatty products. J. E. NYROP (B.P. 324,276, 5.3.29. Addn. to B.P. 297,256; B., 1928, 825. Cf. also B.P. 307,167 and 312,875; B., 1929, 376, 659).—The gaseous medium for spray-drying of cream etc. is first purified by being passed through material such as active charcoal, silica gel, chalk, etc. Instead of inert gas, air may be used as drying medium if deprived of its oxygen content by combustion and subsequent purification. E. B. HUGHES.

Production of artificial cream. S. E. HEMBERG (B.P. 330,307, 6.3.29).—Pasteurised milk freed from butter fat is mixed with melted vegetable or animal, vitamin-containing fats freed from free fatty acids, emulsified by whipping, and homogenised. H. ROYAL-DAWSON.

Treatment of creamery waste. J. T. TRAVERS, ASSR. to OHIO SANITARY ENG. CORP. (U.S.P. 1,747,802, 18.2.30. Appl., 2.8.27).—A process for clarifying effluent from creameries in which 1000 gals. are treated with 3—18 lb. of solid comprising roughly 80 pts. of magnesium sulphate, cement dust, or various waste products from alkali plants, 15 pts. of alkali, usually slaked lime, and 5 pts. of ferrous sulphate, aluminium sulphate, or calcium monophosphate. The proportion of the ingredients is varied to suit the particular requirements. The alkali neutralises any excess acid, making a slightly alkaline solution; the precipitant (e.g., ferrous sulphate) together with the electrolyte present then precipitates the protein, carrying with it the fat. J. H. BUSHILL.

Making cheese. L. A. THOMPSON (U.S.P. 1,745,962, 4.2.30. Appl., 21.10.27).—By maintaining the temperature of milk at 46.1—48.9° for 30 min. in the presence of 20% of malt wort, cooling to 90°, and adding rennet, a cheese may be produced of the Cheddar variety containing partially digested casein and possessing additional nutritional value. J. H. BUSHILL.

Egg products and their production. A. K. EPSTEIN (B.P. 327,701, 9.10.28).—If sucrose or other carbohydrate and common salt or other edible salt together with an edible acid (e.g., acetic) are added to liquid egg before freezing, various physical consistencies of the product after thawing can be obtained, depending

on the amounts and proportions of the above-mentioned ingredients that are added. E. B. HUGHES.

Treatment of cacao beans. G. DEFREN (U.S.P. 1,750,795, 18.3.20. Appl., 4.1.26).—The raw beans are soaked for 2–12 hrs. in water at 60° to extract most of the volatile constituents (acids) and bitter (non-volatile) constituents, thereby enhancing the true chocolate flavour, permitting more uniform roasting subsequently, and facilitating more complete removal of shell during winnowing. E. B. HUGHES.

Fermentation of cacao beans. Further preparing cacao beans after fermentation. B. MÜLLER (B.P. 305,236 and 329,015, [A] 2.2.29, [B] 8.2.29. Ger., [A] 2.2.28).—(A) Complete alcoholic fermentation of beans and pulp to prevent germination is obtained in 3 days by placing them, free from the pods, directly into transportable fermenting boxes having ventilating pipes; the necessity for turning the beans to prevent overheating and undesirable acetic fermentation is thus avoided. (B) The fermented beans, heated on trays in a closed space below 50°, swell, change in colour, and by oxidation lose their bitterness, and are partly dried by a current of air below 60°. The warm beans are finally dried without further heating, by continuous circulation of outside air through perforated pipes, thus avoiding destruction of enzymes. E. B. HUGHES.

Production of chocolate. H. BOLLMANN and B. REWALD (B.P. 330,450, 30.7.29. Ger., 8.7.29).—Deterioration of chocolate by heat, light, and air is obviated by the addition of 0.1–0.5% of animal or vegetable lecithin, previously dissolved in cacao butter at 60°.

E. B. HUGHES.

Sterilisation and preservation of fruit juices. W. DOERBECKER (B.P. 330,368, 29.4.29).—A preservative, e.g., sodium benzoate solution, is added to fruit juices, and the mixture then subjected to the action of ultra-violet rays.

H. ROYAL-DAWSON.

Bleaching of foodstuffs. L. MELLERSH-JACKSON. From PILOT LAB., INC. (B.P. 327,670, 3.10.28).—The bleaching agent claimed consists of a higher fatty acid, peroxide, or a mixture of such peroxides, and is produced from a fat, such as coconut oil, by first forming the fatty acid chlorides and then peroxidising with hydrogen peroxide in alkaline solution. Suitable foodstuffs are those containing chromophoric oil-bearing materials (e.g., flour, meals, egg yolk).

E. B. HUGHES.

Treating comestibles. ATLANTIC COAST FISHERIES Co., Assees. of H. F. TAYLOR (B.P. 311,317, 1.3.29. U.S. 9.5.28).—Before quick-freezing, flesh foods are immersed in a buffer solution to give p_H 7, thus preventing drip on thawing, and in common salt solution to prevent absorption of water from the buffer. Nitrite is also added to preserve colour. Suitable buffers are tri-sodium citrate or phosphate, sodium carbonate or hydroxide, or the corresponding potassium or ammonium derivatives.

E. B. HUGHES.

Treating comestibles with smoke. ATLANTIC COAST FISHERIES Co., Assees. of H. F. TAYLOR (B.P. 309,405 and 316,283, 1.3.29. U.S., [A] 9.4.28, [B] 27.7.28).

—(A) In the smoking of fish and meat, ammonia gas is mixed with the smoke to combine with aldehydes, neutralise acids, and favour colour development (oxidation of pyrogallol-like substances). (B) Apparatus is described for treating fish etc. in a closed room with smoke at about 30°, saturated with water vapour to prevent shrinkage. [Stat. ref.] E. B. HUGHES.

Treatment of comestibles with smoke. H. F. TAYLOR, Assr. to ATLANTIC COAST FISHERIES Co. (U.S.P. 1,760,091, 27.5.30. Appl., 9.4.28).—See B.P. 309,405; preceding.

Production of puffed, popped, or expanded cereal food products. KELLOGG Co., Assees. of E. H. MCKAY (B.P. 300,261, 8.10.28. U.S., 10.11.27).

Method and apparatus for continuous drying and roasting of cacao beans and the like. D. G. STEELY and F. W. BLAKE (B.P. 330,826, 14.8.29).

Smoke-producing apparatus for use in smoking comestibles. ATLANTIC COAST FISHERIES Co., Assees. of H. F. TAYLOR (B.P. 318,194, 1.3.29. U.S., 30.8.28).

Kneading machine (U.S.P. 1,750,558).—See I. Acid calcium phosphate (B.P. 330,777).—See VII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Rapid determination of phenolphthalein in aperients. G. ROSENBERGER (Chem.-Ztg., 1930, 54, 345).—The pastille types of aperient generally contain phenolphthalein as the active constituent (0.1–0.15 g. in each) compounded with sugar and suitably flavoured and coloured. Agar-agar jelly is frequently used as a base. The analysis of these products is based on the insolubility in water of phenolphthalein. A weighed pastille is boiled with 25 c.c. of water and, after cooling to 80°, the residue is collected and washed with about 75 c.c. of water at 75–80°. The residue is then extracted on the filter with boiling absolute alcohol and washed with 95% alcohol until the washings are free from phenolphthalein. The extract and united washings are evaporated to dryness and the dried residue is weighed. To compensate for the partial solubility in water of phenolphthalein a correction is applied to the weight by adding 0.0035 g. for each 100 c.c. of aqueous filtrate and washing water. When agar-agar is present in the material, the initial disintegration is effected by boiling with 50 c.c. of water containing 2 c.c. of hydrochloric acid (d 1.19), and, after cooling and filtering, the residue is washed until the washings are free from chloride. The correction to be applied is 0.0035 g. per 100 c.c. of water used, and is the mean of 50 determinations using 1.5–2.5 g. of phenolphthalein and about 100 c.c. of water at 80°.

H. J. DOWDEN.

Colorimetric determination of novocaine and anaesthesin with β -naphthol. W. EISSNER (Arch. Pharm., 1930, 268, 322–323).—To 0.05–1 c.c. of the approx. 0.2% solution to be analysed are added, successively at 0°, 0.1 c.c. of 2*N*-sulphuric acid, 0.5 c.c. of 1% sodium nitrate, 1.0 c.c. of 2*N*-sodium hydroxide, and 0.2 c.c. of 4% alkaline β -naphthol solution. After 1 hr. each at 0° and at the ordinary temperature, the red colour developed is compared with that of a solution

prepared from the pure drug, compensating, if necessary, for the yellow colour of the β -naphthol. The solutions obtained from novocaine obey Beer's law; those from anæsthesin do not. Cocaine, small amounts of adrenaline, and serum do not interfere. H. E. F. NOTTON.

Seventh Report of the Essential Oil Sub-Committee to the Standing Committee on Uniformity of Analytical Methods. Determination of solubilities [of essential oils] (Analyst, 1930, 55, 386; cf. B., 1930, 639).—It is recommended that tests should be carried out, unless otherwise stated, at 15.5°, and that the concentrations of alcohol used should be given as vol.-%. The terms solubility, opalescence, and turbidity as applied to essential oils are also defined.

D. G. HEWER.

Ambergris and how to recognise it. F. R. MORRISON (Bull. Tech. Mus., Sydney, 1929, No. 15, 9 pp.).—The different varieties of ambergris are described together with their values, uses, and characteristics. All ambergris will float on sea-water and, if the latter is slowly heated, the material will melt to a brown or black liquid before the water reaches its b.p. A piece of ambergris about the size of a pea is almost completely soluble in about $\frac{1}{2}$ oz. of warm alcohol, giving a fluorescent solution. A needle, previously heated in a candle-flame for 10–20 sec., on being pressed into a sample of ambergris to a depth of $\frac{1}{8}$ in., forms a dark brown resinous liquid, which, when touched by the finger, leaves pitch-like "strings" adhering to the skin. If the needle be withdrawn and placed in the flame, the ambergris burns; if the flame be extinguished the odour of the "smoke" is somewhat fatty or resinous and resembles that of burning rubber.

E. H. SHARPLES.

Enzymic processes in the preparation of perfumes from plants. A. ELLMER (Reichstoffind., 1929, 4, 105–109, 139–142, 160–162; Chem. Zentr., 1930, i, 758).—A discussion.

A. A. ELDRIDGE.

Homogenisers. MÖLLERING.—See I. **Detection of ethyl o-phthalate.** SUCHODOLSKI.—See III. **Solubility of sulphur.** HENVILLE.—See VII.

PATENTS.

Production of anæsthetics. W. SCHOELLER and H. G. ALLARDT, Assists. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,765,621, 24.6.30. Appl., 18.8.28. Ger., 6.8.27).—See B.P. 321,968; B., 1930, 348.

Manufacture of santalol compounds. H. WALTER, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,761,376, 3.6.30. Appl., 3.1.28. Ger., 17.7.25).—See B.P. 278,982; B., 1928, 36.

Stable [medicinal acetyl]salicylate composition. M. COPLANS (U.S.P. 1,764,933, 17.6.30. Appl., 20.12.28. U.K., 12.1.28).—See B.P. 315,330; B., 1929, 835.

Basic oxime ethers of cyclic compounds. W. KROPP, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,733,462, 29.10.29. Appl., 25.8.27. Ger., 23.9.26).—See B.P. 301,956; B., 1929, 149.

Apparatus for vaporising chemicals [e.g., medicaments] in rooms [for inhalation purposes].

"AIROSANA" TROCKENINHALATIONS-GES.M.B.H. (B.P. 311,733, 4.5.29. Ger., 15.5.28).

Processes and devices for [air-]drying of tobacco and similar materials. E. QUESTER (B.P. 330,778, 21.6.29).

Sanitary agent (B.P. 330,099).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Spectral sensitisation of photographic emulsions. Bathing with pinacyanol-pinaflavol mixtures. B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1930, 4, 693–701).—Pyridine increases the effectiveness of sensitisation and reduces the tendency to flocculation, which may be a serious difficulty when pinaflavol is mixed with other sensitising dyes. Optimum conditions are: bathing for 1 hr. at not above 10° in a vertical position with an aqueous bath containing, per litre, 4 mg. each of pinaflavol and pinacyanol and 10 g. of pyridine. Good results may also be obtained with some emulsions by bathing for 3 min., with agitation, in a bath containing, per litre, 10 mg. of each of the dyes and 10 g. of pyridine. The pyridine must be free from reducing impurities. W. E. DOWNEY.

Hypersensitising with peroxide and silver salts. U. SCHMIESCHEK (Brit. J. Phot., 1930, 77, 276–277).—Efficient hypersensitising baths for panchromatic materials may be prepared from a combination of a soluble silver salt with hydrogen peroxide, e.g., 1% solution of silver nitrate 4 c.c., 30% hydrogen peroxide 1 c.c., water 400 c.c. The action of such baths is almost independent of the pH , since good hypersensitisation can be obtained in the presence of either acetic acid or ammonia, the increase in sensitivity being slightly the greater with the acid bath. To test the efficiency of various silver salts and their effect on the keeping properties of the plate, baths were prepared with the nitrate, chloride, chromate, carbonate, phosphate, oxalate, arsenate, iodate, molybdate, and tungstate, according to the following formula (in which the silver is present as the ammonium complex salt): 25% ammonia solution 1 c.c., 30% hydrogen peroxide 1 c.c., water 400 c.c., and silver salt equivalent to 0.025 g. Ag. The nature of the acid radical had very little effect on the hypersensitisation, but the molybdate and tungstate showed a marked effect in preventing fog. Non-colour-sensitised and orthochromatic plates were scarcely affected by the silver tungstate bath, and the slow, fine-grain, panchromatic plates showed a greater percentage increase in sensitivity than the faster panchromatic plates. J. W. GLASSETT.

PATENTS.

Manufacture of silver halide emulsions. I. G. FARBERIND. A.-G. (B.P. 305,143, 31.1.29. Ger., 31.1.28. Addn. to B.P. 271,475; B., 1928, 69).—In place of the glyoxaline of the prior patent any other organic reagent capable of forming a stable silver salt of solubility not greater than 1–2 mg. per litre at 18°, e.g., thioglycollic acid, thiolacetic acid, tetrazole, 2:6-dithiol-4-keto-3:5-diphenylpenthiophen, and allylaminothiadiazoole hydrogen sulphide, may be used. C. HOLLINS.

Sensitised coating and method of making and applying same and photographic plate or film produced thereby. E. DE STUBNER (U.S.P. 1,752,069, 25.3.30. Appl., 14.3.27).—The danger of displacement or stripping of a sensitised film from a celluloid base is overcome by eliminating the special cementing agent by using as sensitised coating a soluble cellulose ester which forms a homogeneous union between the coating and film directly. Silver nitrate and potassium iodide solutions are mixed in a tank containing a soluble cellulose ester such as nitro- or acetyl-cellulose, very finely divided silver iodide thus being precipitated on the ester. The cellulose ester is washed free from potassium nitrate, dried with alcohol, and dissolved in a suitable solvent, forming a lacquer containing silver iodide in suspension. This is applied directly to the celluloid film.

J. W. SMITH.

[Resist for] photographic etching. A. B. DAVIS, Assr. to KEYSTONE WATCH CASE CORP. (U.S.P. 1,751,908, 25.3.30. Appl., 16.12.26).—A stable resist is prepared by the action of sulphur chloride dissolved in carbon disulphide on asphalt. The carbon disulphide is removed on a water-bath, and the residue dissolved in benzene, with addition of 2% of lavender oil as accelerator and sensitiser. This solution is applied to the metal plate and dried. Before and after exposure the film is dipped for 15 sec. in ether, which causes the film to become more adherent to the plate. Development is carried out with turpentine and petroleum benzine (3:1).

J. W. SMITH.

Photographic resist and photographic etching. A. B. DAVIS, Assr. to KEYSTONE WATCH CASE CORP. (U.S.P. 1,751,909, 25.3.30. Appl., 28.1.27).—The resist is prepared by dissolving 15 g. of shellac in a solution of 5 g. of borax in 100 c.c. of water; 5 c.c. of 28% ammonia solution and 15 c.c. of 20% ammonium dichromate solution are added. The metal plate is coated with this mixture, dried, exposed, and developed by washing in methylated spirit containing a little dye to facilitate judging the degree of development. After development the plate is dried and placed in alcoholic tannic acid (50 g. of tannic acid dissolved in a mixture of 50 c.c. each of water and methylated spirit), which renders the shellac insoluble after heating. The plate is dried and baked and finally electro-etched in the usual way.

J. W. SMITH.

Preparation of transparent pictures. J. HEIDENHAIN (B.P. 327,068, 18.6.29).—A transparent support is coated with a layer of lead sulphide by immersion in a suspension of the latter prepared by adding a 2% aqueous solution of potassium hydroxide to a mixture of 2% aqueous solutions of lead acetate and thiourea. This is then coated with a light-sensitive layer, e.g., sensitised albumin, which is then printed, developed, coated with asphaltum, and etched to give a negative. When used for screen negatives, the dots may be intensified or reduced by suitable chemical agents or mechanical means.

J. W. GLASSETT.

Production of photographs in natural colours. C. RUZICKA (B.P. 326,764 and 326,779—326,781, [A—D] 13.12.28).—The following methods are described for the

preparation of colour screens. (A) The particles comprising the screen are prepared by hardening a suitably dyed gelatin solution with formaldehyde, allowing this to set to a stiff jelly, and, after breaking up into small particles by pressure through a fine mesh, drying the particles very slowly with agitation. Equal quantities of red, yellow, and blue particles are intimately mixed and incorporated in a nitrocellulose solution which is applied in a thin layer to the support for the sensitive emulsion. (B) The screen is prepared from differently coloured particles of glass, transparent metallic or metalloidal compounds, transparent minerals, starch, or cellulose ester. Equal quantities of the particles are incorporated in a vehicle such as nitrocellulose solution and applied to the emulsion support. (C) A dyed gelatin solution is treated with formaldehyde and, before gelatinisation occurs, the gelatin is precipitated in the form of fine particles by the gradual addition of alcohol with rapid agitation. These particles are then further hardened and dried. Alternatively, a dyed solution of a resin or gum in a suitable solvent is subjected to an increase in pressure sufficient to precipitate the resin or gum in fine particles. Coloured particles from either of these methods are thoroughly mixed and strewn upon a thin sheet of nitrocellulose or cellulose acetate, the surface of which has been made tacky by treatment with a solvent. A similar sheet of material is then placed over the particles and the whole firmly joined under pressure. (D) Before the colour screen is applied, the support is coated with a highly reflecting metallic layer of silver or other suitable metal either by the metal-spraying process or by electrodeposition.

J. W. GLASSETT.

Preparation of pictures to be produced by tanning action. M. P. SCHMIDT, R. ZAHN, and W. KRIEGER, Assrs. to KALLE & Co. A.-G. (U.S.P. 1,762,033, 3.6.30. Appl., 7.8.28. Ger., 22.8.27).—See B.P. 296,008; B., 1930, 218.

[Combined taking screen and panchromatic plate for] colour photography. C. L. FINLAY, and FINLAY PHOTOGRAPHIC PROCESSES, LTD. (B.P. 330,508, 9.2.29).

Light filter (U.S.P. 1,751,220).—See I. Decolorising film (U.S.P. 1,743,155).—See VI.

XXII.—EXPLOSIVES; MATCHES.

Manufacture of nitrocellulose of very high nitrogen content for B.F.P. powder. DEMOUGIN (Mém. Poudres, 1928, 23, 262—267).—Investigation of the maximum degree of nitration of linters, consistent with stability of the product, indicates the necessity for using mixed acid containing about 22% HNO_3 and 10.3—10.5% H_2O ; the nitrocellulose produced yields approx. 213.5 c.c. NO . A series of curves is given which show the variation in nitrogen of the nitrocellulose for varying amounts of water and nitric acid in the nitration acids. These curves have the same form as those obtained by Lunge, but do not show a maximum nitrogen content at 13.8%, equivalent to 220 c.c. NO , found by him. For nitrocellulose for B.F.P. powder, the mixed acid should contain 21.0—21.5% HNO_3 and 15.6—15.7% H_2O , the yield of nitric oxide being then 199 c.c.;

it is not advisable to exceed this if a completely soluble nitrocellulose is required. In the stabilisation of nitrocellulose for B.F.P. powder, it is possible to increase the solubility by long boilings of slight acidity without detriment to the stability or the nitrogen content.

W. J. WRIGHT.

Manufacture and stabilisation of nitrocellulose.

A. H. BRESSER (Ind. Chemist, 1930, 6, 249—251).—Disintegrated cellulose is weighed and nitrated with 50 pts. of mixed acid in either stoneware pots or cast-iron centrifuges with acid-resistant internal lining. The acid may be removed in the same centrifuge or in another one, but in the latter case the management of the connexions and cocks is troublesome and dangerous. A table gives the relation between the composition of the mixed acid and the resulting nitrocellulose. The temperature should not exceed 68°, but has no influence on the composition of the product. The nitrocellulose is washed several times with cold water and then boiled with two changes of hot water and live steam. It is then ground in beaters with addition of a little soda and agitated for 8 hrs. in iron stabilisers lined with stoneware. Bleaching is best effected with hypochlorite.

C. IRWIN.

Elimination of dextrose in the stabilisation of nitrocellulose. H. LÉCORCHÉ (Mém. Poudres, 1928, 23, 275—284).—In experiments to determine the elimination of sulphuric acid and dextrose during the stabilisation of nitrocellulose, the nitrocellulose was kieri at 130°, 8 hrs. being sufficient to ensure stability. On extending this time, increasing amounts of dextrose were found in the wash-waters, and after 24 hrs., decomposition of the nitrocellulose was complete and the amount of glucose was 25%. The origin of the dextrose and the effect of nitrates of sugar on the stability of the product are discussed, and two hypotheses are formulated: (A) the existence of sugars in the cellulose before nitration, (B) the existence of two phases, imbibition and nitration proper, in the nitration. It would appear that if sugars exist in the linters an appreciable amount of nitrated sugars is produced during nitration, depending on the amount of acid imbibed. In regard to the sulphuric acid that is eliminated during stabilisation, this probably existed originally as mixed sulphuric and nitric esters of cellulose, or even of sugars. The sulphuric impurities are more stable than those containing nitric acid.

W. J. WRIGHT.

Nitration of wood and other forms of cellulose.

DEMOUGIN (Mém. Poudres, 1928, 23, 268—274).—Experiments were carried out by the Poudrerie d'Angoulême on the nitration of wood, alfa, and straw, the wood being in the form of wool, unsized paper, and wadding. In the Selwig plant, a product was obtained which yielded 207—210 c.c. NO, had a solubility of 20%, and was suitable for use in powder manufacture. The Thomson process caused difficulties with the wood-wool, and was quite inapplicable with the paper and wadding; the wood-wool was the most satisfactory form of cellulose. Notwithstanding its lower price as compared with linters, the cost of the nitrated product from wood was higher on account of the higher acid concentrations and the greater losses of acid. Nitration of alfa gave

a product intermediate in properties between that from cotton and from wood, but the tendency to form hard lumps renders the material unsuitable. Straw has only the advantage of availability, but has not been tested on an industrial scale.

W. J. WRIGHT.

Hygroscopicity of powder B and various nitroglycerin powders. M. MARQUEYROL (Mém. Poudres, 1928, 23, 300—303).—Three nitroglycerin powders containing, respectively, amyl phthalate, vaseline, and diphenylamine, and vaseline alone were tested for hygroscopicity in comparison with a thick B powder, BM₁₇, by exposure over sulphuric acid, and further tests were made to determine the effect of various periods of stoving. Results are tabulated. The nitroglycerin powders containing vaseline absorbed moisture less rapidly than the powder that contained amyl phthalate. The humidities of these powders in the condition of equilibrium were approximately of the same order of magnitude and much lower than that of BM₁₇.

W. J. WRIGHT.

Loss on heating of powder B and nitroglycerin powders. M. MARQUEYROL (Mém. Poudres, 1928, 23, 304—308).—BM₁₇ powder and three nitroglycerin powders, which had been preheated, were heated at 60° for prolonged periods and their loss in weight was determined at various times. BM₁₇ showed little loss and considerably less than the nitroglycerin powders; the curves for the latter coincided. The results give rise to the theory that in the period of rest between the two stovings the nitroglycerin diffused from the centre to the surface of the grains. Then, during the later heating, it evaporated from the surface. The nitroglycerin is therefore present in the form of a solid solution and not as a nitrocellulose complex.

W. J. WRIGHT.

Determination of residual solvent in B powders. DESMAROUX (Mém. Poudres, 1928, 23, 285—299).—The influence of the time of drying, the intensity of the immersion treatment, the content of soluble nitrocellulose, and the composition of the original solvent on the amount of residual has been investigated. No conclusion could be drawn regarding the effect of varying periods of drying, although generally the ether was retained more tenaciously. Examination of the effect of prolonged immersion in water at 80° showed the same irregularity; the alcohol chiefly was expelled. Increase in the thickness of the sample increased the amount of residual solvent. With increasing nitrocellulose content from 18 to 65% a regular increase in the residual alcohol and decrease in the residual ether were found; up to 98.8% nitrocellulose, however, the reverse took place. The composition of the original solvent has a direct influence on the amount of residual solvent. Analyses of various powders of various ages are given and show the persistence of the solvent.

W. J. WRIGHT.

Graphic method for determining the explosion temperature of SD powders. H. MURAOUR (Mém. Poudres, 1928, 23, 256—261).—Under certain conditions the determination of the explosion temperature from a measurement of pressure in experiments with the bomb gives results in agreement with those calculated from the heat evolved and the composition of the gases. The

latter method is preferable on account of its simplicity. Calculations of the explosion temperature of SD powders by Aunis show that it is a function of the nitric nitrogen content and the centralite content. By plotting the results, a graph has been constructed by means of which the explosion temperature may be calculated for any given nitric nitrogen or centralite content.

W. J. WRIGHT.

PATENTS.

Ammonium nitrate explosive and its manufacture. W. O. SNELLING and J. A. WYLER, ASSRS. to TROJAN POWDER Co. (U.S.P. 1,749,613, 4.3.30. Appl., 14.10.22).—Ammonium nitrate is allowed to react with nitrated toluene, lead, and water, preferably at temperatures above 100°, *e.g.*, 115°. The reaction is hastened by adding an alcohol as well, and it is possible to use a lead salt in place of lead. The product may be used in conjunction with nitrostarch.

S. K. TWEEDY.

[Self-]combustible mixture and its preparation. J. THIECKE, ASSR. to MINIMAX A.-G., and to DEUTS. PYROTECHNISCHE FABRIKEN A.-G. (U.S.P. 1,766,269, 24.6.30. Appl., 20.12.28. Ger., 25.10.27).—See B.P. 299,396; B., 1930, 488.

XXIII.—SANITATION; WATER PURIFICATION.

Removal and determination of nitrites in sewage effluents and waters. J. W. H. JOHNSON (Analyst, 1930, 55, 325—326).—The fact that nitrites can be easily and efficiently removed by boiling the slightly acidified liquid, and that, when distilled off completely, they can be collected and determined in the distillate is confirmed. The author attempts to explain the conflicting statement, appearing in "Methods of Chemical Analysis as Applied to Sewage and Sewage Effluents," published by the Ministry of Health, and taken from Report IV of the Royal Commission, Appendix V, 33—35, that "after reduction from 100 c.c. to 25 c.c.," apparently under similar conditions, "much nitrite remained," by suggesting that the boiled residues were repeatedly examined, in which case the loss observed would represent finality, half the original nitrite being lost during distillation. If a solution containing 0.001 g. or more of nitrous nitrogen is distilled, brown fumes appear which may be absorbed in sodium hydroxide, nitrites and nitrates both appearing in the distillate: $\text{N}_2\text{O}_4 + 2\text{NaOH} = \text{NaNO}_2 + \text{NaNO}_3 + \text{H}_2\text{O}$. In the case of the *iso*-form (cf. Thorpe, "Dictionary of Applied Chemistry," 1912, p. 688) the whole of the nitrites are present in the distillate: $\text{N}_2\text{O}_4 + 2\text{NaOH} = 2\text{NaNO}_2 + \text{H}_2\text{O} + \text{O}$.

D. G. HEWER.

Water purification [in the paper-mill]. J. W. BERRIMAN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 360—379).—Filtration by means of gravity and pressure filters is dealt with, and the plant described. The results of experiments on the treatment of water with aluminium sulphate are given. It is found that the optimum condition for the formation of filterable flocs lies between p_{H} 6.3 and 7.1, according to the turbidity of the water to be treated.

T. T. POTTS.

Air-free water for sulphur dioxide determinations in foods. D. M. FREELAND (Analyst, 1930, 55,

383).—The method of Magnus and Herd (B., 1930, 215) and of the Preservatives Determination Committee of the Chemists of the Manufacturing Confectioners' Alliance and of the Food Manufacturers' Federation (B., 1928, 346) both require the use of air-free water. A "Sparklet" siphon is filled with cooled de-aerated distilled water, a wash-bottle siphon inserted in the neck with the shorter limb at the level of the red line, and water displaced to this level by carbon dioxide. The wash-bottle fitting is removed, the components of the "Sparklet" are rapidly assembled, and the water is charged with carbon dioxide. Water stored thus yields results in sulphur dioxide determinations similar to those obtained with freshly de-aerated water.

D. G. HEWER.

Drinking waters for cattle. T. McLACHLAN (Analyst, 1930, 55, 372—375).—Comparison of samples of water from five ponds used for years by healthy cattle with samples of fourteen other waters shows that it is not safe to condemn such waters for cattle-drinking on the results of chemical analysis unless a definite chemical poison is present, such as may be derived from sheep dip, minerals introduced from the neighbourhood, an excess of hydrogen sulphide, free chlorine, or possibly certain weeds. A recommended water should not show an oxygen absorption figure of more than 1 pt. per 100,000 in 3 hrs., but even a figure of 7.8 may be perfectly safe. A water should only be condemned on the results of bacteriological examination when bacteria pathogenic to cattle can be isolated. If contagious disease, which may be water-borne, is present in a herd, the water supply should be very good, and no surface water allowed to be drunk.

D. G. HEWER.

Water for brewing. CAPRINO.—See XVIII.

PATENTS.

Sanitary cleansing and deodorising agent. H. PICKUP and W. E. CLARINGBOLD (B.P. 330,099, 3.5.29).—Pine, cedarwood, or clove oil, or a mixture of two or more of these oils, is added to the material described in B.P. 318,344 (B., 1929, 912).

L. A. COLES.

Purification of water. J. MUCHKA (B.P. 316,965, 20.6.29. Austr., 7.8.28).—Filtering apparatus is described in which chlorinated water is freed from suspended organic matter, iron, and manganese before passing forward to the dechlorinator.

C. JEPSON.

Softening of water by base exchange. K. MORAWE (G.P. 460,743, 1.11.24).—The water is filtered through disintegrated lignite which has been treated with solutions of alkali silicate and acid salt solutions or dilute acids. The softening action of spent lignite may be regenerated by means of an alkali salt (sodium chloride) solution.

S. K. TWEEDY.

Disinfecting, bactericidal, insecticidal, fungicidal, and vermin-destroying preparation. R. LIESKE and W. SCHEPSS, ASSRS. to WINTHROP CHEM. Co., INC. (U.S.P. 1,766,441, 24.6.30. Appl., 14.2.25. Ger., 10.4.24).—See B.P. 232,249; B., 1925, 940.

Air filter (U.S.P. 1,751,999).—See I. **Preparations for respirators** (B.P. 305,101).—See VII. **Creamery waste** (U.S.P. 1,747,802).—See XIX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 29 and SEPT. 5, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Economics of high-pressure steam. G. A. ORROK (Engineering, 1930, 130, 25—26).—Two sets of curves are plotted correlating: (1) maximum steam temperatures and B.Th.U. per kw.-hr., for the Carnot cycle, and for regenerative-reheat cycles at five selected pressures and both 100% and 80% of the theoretical efficiency, the 80% being considered to be sufficient allowance for all losses from the fuel to the switchboard; (2) steam pressures and savings over a standard of 200 lb./in.² for two selected temperatures (750° and 1000° F.). Three tables are also given showing operating data of stations using steam at 250—1450 lb./in.² It is concluded that raising the pressure increases the economy very largely at the lower ranges, the effect being much reduced at 800 and almost negligible at 1600 lb./in.²; raising the temperature has a much smaller effect. The installation costs of high-pressure stations are at present higher than for low-pressure, but the difference is likely to disappear; there is already no difference in maintenance costs. B. M. VENABLES.

Evaporation tests with models of Ruths' steam accumulator. K. NESSELMANN and F. DARDIN (Wiss. Veröff. Siemens-Konz., 1930, 9, [1], 369—389).—It is shown that with suitable precautions surface charges of more than 400 m.³/m.²/hr. may be obtained without overflowing; e.g., by fixing a cone just below the water surface and extending it into a cylinder reaching almost to the bottom of the accumulator it is possible to work with surface charges of 1650 m.³/m.²/hr. without causing foaming and the formation of large steam bubbles which break explosively from the surface of the water. This arrangement produces good circulation of the water, avoids the tendency to the formation of numerous layers of varying temperature, and reduces the capacity losses.

A. R. POWELL.

Chemistry of disintegration. A. CHWALA (Kolloid-chem. Beih., 1930, 31, 222—290).—Emphasis is laid on the importance of producing a satisfactory state of subdivision of various industrial products, such as paints, dyes, disinfectants, foodstuffs, etc., and the means of effecting the subdivision are considered. These are treated under three headings: (1) purely mechanical disintegration, (2) mechanical disintegration aided by the presence of almost chemically inert substances, (3) colloid-chemical processes aided by mechanical action. Types of colloid mills are described and the results obtained and their applications are outlined. The mechanism of peptisation is discussed.

E. S. HEDGES.

Adhesives and adhesion. Mechanical properties of materials and glued metal joints. W. B. LEE

(Ind. Eng. Chem., 1930, 22, 778—780).—In continuance of the work of McBain and co-workers (cf. B., 1928, 695), the tensile strengths of celluloid, cellophane, and fishing gut were measured, and the technique of the preparation of thin test-films and slabs is described. The strengths of the materials were higher as the rate of loading increased (cf. "lithographic gelatin"), and the strength of cellophane and the gut were decreased by increased relative humidity. A shellac-cresote cement and Bakelite "C" resin ($E = 34,600$ kg./cm.²) were studied in compression and bending. Of several common adhesives examined, shellac compositions gave the strongest joints between smooth nickel surfaces, but the adhesion between metal surfaces (in contrast to wooden ones) was less the rougher were the surfaces. The strengths of the joints were lower if the load was applied very slowly. The adhesion between optically polished quartz surfaces "wrung" with some highly purified ethylalkylcarbinols was measured (2.8—4.9 kg./cm.²).

E. LEWKOWITSCH.

Revivification of foam solutions for fire protection with carbon dioxide. C. B. WHITE (Chem. and Met. Eng., 1930, 37, 379—380).—By a few simple additions to the fire-protection pump the solution of sodium bicarbonate is circulated from the bottom of the storage tank, through a small tower, and back to the upper part of the tank; it is recarbonated in the tower by gas from a cylinder. The method is preferable to adding additional bicarbonate because if that is done the aluminium sulphate or other acid solution which must also be added has to be increased *pro rata* and eventually both will have to be scrapped. B. M. VENABLES.

Measurement of the heat expansion of solid bodies with the aid of photographic and mechanical recording devices. W. M. COHN (Chem. Fabr., 1930, 257—259).—The literature of the subject is briefly reviewed and a photographic recorder, devised by the author for measuring the thermal expansion of solids, using fused silica or Marquardt refractory as the standard rod, is described and illustrated.

A. R. POWELL.

See also A., July, 867, **Catalytic reactions at high pressures** (MORGAN). 883, **Thermoregulators** (BEATTIE and JACOBUS; HIBBEN; WESSON; EGERTON). **Protection of ventilating fans** (DREIFUSS and STAALE). 884, **High-vacuum distillation with the aid of liquid air and active charcoal** (LEWIN). 885, **Rapid determination of density of small amounts of solids** (CALEY).

Tower-absorption coefficients. SIMMONS and LONG. —See II. **Purifying gases.** HEALY.—See XI. **Measurement of colour-tone.** VAN WÜLLEN-SCHOLTEN.—See XIII.

* The remainder of this set of Abstracts will appear in next week's issue.

PATENTS.

Muffle furnaces. GIBBONS BROS., LTD., and T. E. BRIDGFORD (B.P. 329,787, 19.4.29).—The roof and sides of the muffle are built of hollow, triangular shaped bricks so arranged as to form a number of transverse, parallel flues, each of which communicates with a passage extending across the furnace below the floor of the muffle and all of which are spaced away from the roof and outer structure of the furnace. The hot gases are admitted to the flues at one side of the roof of the muffle and pass across the roof, down the opposite side, under the muffle, and up the other side to the main flues.
A. R. POWELL.

Muffle furnaces etc. E. A. HAILWOOD (B.P. 330,512, 4.3.29).—The walls of the muffle proper are constructed of metal frames with panels of thin sheet metal, such as rustless iron or steel, preferably corrugated.
B. M. VENABLES.

Heat-exchange device. G. KRÄNZLEIN, and SAMESREUTHER & Co., G.M.B.H. (B.P. 309,445, 8.4.29. Ger., 10.4.28).—A number of parallel tubes are connected to transverse tubes at the ends and the whole is sandwiched between two plates which may be either flat or corrugated and are provided with apertures for the outer fluid or for the leads of electric heaters.
B. M. VENABLES.

Steam generators, accumulators, etc. RUTHS-ACCUMULATOR AKTIEBOLAGET (B.P. 306,135, 7.2.29. Ger., 17.2.28).—In a steam accumulator priming may be caused by steam evolved from irregularities in the lower part passing through superheated water in the upper part and causing violent ebullition of the latter. In this invention evolution of steam is promoted originally in the upper part by means of injected gas bubbles or by causing the water to circulate through steel brushes or other spiky device. [Stat. ref.]
B. M. VENABLES.

Apparatus for carrying out endothermic catalytic gas reactions. I. G. FARBENIND. A.-G. (B.P. 330,872, 9.12.29. Ger., 15.12.28).—The catalysing units comprise return-flow, twin-concentric tubes; the closure of the return end of the outer tube is detachable and protrudes outside the heating chamber or furnace, and the other end only is secured to the furnace wall.
B. M. VENABLES.

Centrifugal apparatus for effecting physical or chemical changes [e.g., evaporation of liquids]. J. E. NYROP (B.P. 330,805, 17.7.29).—A liquid is spread centrifugally in a thin layer on the interior of a rotating cylinder, and gases or vapours are caused to pass in intimate contact with it by means of an interior device which is either stationary or rotated in the opposite direction, or is shaped so that the gases acquire an eddying motion.
B. M. VENABLES.

Tube mills. MASCHINENBAU-ANSTALT HUMBOLDT (B.P. 310,342, 23.4.29. Ger., 23.4.28).—In a tube mill with internal sifting means from which the ground material is removed by air, the stream of air is divided into two currents, one of which expands into the grinding space and picks up the material; the other passes straight through the mill in an axial pipe and does not entrain any dust until it mixes with the other stream in the

outlet pipe and produces a strong current for delivery. A damper is provided in the axial pipe to regulate the second stream.
B. M. VENABLES.

Pulverising machines. L. MELLERSH-JACKSON. From JEFFREY MANUF. Co. (B.P. 330,802, 12.7.29).—A disintegrator is provided with an inlet and a pocket for uncrushable material at the upper periphery and an abutment between the two. Within the pocket are shelves with upturned edges or like devices to retain the uncrushable material flung off the abutment.
B. M. VENABLES.

Drying of [expressing liquid from] pulp and the like. BLAIR CAMPBELL & McLEAN, LTD., and S. HUTCHIN (B.P. 330,663, 20.3.29).—The apparatus comprises a vessel with a rising strainer bottom and with a lid that can be swung aside also formed as a strainer. After filling, the bottom is pressed upwards by a hydraulic ram, and, after pressing, the lid is swung aside and the upward motion continued to discharge the cake. The apparatus is suitable for use as a filtering medium in the brewing industry.
B. M. VENABLES.

Automatically regulating the concentration of materials [e.g., paper pulp] suspended in liquids. T. KÄLLE (B.P. 307,300, 4.3.29. Swed., 3.3.28).—The control device is situated in the outlet pipe of a pump and the additional water or other diluent is added to the inlet pipe so that the pump forms an effective mixer. A portion of the mixed liquid is drawn through a by-pass to the main pipe by means of a propeller that produces a forward and rotating movement; the greater the concentration of the fluid, the less is the rotation. After passing through an orifice situated within the pipe, the fluid impinges on a flexibly supported target; the outer end of the support operates electrical contacts the current through which controls the supply of dilution water.
B. M. VENABLES.

Screening devices for liquids. H. J. VAN DER BIJL (B.P. 330,576, 11.3.29).—A screen (usually drum-shaped) for circulating water or other liquids is formed with apertures of which the diameter is not greater than their length in the direction of flow and is less than the diameter of the tubes of the condenser or other device to be protected. The apertures may be formed by drilling holes in a thick plate or by fastening short pieces of tube in a thin plate; the device is intended to catch long splinters, leaves, etc. that might pass through ordinary mesh-work.
B. M. VENABLES.

Emulsifiers, mixers, etc. H. A. HARRISON (B.P. 330,314, 12.3. and 4.6.29).—The material is forced by a plunger through a cylinder head or plug having a number of concentric grooves connected by small-bore passages which are preferably not quite radial, so as to produce a swirling action.
B. M. VENABLES.

Treatment of liquids with gases. KUPFERHÜTTE ERTEL, BIEBER & Co. (B.P. 308,254, 4.3.29. Ger., 20.3.28).—For aerating a liquid contained in a tank (or treating it with a gas which forms the atmosphere at the surface of the liquid), the liquid is circulated from the bottom of the tank, and then through a pump which forces it through a jet downwardly directed upon the surface of the liquid.
B. M. VENABLES.

Effecting intimate contact between gases and liquids. J. R. ANGER (B.P. 330,770, 14.6.29).—The gas is blown upon the surface of a foam-producing liquid below an inclined baffle in the containing chamber which forms, above it, a quickly expanding space for the outgoing gases and is provided with gutters which lead muddy condensed foam to a sump. B. M. VENABLES.

Continuous gas dryer. (SIR) G. C. MARKS. From H. A. BRASSERT & Co. (B.P. 330,284, 5.3.29).—A pair of moisture eliminators are connected to the mains by water-seals which are emptied to bring an eliminator into use, and filled when it is desired to cut one out for cleaning. B. M. VENABLES.

Devices for distributing air or other gases. Apparatus for separating solid substances from gases. ANEMOSTAT, LTD., and L. M. HIRCHBERG (B.P. 330,270 and 330,713, [A] 4.3.29, [B] 4.5.29).—(A) A device comprising a number of conical sheet guides, of common axis but differing angle, is rendered self-cleaning from adherent dust by dividing the cones into sectors each of which is pivoted near its narrow end and has the centre of gravity (when clean) brought to the axis of the pivot by addition of a counter-weight at the point. Any dirt accumulating will cause the sectors to hang vertically when the air current is off, but they will swing up towards their normal angle when the air current restarts, the resultant shaking dislodging the dust. (B) The gases are admitted downwardly at one end and exhausted upwardly at the other end of a tank which has a baffle depending from the top and a layer of fluid on the bottom. Distributing devices as described in (A) are used at both entrance and exit. B. M. VENABLES.

Means for extracting dust, grit, and like solid matter from gases. O. STOTT, and MATTHEWS & YATES, LTD. (B.P. 330,405, 11.6.29).—The gases are passed in a curved path between a number of curved plates and are moistened by sprays at the entrance and, if desired, intermediately. The cleaner may surround or be adjacent to a suction fan, the outlet of the former acting as the inlet of the latter.

B. M. VENABLES.

Methods and apparatus for gas analysis. I. FAG-ELSTON (B.P. 330,799, 12.7.29).—A sensitive form of viscosity-effusion bridge and accessories are described for comparing a standard gas with a sample. The regulating and indicating manometers are across the ends of the capillaries; beyond the latter, but before the gases unite, are orifices, preferably of rather smaller diameter than that of the capillaries, causing a decrease of pressure of at least 0.5 and preferably 0.6 of the pressure at entry to the orifices. The apparatus is suitable for all pressures and will indicate 2–3 mm. of water for every 1% change of oxygen in an oxygen-air mixture. B. M. VENABLES.

Substantially suppressing phosgene-formation when extinguishing fires with carbon tetrachloride. I. G. FARBERIND. A.-G. (B.P. 319,320 and Addn. B.P. 331,147, [A] 19.8.29, [B] 30.9.29. Ger., [A] 20.9.28, [B] 11.10.28).—(A) Alkyl or aryl phosphates (e.g., 1–3% of tritolyol phosphate), 0.1–3% of ammonia,

and, if desired, small quantities of hydrocarbons, or (B) animal or vegetable oils or fats (e.g., 2–4% of coconut oil or beef fat), alkyl or aryl phosphates, and ammonia, are added to the carbon tetrachloride (cf. B.P. 317,843; B., 1930, 169). L. A. COLES.

Heat-exchange apparatus. C. W. STANCLIFFE (U.S.P. 1,769,808, 1.7.30. Appl., 2.11.27. U.K., 12.11.26).—See B.P. 286,757; B., 1928, 352.

Cooling towers. J. M. SEYMOUR (B.P. 331,427, 28.8.29).—See U.S.P. 1,739,867; B., 1930, 398.

Grinding and mixing apparatus. R. O. CHILD, ASST. to D. ANDERSON & SONS, LTD. (U.S.P. 1,770,459, 15.7.30. Appl., 1.7.29. U.K., 20.7.28).—See B.P. 302,561; B., 1929, 267.

[Rotary drum] machine for treating granular material. H. BEHRENS, ASST. to MASCHINEN- U. WERKZEUGFABR. KABEL VOGEL & SCHEMMANN A.-G. (U.S.P. 1,769,670, 1.7.30. Appl., 27.3.29. Ger., 11.10.27).—See B.P. 322,405; B., 1930, 125.

Mixing and emulsifying apparatus. A. W. EMPSON (U.S.P. 1,770,302, 8.7.30. Appl., 25.6.27. U.K., 26.6.26).—See B.P. 282,466; B., 1928, 175.

Treatment of pulp. W. C. WEBER, ASST. to DORR Co. (U.S.P. 1,770,353, 8.7.30. Appl., 5.12.25).—See B.P. 262,479; B., 1928, 40.

Rotary spraying separator. F. H. LUENSE, ASST. to ROTOSPRAY MANUF. Co. (U.S.P. 1,769,588, 1.7.30. Appl., 4.5.27).—See B.P. 289,847; B., 1929, 268.

Liquid mixture of low f.p. G. REINHART, JUN., ASST. to GES. F. KÄLTECHEMIE GES. (U.S.P. 1,768,632, 1.7.30. Appl., 3.10.28. Ger., 17.11.27).—See B.P. 322,859; B., 1930, 127.

Air-heating installation for [boiler] furnaces. J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 331,583, 6.4.29 and 3.2.30).

Heat-exchanging means particularly applicable to refrigerators. GEN. ELECTRIC Co., LTD., and F. H. BRITTAIN (B.P. 331,565, 4.3.29).

Absorption refrigerating machine with neutral circulating gases. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 304,122, 29.12.28. Ger., 14.1.28).

Refrigerating methods and apparatus. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 308,692, 26.3.29. Ger., 26.3.28).

Absorption refrigerating machines applicable also for heating purposes. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 309,443, 21.3.29. Ger., 10.4.28).

Condensers for refrigerating systems. BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of C. STEENSTRUP (B.P. 331,449, 3.10.29. U.S., 4.10.28).

Containers for storing and transporting liquefied gases and applicable for cooling air or other gases. S. BRAUNSTEIN (B.P. 308,621, 23.3.29. Ger., 24.3.28).

Frothing agents (B.P. 329,622).—See III. Heating of gases (B.P. 307,027).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coal-washing investigations—methods and tests.

H. F. YANCEY and T. FRASER (U.S. Bur. Mines Bull. 300, 1929, 259 pp.).—The results of an investigation of the washing characteristics of typical bituminous coals from the eastern, central, and western coalfields of the United States are described. The advantages of clean coal over raw coal and the effect of impurities in coal on its utilisation are discussed. The relation of the coal-bed structure, the physical and chemical impurities in the coal, and its washability are described. Systematic methods for testing the washability of a coal and the factors to be considered in the construction of a satisfactory cleaning plant are given. The sp. gr. of U.S. coals and the physical forms of their impurities vary greatly, consequently they vary in washing characteristics and in the method of treatment required. The sp. gr. of a coal is the chief factor on which ease or difficulty of washing depends. The two most important impurities in coal, viz., ash and sulphur, are not always associated in equal degree in the same sp.-gr. fraction of the raw coal, hence reduction of ash and reduction of sulphur are often separate and distinct problems. Methods for the treatment of fines and the results obtained are reviewed and the following subjects are briefly discussed: minimum sizes cleaned in jig and table operation, re-washing of fines, re-treatment of table middlings by hindered-settling operations, advantages of pretreatment, size classification, rheolaveur, Trent and froth-flotation processes, methods of drying washed coals, and dewatering tests of slurry with vacuum filters. C. B. MARSON.

Sp. gr. and proximate composition of some Indian vitrains. L. L. FERMOR (Fuel, 1930, 9, 246—265; cf. B., 1929, 155).—Indian vitrains from the following stratigraphical horizons have been analysed: Barakar, Raniganj, Eocene, Miocene, and Siwalik. Taking all the data used, the limits of moisture are 0.63% and 16.61%, and the limits of sp. gr. are 1.214 (moisture 2.25%) and 1.414 (moisture 15.15%), calculated on the ash-free basis; the ash contents varied from 0.32% to 6.22%. Most of the vitrains with less than 9–10% of moisture are caking, whilst those of higher moisture content have no caking power; there is no correlation between caking properties and either volatile matter or fixed carbon content. The sp. gr. increases with the moisture content, irrespective of the age of the coal or of variations of moisture content on keeping the specimen. The relation between sp. gr. and moisture content is discussed in detail from the viewpoint of the colloidal character of vitrain. Within each coalfield the analyses show an orderly progression of properties in stratigraphical sequence; no such orderly progression is observed, however, when the whole of the data are so arranged. It is concluded that the variation in character is due to changes in the conditions of original deposition rather than to subsequent metamorphism. A. B. MANNING.

Chemical examination of lignite. W. FUCHS (Brennstoff-Chem., 1930, 11, 205—208).—A number of samples of lignite from the surface workings of a brown-coal mine at Türrnich have been analysed. When the methoxyl, lignin, cellulose, and humic acid contents

(on the ash- and bitumen-free basis) are plotted the results show clearly the general direction of the processes occurring in the gradual transformation of wood through lignite to brown coal, *e.g.*, the progressive diminution in the percentage of cellulose, the accompanying increase in the percentage of lignin and methoxyl, and the transformation of lignin into humic acids (cf. B., 1930, 540). Only one sample deviated markedly from the general scheme; this was characterised by a high content of bitumen (36.4%), which had apparently retarded the decomposition of the cellulose and to some extent the transformation of the lignin.

A. B. MANNING.

Coal-dust explosibility factors indicated by experimental mine investigations, 1911—1929. G. S. RICE and H. P. GREENWALD (U.S. Bur. Mines Tech. Paper 464, 1929, 45 pp.).—The knowledge gained from experiments on the explosibility of coal dust under various conditions and from studies, following explosion disasters, is classified and summarised and methods for minimising or preventing mine disasters are described (cf. B., 1925, 433; 1926, 178). C. B. MARSON.

Production of fuels from brown coals and waste bituminous coals in the Lurgi plant. O. HUBMANN (Brennstoff-Chem., 1930, 11, 219—222).—The coal is carbonised in a vertical shaft wherein it is brought into direct contact with the heating gases. Units of this type have now been constructed capable of carbonising 200 tons per day. The semi-coke is briquetted, using as binding agent the pitch produced in the process supplemented, as is generally necessary, with other coal-tar pitch, or asphalt. It is preferable to separate the finer material from the raw coal before carbonisation and use this directly for steam-raising and power production; the throughput of the retort is thereby increased, whilst the ash content of the coke and the free carbon in the tar are lowered. The semi-coke should be allowed to come into contact with air before it is briquetted in order that its tendency to spontaneous ignition should be diminished by a limited oxidation. The semi-coke has a marked wearing action on the transporting and briquetting apparatus; the conveyors are preferably made of rubber, and the briquetting machine of the type forming rectangular briquettes. A considerable economy in pitch is achieved by mixing the coal with the hot atomised pitch in a suitable apparatus, the necessary proportion of pitch being about 7.5%. If desired, the larger-sized coke may be separated and marketed without briquetting. The briquettes behave well in the fire and are ideal for the open grate. Although high in sulphur (2%), 70% of this is non-volatile and is retained in the ash. A. B. MANNING.

Dehydration of manufactured gas. F. W. SPERR (Fuel, 1930, 9, 266—281).—The various methods available for drying gas, *e.g.*, by compression, by refrigeration, by treatment with hygroscopic substances, or by treatment with adsorbents, are described and compared (cf. B., 1927, 243). Several methods are available, the cost of which should not exceed 1 cent per 1000 cub. ft. The importance of oil-fog lubrication as a protection against corrosion in the distribution of either moist or dry gas is emphasised. A. B. MANNING.

Removal of sulphur dioxide from flue gases.

A. THAU (Brennstoff-Chem., 1930, 11, 222—226).—By scrubbing the flue gases from a plant burning a North-umberland or Scottish coal, of sulphur content 3.9% and 1.23% respectively, with cold water, complete removal of the sulphur dioxide was effected when the time of contact between the water and the gases was 12 sec., corresponding with a water consumption of 45 tons per ton of coal fired. The sulphur content of the flue gases, expressed as H_2SO_4 , varied from 1—5 mg./m.³ To avoid too great a cooling of the flue gases it was found preferable to heat the water used for scrubbing; this had the further advantage of greatly accelerating the oxidation of the sulphur dioxide to trioxide, so that the water remained almost odourless. A design for a flue gas washing plant suitable for the proposed power station at Battersea is described.

A. B. MANNING.

"Flüssiggas." C. GERDES (Chem.-Ztg., 1930, 54, 509—510).—"Flüssiggas" is the fraction of oil-gas which may be made available for transport by compression into steel cylinders at ordinary temperatures, the resulting liquid contents of the cylinder reverting to gas on releasing the pressure. The properties and composition of the gas are discussed. The preparation of the crude gas by cracking oil in retorts or oil-gas generators (for large quantities) is described, and the subsequent elimination of difficultly condensable gases (chiefly methane, with some hydrogen and traces of carbon monoxide) and undesirable, easily liquefied hydrocarbons, is outlined. Apparatus of the Linde type may be used for fractionating the crude gas. The application of "Flüssiggas" for the lighting of buoys, railway carriages, etc. is discussed, and its use for aircraft propulsion suggested. The heating and storage properties of the gas are compared with those of coal gas, oil gas, and acetylene.

H. E. BLAYDEN.

Analysis of complex gaseous mixtures. W. L. WOOD (Fuel, 1930, 9, 288—291).—A preliminary analysis is carried out in the Bone and Wheeler apparatus. Attention is directed to the following possible causes of error: (a) the appreciable solubility of the higher hydrocarbons in the dilute acid used for washing out the absorption vessel, (b) the absorption of olefines by ammoniacal silver chloride when this is used to determine acetylene, and (c) the solubility of the paraffin hydrocarbons in the ammoniacal cuprous chloride used to absorb the carbon monoxide, all traces of which must be removed to avoid subsequent poisoning of the colloidal palladium used for the absorption of the hydrogen (cf. Beet, B., 1928, 113). Acetylene is removed by a 20% solution of mercuric cyanide in 2N-potassium hydroxide. The higher olefines are removed separately by using sulphuric acid of d 1.80, and introducing a suitable correction for the absorption of ethylene. The first stage of the more complete analysis consists of the removal of hydrocarbons higher in the series than butane and butylene, by cooling the gas in an ice and salt freezing mixture, and subsequently allowing the condensed liquid to warm up to 10° to eliminate dissolved gases. A known volume (5—6 litres) of the gas is then passed through alkaline mercuric cyanide to remove acetylene, through bromine to remove olefines, and

through a tube cooled to -76° (solid carbon dioxide and ether), wherein the propane and butane are condensed. The residual gas, and the mixture of propane and butane obtained by volatilising the condensed gas, are analysed by explosion with air or oxygen. The olefines are recovered from their bromides and determined by a slight modification of the method described by Manning, King, and Sinnatt (B., 1928, 217).

A. B. MANNING.

Position of the activated carbon process for the recovery of benzol from coke-oven gas and coal gas. E. REISEMANN (Brennstoff-Chem., 1930, 11, 226—229).—Some account is given of the Bayer process, and of the experiments carried out by the Gas Light and Coke Co. (cf. B., 1930, 42; Hollings and others, *ibid.*, 43).

A. B. MANNING.

Tower-absorption coefficients. III. Absorption of benzene by mineral oil. C. W. SIMMONS and J. D. LONG (Ind. Eng. Chem., 1930, 22, 718—721; cf. Cantelo and co-workers, B., 1927, 799).—The absorption by petroleum wash-oil of benzene from air as the inert carrier has been investigated. The rate of absorption varies linearly with the rate of flow of the absorbent; it decreases rapidly with an increase in flow ratio until a critical flow ratio is reached, after which it remains constant.

L. S. THEOBALD.

Artificial ageing of mineral oils. F. EVERS and R. SCHMIDT (Brennstoff-Chem., 1930, 11, 214—219; cf. B., 1928, 841).—The method of testing insulating and turbine oils previously described has been further developed. The oil (15.5 g.) is uniformly distributed over the catalyst (60 g. of silica impregnated with 1.59 g. of ferric oxide) contained in a glass tube, which is immersed in a water-bath at 100°; the tube is connected to an apparatus supplying electrolytic oxygen, and the oxygen absorbed by the oil is determined at intervals over a period of about 100 min. The current passing through the electrolytic cell is used to control and to measure the quantity of oxygen supplied to the reaction tube. The graph obtained by plotting the oxygen absorbed against time is only slightly curved, and may be replaced by a straight line without appreciable error; the slope of this line is taken as a measure of the rate of ageing ("ageing constant") of the oil. With time expressed in min. and oxygen absorbed as amp.-min., the values for the ageing constant of good insulating oils averages 0.13; it should not exceed 0.21. The ratio of ferric oxide to silica used in the catalyst is that giving approximately the maximum ageing constant. The same constant is obtained if the ferric oxide is replaced by the equivalent amount of copper oxide; lead oxide gives lower figures. The tar value (V_t) and the saponification value (V_s) of an oil are related to its "age" (A), i.e., its oxygen absorption as expressed above, by the equations $V_t = 1.0 \times 10^{-3} AM$, and $V_s = 0.56A$, where M is the mean mol. wt. of the oil.

A. B. MANNING.

Artificial ageing of mineral oils. III. F. EVERS and R. SCHMIDT (Wiss. Veröff. Siemens-Konz., 1930, 9, [1], 357—368; cf. B., 1928, 841).—Using the catalyst and procedure previously recommended for ageing tests on mineral oils, it is shown that the ageing curve

may be expressed by the equation $y = 2.3 \log x. C + K$, where $C = 10.1$ and $K = -11.3$. From the ageing constant of a mineral oil the saponification value, acid value, and tendency to polymerisation during various stages in ageing can be calculated, and a close approximation made of the average life of the oil under electrical conditions.

A. R. POWELL.

Decolorisation of oils with mixed adsorbents. J. K. CHOWDHURY and S. C. DAS (J. Indian Chem. Soc., 1930, 7, 379—400; cf. B., 1928, 324).—The mixed adsorbents studied—bauxite-silica, alumina-silica, and carbon-alumina—were prepared in each case by precipitating one component (silica or alumina) on the surface of the second, suspended in the liquid, followed by activation by roasting. The maximum decolorising power (for kerosene or ground-nut oil) with bauxite-silica mixtures corresponded with addition of 21.88% of silica, and with alumina-silica mixtures, 83.3% of silica. With activated carbon-alumina mixtures only a slight increase in efficiency was observed, but 30% of alumina may be added without loss of efficiency. The mixtures are readily regenerated by roasting, with unimpaired efficiency. The temperature of roasting (for regeneration) and the water content are interconnected, completely dehydrated mixtures having the lowest efficiency. The power of decolorisation attains a maximum with 6.33% of water for bauxite, 5.95% for the optimum bauxite-silica mixture, and 4.27% for the optimum alumina-silica mixture. The action of the mixed adsorbent is explained by assuming that the pores of one adsorbent are filled up with the second deposited on it, thus forming a large number of narrow capillaries, the two walls of which are oppositely charged. The water present may prevent the molecules of the adsorbed substance from coming into close contact with those of the adsorbent, whilst complete absence of water will affect the electrical properties and thus impair the adsorption capacity. J. R. I. HEPBURN.

Chemical examination of gasolines and light mineral oils. D. A. HOWES (J. Inst. Petroleum Tech., 1930, 16, 54—88).—The most important analytical methods that have from time to time been proposed are collected together, and these are now briefly described, examined, and criticised. The paper is divided into three parts, dealing with the determination of unsaturated, aromatic, and naphthene and paraffin hydrocarbons. A full bibliography is appended.

Motor-fuel volatility. II. Starting volatility. G. G. BROWN, C. L. NICKOLS, and P. BIGBY (Ind. Eng. Chem., 1930, 22, 649—652; cf. B., 1930, 544).
 III. Effective volatility under driving conditions. J. E. MILLER and G. G. BROWN (*Ibid.*, 653—662).
 IV. Relation between atmospheric temperature, fuel volatility, and engine performance. C. L. NICKOLS and G. G. BROWN (*Ibid.*, 662—671).
 V. Vapour pressure and vapour lock. E. A. CLARKE, H. B. COATS, and G. G. BROWN (*Ibid.*, 672—680).—II. Equilibrium volatility, as previously described (*loc. cit.*), is related to ease of starting as determined by actual engine tests.

III. Effective volatility under driving conditions is defined, and methods for its determination from the

equilibrium volatility or A.S.T.M. distillation data are described.

IV. Effective volatility is related to engine performance, and the A.S.T.M. distillation characteristics required for satisfactory performance under different conditions of atmospheric temperature are described.

V. The relation of vapour pressure to vapour lock is discussed, and fuel characteristics necessary to eliminate this trouble are suggested. L. S. THEOBALD.

Anti-knock fuels. L. EDELEANU and W. GROTE (Brennstoff-Chem., 1930, 11, 212—214).—The anti-knock value of a cracked spirit is greatly increased if the fraction boiling above 175° is extracted with liquid sulphur dioxide, and the extract, consisting principally of aromatic and unsaturated hydrocarbons, is recovered and added to the fraction boiling up to 175° (cf. U.S.P. 1,661,566; B., 1928, 326). Engine tests on such treated spirits show them to compare favourably in anti-knock value with straight-run spirits to which lead tetraethyl has been added. A. B. MANNING.

Detection and determination of carbon disulphide in fuels. F. SPAUSTA (Petroleum, 1930, 26; Motoren-betrieb, 3, No. 7, 7—10).—The presence of as little as 0.05% of carbon disulphide in such fuels as benzine, benzol, etc. may be detected by means of alcoholic caustic potash or soda, the xanthate formed being determined by means of an aqueous ferric chloride solution acidified with sulphuric acid. Even 1 mg. of iron xanthate dissolved in the fuel imparts a red colour. 5 C.c. of the fuel and 3 c.c. of *N*-caustic potash solution (in 96% ethyl alcohol) are shaken vigorously for 2 min., then diluted to dissolve the xanthate, and ferric chloride solution acidified with sulphuric acid is added. In the presence of carbon disulphide a brownish-black precipitate is formed, becoming red on shaking with the fuel. A colorimetric method of determination is given. Homologues of ethyl alcohol give the same results. In place of the ferric chloride, salts of nickel or cobalt may be used, the xanthates of which are yellow; nickel salts are soluble only when benzol, alcohol, or acetone is present, and cobalt salts require the addition of ether. The "doctor test" is unsuitable for the detection of small amounts up to 0.2% of carbon disulphide, owing to the slow rate of precipitation of the lead sulphide.

W. S. E. CLARKE.

Properties of tar-petroleum asphalt mixtures. P. SCHLÄPFER (Petroleum, 1929, 25, 1457—1464, 1585—1590).—Nine tars of varying characteristics have been blended in different proportions (10, 20, and 30%), with each of two petroleum asphalts. The physical properties of these tars and asphalts are tabulated and the more important characteristics of the mixtures, *e.g.*, viscosity and dropping point, are shown graphically in relation to varying temperatures and percentage of asphalt. Up to a 30% asphalt content the viscosity of the mixture is fundamentally a property of the original tar. In a second series of tests mixtures of asphalts and tars were made of 20, 40, 60, and 80% asphalt content. Irregularities in the curves for the 40% mixture suggest that disturbances occur. This is confirmed microscopically. Generally, tar-asphalt mixtures of less than

30% asphalt content were homogeneous under the microscope, with the carbon uniformly distributed, and did not change appreciably even after 1 year, but in individual cases poor ductility was shown, the carbon was flocculent, and emulsions were visible under the microscope. Mixtures containing 40% of asphalt showed, with few exceptions, a deposit of flocculent carbon and unevenly distributed emulsions. W. S. E. CLARKE.

Purification of normal paraffin hydrocarbons by chlorosulphonic acid treatment. A. F. SHEPARD and A. L. HENNE (Ind. Eng. Chem., 1930, 22, 356—357).—Aschan's method (B., 1898, 798) for the purification of normal paraffins has been applied. The decane fraction obtained from gasoline by fractionation was treated with chlorosulphonic acid, the mixture being stirred during the day and the used acid replaced by fresh every 3 or 4 days. After treatment for 4 weeks the material, on being fractionated, gave a fraction having d_4^{20} 0.7264, which compares well with values of 0.72686 and 0.7266 obtained by Simon and Krafft, respectively, on synthetic samples of decane. The authors state that this method of purification is superior to any other they have tried, and can be used to remove hydrocarbons with side chains from the normal paraffins. A. SHORE.

Direct oxidation of hydrocarbons by air. P. MONDAIN-MONVAL and B. QUANQUIN (Bull. Soc. Ind. Mulhouse, 1930, 96, 265—283; cf. B., 1928, 917).—Observations have been made of the changes of pressure and temperature which occurred when a mixture of a hydrocarbon and air was heated in a steel bomb. The bomb was of 700 c.c. capacity, and the initial pressure was 5.5 kg./cm.² Using petrol, no spontaneous ignition took place with less than 0.35 c.c. With mixtures containing between 0.35 and 0.8 c.c. a violent explosion accompanied by a marked rise in temperature occurred when the temperature reached 220—225°. With from 0.8 to 3.6 c.c. of petrol the sudden increase in pressure occurred at gradually falling temperatures, while the magnitude of the effect at first diminished, passed through a minimum, and thereafter again increased rapidly; also over this range carbon was deposited in the bomb. With 3.7 c.c. a remarkable discontinuity occurred in the phenomena observed; the pressure and temperature increase fell to a lower value, which was approximately constant for higher concentrations of the hydrocarbon, and the deposition of carbon no longer took place. This discontinuity corresponded also with a discontinuity in the composition of the combustion gases, and in the appearance of the flame in the bomb. In a second series of experiments the hydrocarbon-air mixtures were passed through a heated tube, at the ordinary pressure, and the oxidation products were examined. Interaction began at 160° and proceeded rapidly at 300°, with the production of carbon dioxide, alcohols, aldehydes (formaldehyde, acetaldehyde, butaldehyde, etc.), and acids. No true ignition of the mixture occurred even at 360°. It is concluded that the initial reaction in the bomb is of a similar nature, consisting of an exothermic reaction unaccompanied by flame; the temperature and pressure of the mixture then rise, and, if the concentration of the

hydrocarbon is within the requisite limits, ignition follows. A. B. MANNING.

Formation of methane from carbon monoxide and hydrogen by bacteria. R. LIESKE and E. HOFMANN (Brennstoff-Chem., 1930, 11, 208—212).—Bacteria have been found in the mud of ditches, in sewage sludge, etc. which bring about the reaction $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$. They are Gram-positive, non-spore-forming bacilli, without cilia, identical with or closely related to the carbon dioxide-reducing bacteria described by Söhngen (B., 1910, 980). Cultures of these bacteria convert the carbon dioxide and carbon monoxide of water-gas or coal gas into methane, the yield of which amounts to over 90% of the theoretical. These results have been obtained from experiments carried out in closed vessels; it has not yet been possible to bring about a similar conversion of carbon monoxide into methane when a continuous current of the gas is passed over the culture. A. B. MANNING.

See also A., July, 851, **Activity of charcoal** (ALEXEEVSKI and MUSAKIN). 868, **Catalytic reactions at high pressures** (EGERTON). 869, **Reduction of carbon dioxide** (BAHR). 875, **Graphite and graphitic acid** (THIELE; HOFMANN and FRENZEL).

Determination of hydrogen in gases. AMBLER.—See VII.

PATENTS.

Treatment of coal [especially anthracite]. DELAWARE, LACKAWANNA, & WESTERN COAL CO., ASSEES. OF (A) E. FARLEY and N. R. LANDON, (B) N. R. LANDON (U.S.P. 1,748,335 and 1,748,384, 25.2.30. Appl., [A, B] 25.2.29).—To restore the lustre and improve the appearance of anthracite or other coal which has become dull on storage, the material is treated with solutions of (A) oxalic or other acid, or (B) glycerin or dextrin, at the ordinary or at higher temperatures. A. B. MANNING.

Method of identifying fuel. H. S. MORK, ASST. TO DELAWARE, LACKAWANNA, & WESTERN COAL CO. (U.S.P. 1,748,787, 25.2.30. Appl., 17.1.28).—Coal is coloured for purposes of identification by dissolving a basic dye in water, adding a clay which has the property of adsorbing the dye from solution, and spraying the coal with the suspension of coloured clay. A. B. MANNING.

Fuel briquettes. J. S. FORD and F. MURRAY (B.P. 329,437, 18.3.29. N.Z., 17.1.29).—Coal dust, or other carbonaceous material, is mixed with 5% of its weight of molasses, the mixture is formed into briquettes without using appreciable pressure, and the briquettes are heated at 500—700° for 10—20 min. in order to draw a portion of the binding material to the surface of the briquettes where it is carbonised to form a hard compact crust. A. B. MANNING.

Recovery of waste heat in the drying of brown coal and the like. TELEX APPARATEBAU-GES.M.B.H. (G.P. 462,779, 3.2.24).—A condensing system is provided wherein the heat in the steam evolved during drying is transferred to air, and the warm air is then used in the preliminary drying of the raw brown coal supplied to the plant. A. B. MANNING.

Heating coal and like furnaces. W. SCHAEFER and W. POHL, ASSTS. TO KERAMISCHE IND.-BEDARFS A.-G.

(U.S.P. 1,747,846, 18.2.30. Appl., 26.10.26. Ger., 13.2.25).—Separate currents of cold and hot air are introduced into the combustion gases supplied for heating the furnace at a point prior to their introduction into the furnace. The amount and temperature of each of the air currents are regulated in such a way that any desired length of flame and a uniform temperature distribution are attained within the heating zone. A. B. MANNING.

Apparatus for mixing air and pulverised coal applicable to other similar operations. G. H. ROBINSON (B.P. 329,483, 27.4.29).—The apparatus comprises a Venturi tube, through which the air is made to pass, and a device for spraying the pulverised fuel into this tube through an aperture therein at the point of minimum cross-section. The spraying device consists of a wire-brush rotating within a cylinder into which the powdered coal is fed from the storage bin.

A. B. MANNING.

Energy production and distribution in the working up of brown coal. P. SCHMIDT (G.P. 462,388, 14.11.25).—The electrical energy produced by the utilisation of the steam from a brown coal drying plant is used to produce hydrogen by electrolysis under pressure, and the hydrogen is then transported to the points of consumption by high-pressure distance transmission.

A. B. MANNING.

Coal-distillation and coke-production plant. BABCOCK & WILCOX, LTD., E. G. WEEKS, and W. A. RILEY (B.P. 329,441, 23.3.29).—The coal is delivered from a bunker to a preheater and thence to a retort provided with means whereby the rate of extraction is controlled by the movement of a float on the surface of the fuel bed (cf. B.P. 324,853; B., 1930, 357). The coke is discharged into a quenching device in the form of a closed container (cf. B.P. 324,852; B., 1930, 357), wherein the cooling of the coke is completed by the admission of a controlled quantity of air. The process proceeds automatically from the delivery of the coal to the bunker to the discharge of the quenched coke out of contact with air other than that intentionally supplied.

A. B. MANNING.

Carbonisation of bituminous materials. DEMAG A.-G. (G.P. 462,876, 3.9.24).—Bituminous or brown coal etc. is first converted into semi-coke by low-temperature carbonisation in known manner, and is then subjected to further carbonisation at above 600° in a special chamber. The heat required for the second stage of the process is provided by the combustion of the necessary quantity of the still hot semi-coke produced in the first stage.

A. B. MANNING.

Low-temperature distillation of bituminous coal and lignite. COMP. GÉN. DE DISTILLATION ET COKÉFACTION À BASSE TEMPERATURE ET MINIÈRE (INTERTRUST) SOC. ANON., Assees. of INTERNAT. HOLDING DE DISTILLATION ET COKÉFACTION À BASSE TEMPERATURE ET MINIÈRE (HOLCOBAMI) SOC. ANON. (B.P. 329,542, 17.6.29. Belg., 15.3.29).—The material, in layers not greater than 8–10 cm. thick, is heated gradually until the outside temperature reaches 450°, and this temperature is maintained until that of the coolest part of the material reaches 350°. The temperature is then quickly raised to 500–550°. The process, which is most conveniently

carried out in a retort of the type described in B.P. 229,880 (B., 1925, 345) or B.P. 308,760 (B., 1929, 841), yields a hard, compact, and easily ignitable semi-coke, a low-temperature tar which has undergone no cracking, and a gas of high calorific value. A. B. MANNING.

Retorts for the low-temperature distillation of carbonaceous material. CARLTON MAIN COLLIERY CO., LTD., R. ADDY, and S. GILL (B.P. 329,464, 11.4.29).—Each retort consists of two vertical, slightly coned, concentric, metal chambers, the coal being charged into the narrow annular space so formed. The inner chamber is movable vertically and is provided at its lower end with a projection which serves to close the lower end of the annular space during carbonisation. When carbonisation is complete the inner chamber is lowered to permit discharge of the coke. The outer surface of the inner chamber may be provided with vertical ribs which divide up the annular space into vertical compartments for receiving the coal. The wall of the inner chamber is perforated by upwardly directed slots for the passage of a portion of the distillates into the interior of the chamber and thence to the offtake. The retorts are mounted in a brickwork setting and adapted to be heated externally, or both externally and internally, by heating gases. A. B. MANNING.

Gas producer. J. U. M. VOITEUR (B.P. 329,573, 30.7.29. Fr., 29.4.29).—A gas producer for use with moist fuels, particularly wood, operates with reversed draft, the air entering through apertures in the wall of the shaft at a suitable height above the grate, and the combustible gas being withdrawn through a conduit opening into the ash pit. The upper part of the fuel shaft is connected with a condenser by means of an elbow-coupling hinged so that it may be moved aside laterally to permit charging.

A. B. MANNING.

Production of gas of high calorific value, using bituminous coal in a gas producer. R. NÜBLING and R. MEZGER (G.P. 462,309, 6.5.25).—In a plant in which the waste heat is used for preheating the circulating gases and for superheating the steam in known manner, the "blow" period is replaced by a "half-water-gas" period by adding steam to the air. The heat produced by the combustion of this gas and used for preheating purposes is thereby increased; the ratio of carbonisation gas to water-gas, and therefore the calorific value of the resultant gas, are correspondingly increased.

A. B. MANNING.

Gas producer for wet brown coal. A. KADEN (G.P. 462,607, 5.4.25).—The producer is provided with a ring-shaped, internally and externally heated, carbonising and drying chamber, and with separate off-takes for the carbonisation and producer gases. Below the grate is a rotatable ash-tray which forms also a water seal. A vertical grate surrounds the gasification zone and connects the inner part of the shaft with the ring chamber. The producer gas off-take is formed by a central, telescopically adjustable tube.

A. B. MANNING.

Production of enriched water-gas. A. SCHWARZ, ASSR. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,745,454, 4.2.30. Appl., 18.11.25).—The apparatus comprises two generators, an oil still and circulating pump, a

steam generator, and preheating devices for the oil and steam. The heat of the "blow" gases is used to distil the oil, the heavy unvaporised portion of which is introduced into the generator during the "run" in order to enrich the water-gas produced. The residual heat in the blow gases serves to generate the steam which is superheated by being passed through tubes in the furnace of the oil still. A. B. MANNING.

Purification of coal-distillation gas. GEWERKSCHAFT M. STINNES (B.P. 304,145, 14.1.29. Ger., 14.1.28).—Coal gas which is intended for long-distance transmission is compressed, charged with a naphthalene solvent, and then cooled to such a temperature that the solvent separates out again together with the naphthalene. The solvent may also extract traces of benzene from the gas, and the cooling may be carried to such a point that the gas is also dried. A. B. MANNING.

Purification of gases [e.g., coal gas] containing hydrogen sulphide. W. D'LENY, J. R. PARK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,381, 15.5.29).—The gases are scrubbed with an alkaline suspension of 0.02 g.-mol. of ferric hydroxide and 0.005 g.-mol. of nickel carbonate per litre of solution containing 0.15 g.-mol. of sodium carbonate. A. R. POWELL.

Removing soot from gases. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. DANTSZEN (B.P. 306,043, 14.2.29. U.S., 14.2.28).—The soot is electrically precipitated and is conveyed by the circulation of a current of water to a quiescent pool in which the soot particles are separated by flotation and from which they are removed intermittently or continuously. In order to avoid the accumulation of soluble products in the water, a small fraction may be continuously withdrawn and replaced by fresh water. The flotation of the soot may be assisted by introducing oil in atomised form into the smoke to be treated. A. B. MANNING.

Separation of ammonia and benzol hydrocarbons from coal gases containing hydrogen sulphide, carbon dioxide, and oxygen. GES. F. KOHLENTCHNIK M.B.H., Assees. of F. HÄUSSER (G.P. 462,186, 8.7.21).—The corrosive components of the gas, ammonia, hydrogen sulphide, and carbon dioxide, are removed first, in apparatus constructed of chemically resistant material but possessing small mechanical strength. The separation of the benzol is carried out subsequently under pressure in mechanically stronger apparatus constructed of less chemically resistant material. Methods of carrying out the process are described. A. B. MANNING.

Distillation of wash oil. ZEHE M. STINNES (G.P. 462,592, 18.3.24).—The wash oil is preheated in an apparatus offering as large a surface as possible to the oil, which is thereby freed from gases and the lower-boiling constituents. It is then further heated and distilled in the usual manner. Heat recovered from the vapours or from the hot oil is utilised in the preheating. The gases and vapours evolved are then fractionally condensed. The oil, freed from gases and low-boiling constituents, or fractions thereof, are washed in vessels serving also as coolers. A. B. MANNING.

Mine-gas detector. E. HECKERT (U.S.P. 1,746,425, 11.2.30. Appl., 16.10.24. Ger., 25.10.23).—The apparatus comprises a number of diffusion cells, each consisting of a pair of cell chambers separated by a porous division wall, a pressure gauge, and means for connecting one chamber of each pair to the pressure gauge while the gas to be tested is simultaneously admitted to the other chamber. The difference in pressure gives a measure of the firedamp in the mine air being tested. A. B. MANNING.

Apparatus for production of carbon black. J. MACHTOLF, ASSR. to C. HOSTMANN-STEINBERG'SCHE FARBENFABR. G.M.B.H. (U.S.P. 1,746,003, 4.2.30. Appl., 9.12.26. Ger., 11.12.25).—An apparatus for splitting acetylene to produce carbon black consists of a number of pipes capable of withstanding high pressures, arranged horizontally one beside the other, and connected at their ends to form one continuous pipe. The acetylene is introduced into the apparatus under pressure, and is decomposed by means of an electric spark. The apparatus is preferably immersed in a cooling bath. A. B. MANNING.

Manufacture of products from ethylene and/or its homologues. H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 331,186, 18.3.29).—The ethylene etc. is subjected to polymerisation treatment under pressure and at elevated temperatures in the absence of catalysts but in the presence of an inert liquid medium such as mineral or vegetable oils or products obtained therefrom which remain substantially unchanged under the conditions of the reaction. H. S. GARLICK.

Conversion of hydrocarbons into lighter hydrocarbons. A. LOSEY, P. GOTTLIEB, and L. L. HAUPT (B.P. 330,570, 11.3.29. U.S., 7.1.29).—A hydrocarbon distillate and a suitable conversion catalyst, e.g., a volatile metallic chloride, is held under superatmospheric pressure while heated by indirect contact with a flow of vaporised "bottoms" under a lower pressure, being fractionally condensed thereby. The resulting vapours are subjected to thermodynamic expansion, the uncondensed vapours are drawn off, and the condensate is returned for further conversion. The unvaporised portion of the original charge is recycled in heat-exchange relation with the separate flow of hydrocarbon vapours which are fractionally condensed thereby, the fractions being collected and withdrawn, the vapours drawn off and fractionally condensed, and the condensate recycled. The heaviest fraction formed by the loss of latent heat from the heating vapour is collected, withdrawn, and reheated, and the vapours are returned in fractionating relation with the hottest portion of the separate flow of hydrocarbon condensate, thereby forming further vapour consisting of a lighter series of hydrocarbons evolved from the high-temperature heating vapours. The "bottoms" formed by reheating the heaviest fraction is separately reheated to a higher temperature than that of the catalyst and the latent heat is employed to effect recuperation of the impaired catalyst and conversion of the original mixture. H. S. GARLICK.

Recovery of refined products from carbonaceous materials such as coal, tars, mineral oils, etc.

J. Y. JOHNSON. From I. G. FARBEININD. A.-G. (B.P. 329,331, 12.11.28. Cf. B.P. 328,997; B., 1930, 752).—The reaction products from the destructive hydrogenation of carbonaceous materials, or from the extraction under pressure of bituminous materials, are fractionated by passing them directly from the reaction chamber into several vertical columns wherein they are cooled by means of gases or liquids flowing countercurrent thereto. Thus the waste gases from the process may be used as the cooling agent by circulating them through tubes arranged within the fractionating columns. The fractionation may be effected without releasing the pressure on the reaction products. The heavier condensates may be returned to the reaction chamber for re-treatment. A. B. MANNING.

Catalytic hydrogenation of heavy hydrocarbons, tars, crude petroleum, etc. SOC. INTERNAT. DES PROC. PRUDHOMME-HOUDRY (F.P. 637,022, 26.10.26).—In order to prevent poisoning of the catalyst, and to remove any material deposited thereon, light hydrocarbon vapours, especially those of the lighter fractions of the hydrogenation product, are passed, periodically or continuously, through the hydrogenation chamber.

A. B. MANNING.

Production of hydrocarbons of low b.p. from coal, oil, etc. H. S. WAITE (B.P. 330,934, 20.2.29).—The volatile products from the distillation of coal are passed direct from the retorts into a chamber where they come in contact with absorbent material, such as coke, maintained at 400–550°. The coke is supported on travelling screens and is fed into and removed from the chamber at a regular rate. The yield of condensible hydrocarbons in the gases is increased. T. A. SMITH.

Transformation of petroleum hydrocarbons into gasoline. JENKINS PETROLEUM PROCESS Co. (B.P. 330,539, 8.3.29. U.S., 16.11.28).—A mixture of the hydrocarbons with an absorptive binder of ash-forming content is subjected to incipient cracking conditions and then transferred to a non-extraneously heated zone of reduced pressure where separation occurs into a readily convertible distillate vapour and a residuary compound in liquid form. This residuum is reheated under such conditions of temperature and pressure that when transferred to a further non-extraneously heated zone of suitably reduced pressure it separates into additional light vapours and leaves a liquid residue that on cooling solidifies to a dry, coal-like structure.

H. S. GARLICK.

Apparatus for cracking oils. W. S. HADAWAY, JUN., ASSR. to TEXAS Co. (U.S.P. 1,751,148, 18.3.30. Appl., 28.8.25).—The presence of free carbon in the oil in cracking stills is determined by the fall in electrical resistance of the oil. Electrodes are placed in a conduit through which the oil is being passed and the power absorbed is measured on a wattmeter. Increase in the power absorbed shows that it is necessary to remove the oil from the still if the deposition of carbon is to be prevented. Several such indicators placed in different parts of the plant enable the cracking operation to be carefully controlled. T. A. SMITH.

Simultaneously obtaining refined mineral oils and pure bitumen from crude mineral oils. N.V.

MIJNBOW-EN CULTUURMAATS. "BOETON" (B.P. 330,565, 9.3.29. Holl., 26.10.28).—Crude oil is treated with a concentrated solution of iron or zinc chloride (800 pts. of the salt to 200 pts. of water). The oil is separated and the unsaturated substances which remain in the salt solution are coagulated by agitation. The product, steamed or washed with boiling water, yields a "carbene"-free bitumen, 18% of the oil being so recovered. T. A. SMITH.

Manufacture of petroleum products of the type known as "white oils." STANDARD OIL DEVELOPMENT Co., Assees. of F. A. HOWARD (B.P. 308,297, 9.2.29. U.S., 21.3.28).—"Petrolatum liquidum" is prepared from hydrogenated crude oil. The oil is hydrogenated catalytically, if desired, at 360–455° and under 50–200 atm. A fraction of suitable viscosity (50–600 sec. Saybolt at 100° F.) is then treated with fuming sulphuric acid (10–50 vol.-%), washed, dried, and filtered through fuller's earth. T. A. SMITH.

Apparatus for refining used lubricating oil. W. D. HARRIS and R. V. AYCOCK (B.P. 331,290, 10.4.29).—Oil from a still is discharged below a perforated contact plate, supporting a layer of fuller's earth and a neutralising agent, and passes upwardly through the clarifying material. Reaction with the fuller's earth causes coagulation of a sludge which sinks through openings in the contact plate and rests on and is supported by a screen. The oil is filtered by opening a valve and allowing the oil to percolate through the bed of coagulated sludge. H. S. GARLICK.

Manufacture of motor fuels. J. Y. JOHNSON. From I. G. FARBEININD. A.-G. (B.P. 330,593, 9.1.29).—Approx. 1–2 pts. of liquid hydrocarbons, b.p. up to 105°, recovered from natural or cracking gases is blended with 3–4 pts. of the heavy benzine, b.p. 100–180°, obtained by the destructive hydrogenation of carbonaceous materials. [Stat. ref.] H. S. GARLICK.

Manufacture of fuel mixtures. P. E. and F. A. WEBB (U.S.P. 1,746,172, 4.2.30. Appl., 7.5.28).—The fuel consists of a mixture of acetylene and pentane produced by passing the former through the liquid pentane and subsequently adjusting the mixture ratio by adding a further quantity of acetylene. Other volatile liquid paraffin hydrocarbons may be used in place of pentane. A. B. MANNING.

Refrigeration of gases for recovery of gasoline. F. E. HOSMER (B.P. 309,121, 14.1.29. U.S., 5.4.28).—In systems in which gasoline is recovered from natural gas by refrigeration, the formation of ice from the moisture contained in the gas is prevented by bringing the gas in direct contact with cold brine, which removes the moisture and cools the gas. The gas is then further compressed and cooled to remove the gasoline. After removal of the gasoline the residual gas is passed through the weakened brine solution to remove water and reconcentrate the brine for further use. The traces of water which remain in the gas and are precipitated with the gasoline are prevented from freezing by the addition of a little brine. T. A. SMITH.

Drying or carbonising machine [for fuel agglomerates]. F. M. CROSSMAN (U.S.P. 1,769,491, 1.7.30.

Appl., 18.5.29. U.K., 24.5.28).—See B.P. 318,506; B., 1929, 968.

Manufacture of oil gas. K. N. WANNEBO (U.S.P. 1,770,563, 15.7.30. Appl., 26.3.26. Ger., 11.9.23).—See B.P. 269,711; B., 1927, 468.

Conversion of heavy mineral oils into lower-boiling products. W. G. LEAMON (U.S.P. 1,769,789, 1.7.30. Appl., 26.6.25).—See B.P. 317,868; B., 1929, 882.

Liquid fuel. A. LAURENT (U.S.P. 1,770,315, 8.7.30. Appl., 6.7.26. Belg., 21.11.25).—See F.P. 615,749; B., 1927, 835.

Clouderising coal dust for power purposes. C. H. VERITY (B.P. 331,221, 12.3.29).

Apparatus for making illuminating and heating gas [from volatile liquid fuels]. J. WHITEHART (U.S.P. 1,747,094, 11.2.30. Appl., 1.7.27).

Gas-analysis apparatus (B.P. 330,799).—See I. **Acetic acid from pyroligneous acid** (B.P. 330,026).—See III. **Treatment of non-fibrous materials** (B.P. 330,649).—See V. **Ammonium sulphate** (B.P. 330,945 and 330,947).—See VII. **Castor oil soluble in mineral oil** (B.P. 317,391).—See XII. **Stoving lacquers** (B.P. 329,954).—See XIII.

III.—ORGANIC INTERMEDIATES.

See A., July, 867—8, **Catalytic reactions at high pressures** (MORGAN; BONE; GREEN). 903, **Detection of some primary arylamines** (RUZICKA). 908, **Reaction of α -naphthol** (CARLETTI). 919, **Preparation of tri- and tetra-chloro-*p*-benzoquinones and *s*-trichloroaniline** (ERDELYI). **Synthesis of 1-methylantraquinones** (FAIRBOURNE and FOSTER). **Nitration of 2-hydroxy-3-methylantraquinone** (MITTER and PAL).

Direct oxidation of hydrocarbons by air. MONDAIN-MONVAL and QUANQUIN.—See II. **Insecticidal pyridine and pyrrolidine derivatives.** RICHARDSON and SHEPARD.—See XVI.

PATENTS.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 315,854, 29.5.29. U.S., 21.7.28).—The "base-exchange" catalyst preparations of earlier patents are effective after lixiviation with acid, which removes part or all the basic constituents, but leaves the porous physical structure.

C. HOLLINS.

Manufacture of butadiene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,396, 22.2.29).— γ -Butylene glycol vapour is passed with steam over a hydrating catalyst, composed of an oxide or borate (or a compound furnishing the oxide), with or without activators such as copper sulphate or red phosphorus, at 300°. Alumina, *e.g.*, gives 65% conversion when 30 pts. of the glycol vapour are mixed with 70 pts. of steam.

C. HOLLINS.

Production of 1:3- $[\alpha\gamma]$ -butadiene. C. J. LEYES (B.P. 329,748, 20.3.29).—Vinyl bromide vapour is

passed through a bath of molten tin, stannous bromide and $\alpha\gamma$ -butadiene being withdrawn as formed.

C. HOLLINS.

Production of acetylene dichloride of low b.p. from acetylene and chlorine. I. G. FARBENIND. A.-G. (B.P. 310,964, 29.4.29. Austr., 5.5.28).—*trans*-s-Dichloroethylene, b.p. 48—51°, is the main product when acetylene and chlorine are led over active carbon below 100° (optimum 40°). A high velocity is preferred, and the excess of acetylene is adjusted so that the unused chlorine does not exceed 5% in the exit gases after removal of the products. The exit gas is re-circulated with fresh acetylene and chlorine, which are conveniently added as equimolecular mixture.

C. HOLLINS.

Extraction of acetic acid from pyroligneous acid. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 330,026, 16.3.29. Belg., 25.1.29).—A single solvent is employed (*a*) to wash out tars, (*b*) to extract the acetic acid, the solvent being chosen so that the amount used in the first operation is only a small fraction of that used in the second; the amount of solvent to be recovered from the tar washing is thus minimised. Suitable solvents are amyl acetate, mesityl oxide, dipropyl ketone, cyclohexanones, or mixtures of these.

C. HOLLINS.

Manufacture of soluble wood ethers. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,011, 5.3.29).—A wood ether, obtained, *e.g.*, by heating with ethyl chloride under pressure a mixture of pine sawdust and 3 pts. of 50% soda lye, is made completely soluble in alcohol-benzene (1:1—9) by treatment with chlorine dioxide in aqueous or acetic acid solution. If water is used chalk may be added. The chlorine dioxide may be produced *in situ* from chloric acid by reduction with vanadous sulphate or electrolytically.

C. HOLLINS.

Manufacture of acetaldehyde [from acetylene]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,867, 12.6.29).—Acetylene and steam are passed at 300—400° over a tungsten catalyst, *e.g.*, silicotungstic acid on clay or ammonium tungstate ignited on clay or zinc oxide.

C. HOLLINS.

Manufacture of crotonaldehyde and its homologues. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 637,517, 12.7.27. Belg., 14.7.26).—Acetaldehyde (*etc.*) is aldolised with alkali at 5°, and the product is neutralised with phosphoric acid and distilled; aqueous crotonaldehyde passes over at 84—85° in 92% yield on unrecovered acetaldehyde.

C. HOLLINS.

Manufacture of acetone from acetylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,350, 13.4.29).—In the catalytic production of acetone from acetylene the gases emerging from the catalyst chamber (which may contain, *e.g.*, zinc oxide at 400°) are passed without cooling over a similar catalyst to convert acetic acid, acetaldehyde, and unconverted acetylene into acetone.

C. HOLLINS.

Manufacture of organic acyl halides. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 329,721, 1.3.29).— α -Halogeno- and $\alpha\beta$ -dihalogeno-ethyl

esters, obtained, *e.g.*, by addition of hydrogen halide and halogen, respectively, to vinyl esters, are distilled slowly with an acid catalyst (0.01–0.02% of zinc chloride, sulphuric acid, phosphoric acid). From α -chloroethyl acetate there are obtained acetyl chloride and acetaldehyde; from α -chloroethyl chloroacetate, chloroacetyl chloride and acetaldehyde; from α -chloroethyl butyrate, butyryl chloride and acetaldehyde; from $\alpha\beta$ -dichloroethyl acetate, acetyl chloride and chloroacetaldehyde; from $\alpha\beta$ -dibromoethyl acetate, acetyl chloride and bromoacetaldehyde, etc.

C. HOLLINS.

Manufacture of pure benzoic acid. I. G. FARBENIND. A.-G. (B.P. [A] 329,375 and [B] 329,389, 18.2.29. [A] Addn. to B.P. 307,343; B., 1930, 603).—(A) Crude benzoic acid is reduced with nascent hydrogen (*e.g.*, with magnesium–aluminium and acetic acid), or is dissolved in benzene, xylene, etc. and shaken with aqueous bisulphite, or is melted under aqueous bisulphite in a stirring autoclave (1–2 atm.). Phthalic acid may be removed by dissolution in the calculated quantity of alkali. (B) The reduction may be omitted if the crude acid is free from naphthaquinone, in which case it is melted under water containing the requisite amount of sodium hydroxide or lime.

C. HOLLINS.

Manufacture of benzoylhydroperoxides [perbenzoic acid] etc. P. A. A. VAN DER BEEK and W. P. JORISSEN (Dutch P. 17,393, 17.2.26).—Benzaldehyde (etc.) in solution in acetone, benzene, chloroform, carbon tetrachloride, etc. is treated with oxygen or air in sunlight or other source of active rays.

C. HOLLINS.

Manufacture of carboxylic acid halides. I. G. FARBENIND. A.-G. (B.P. 308,666, 25.3.29. Ger., 23.3.28).—Methyl chloride is led with 8 vols. of carbon monoxide over a phosphoric or boric acid catalyst (sodium metaphosphate on pumice) at 700–800°; acetyl chloride is obtained in 10–12% yield. Other halogenated hydrocarbons behave similarly.

C. HOLLINS.

Manufacture of alkylene-substituted phenols. SCHERING-KAHLBAUM A.-G. (B.P. 304,727, 22.1.29. Ger., 25.1.28).—A dihydroxydiphenylmethane of the type $(\text{OH} \cdot \text{C}_6\text{H}_4)_2 \cdot \text{CR} \cdot \text{CHR}'$ loses a mol. of phenol when heated at 130–230° with a porous catalyst (tonsil, frankonite), yielding substituted hydroxystyrenes, $\text{OH} \cdot \text{C}_6\text{H}_4 \cdot \text{CR} \cdot \text{CR}'\text{R}''$. Thus the dihydroxydiaryl methanes from acetone and phenol and *m*-cresol give, respectively, 4-hydroxyisopropenylbenzene and 3-hydroxy-4-isopropenyltoluene; the product from methyl ethyl ketone and phenol gives 4-hydroxyisobutenylbenzene. From such products perfumes, pharmaceuticals, and disinfectants may be derived.

C. HOLLINS.

Manufacture of hydrogenated amines. I. G. FARBENIND. A.-G. (B.P. 306,414, 18.2.29. Ger., 18.2.28).—A phenol or a hydrogenated phenol is mixed with ammonia or an amine and either heated at 180–190° with reduced nickel in an autoclave with hydrogen under pressure or passed in vapour form with hydrogen over nickel at 180–190°. The hydrogen is omitted when cyclohexanols are employed. The preparation of mono- and di-cyclohexylamine mixture is described.

C. HOLLINS.

Manufacture of amino-alkyl compounds. I. G. FARBENIND. A.-G. (B.P. 307,305, 4.3.29. Ger., 2.3.28).—Aromatic or heterocyclic magnesyl compounds are treated with amino- or alkylated amino-alkyl halides. β -Diethylaminoethylbenzene, b.p. 100°/10 mm., is obtained from magnesium phenyl bromide. The preparation of δ -diethylaminobutylbenzene, b.p. 132°/10 mm., 4- β -diethylaminoethylanisole, b.p. 140°/10 mm., 4-bromo-1- β -diethylaminobutylbenzene, b.p. 140°/10 mm., and 2-methyl-3- β -diethylaminoethylindole, b.p. 171°/2 mm., is described.

C. HOLLINS.

Manufacture of amino-substituted tertiary alcohols. I. G. FARBENIND. A.-G. (B.P. 307,307, 4.3.29. Ger., 2.3.28).—Esters of aromatic carboxylic acids, carrying as nuclear substituent an alkylated amino-alkylamino- or aminoalkoxyl group, are treated with a Grignard reagent (2 mols.) to give basic tertiary alcohols which form salts soluble in water to neutral solutions. Ethyl *N*- β -diethylaminoethyl-*p*-aminobenzoate, b.p. 171°/4 mm., reacts with magnesium phenyl bromide (2 mols.) to give 4- β -diethylaminoethylaminotriphenylcarbinol, b.p. 215°/2 mm. The preparation of 2- β -diethylaminoethoxyphenyldiethylcarbinol, b.p. 160–164°/3 mm., and 4 : 4'-diethoxy-4''- β -diethylaminoethoxytriphenylcarbinol, b.p. 245–249°/4 mm., from ethyl *o*- β -diethylaminoethoxybenzoate, b.p. 180–183°/10 mm., is also described.

C. HOLLINS.

Manufacture of amino-alcohols. I. G. FARBENIND. A.-G. (B.P. 307,304, 4.3.29. Ger., 2.3.28).—Secondary and tertiary alcohols containing as substituent an aromatic residue carrying an aminoalkyl or alkylated aminoalkyl group are obtained from corresponding ketones by the Grignard reaction. *N*-Methyl-*N*- β -diethylaminoethyl-*p*-aminobenzaldehyde reacts with magnesium *p*-anisyl bromide to give 4-methyl- β -diethylaminoethylamino-4'-methoxybenzhydrol, b.p. 240°/2 mm. Similar compounds are obtained from *N*- β -diethylaminoethyl-*p*-aminoacetophenone, b.p. 171°/2 mm., and magnesium *p*-phenetyl bromide (product, m.p. 101°, b.p. about 170°/2 mm.); from *o*- β -diethylaminoethoxybenzaldehyde, b.p. 152–155°/3 mm., and magnesium ethyl bromide (product, b.p. 142–145°/3 mm.); and from 2- β -diethylaminoethoxy-3-allylbenzaldehyde and magnesium ethyl bromide (product, b.p. 184–187°/5 mm.). The salts of the new bases give neutral solutions in water and have therapeutic properties.

C. HOLLINS.

Manufacture of naphthylaminecarboxylic acid derivatives [naphthylaminosalicylic acids]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 329,741, 13.3.29).—The Kolbe reaction is applied to 4- β -naphthylaminophenols. The preparation of 5- β -naphthylaminosalicylic acid, m.p. 175°, 6- β -naphthylamino-*m*-cresotic acid, 213–214°, and 5- β -naphthylamino-*o*-cresotic acid, m.p. 214–215°, is described.

C. HOLLINS.

Manufacture of *o*-hydroxycarboxylic acids of fluorene. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,305, 6.3.29).—2-Hydroxyfluorene is readily converted by the Kolbe reaction into 2-hydroxyfluorene-3-carboxylic acid, m.p. 256–260° (decomp.), and a smaller amount of the 1-carboxylic acid, m.p. 236–240°. These are separated by crystallisation from

alcohol. The 3-carboxylic acid gives an *o*-anisidide, m.p. 157°.

C. HOLLINS.

Manufacture of *o*-aminocarboxylic esters of the anthraquinone series. I. G. FARBERIND. A.-G. (B.P. 314,028, 20.6.29. Ger., 21.6.28. Addn. to B.P. 267,164; B., 1928, 361).—Anthraquinone-1:2-*isooxazole* is heated with an alcohol or phenol in presence of an alkaline agent (potassium cyanide, sodium alkoxides). The *isooxazole* ring is opened and an ester of 1-aminoanthraquinone-2-carboxylic acid is produced. The methyl (m.p. 228°), ethyl (m.p. 198°), and phenyl (m.p. 198°) esters are described.

C. HOLLINS.

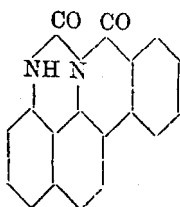
Manufacture of β -anthraquinonecarboxylic acids and esters thereof. I. G. FARBERIND. A.-G. (B.P. 314,020, 13.6.29. Ger., 21.6.28).—2-Aldehydoanthraquinones react in presence of alkali cyanide with water to give leucoanthraquinone-2-carboxylic acids, or with alcohols or phenols to give the corresponding esters; air oxidation of the resulting vats yields anthraquinone-2-carboxylic acids or esters. The preparation of anthraquinone-2-carboxylic acid, m.p. 286—287°; its methyl and ethyl esters, m.p. 170° and 147°, respectively; methyl 1-iodoanthraquinone-2-carboxylate, m.p. 172—175°; methyl, ethyl, and phenyl 1-aminoanthraquinone-2-carboxylates, m.p. 228°, 198°, and 198°, respectively, is described.

C. HOLLINS.

Dye intermediates [alkyl ethers of leuco-acylaminoanthraquinones]. J. THOMAS, D. A. W. FAIRWEATHER, and SCOTTISH DYES, LTD. (B.P. 330,215, 27.11.28).—An acylaminoanthraquinone is reduced with zinc and acetic acid and the leuco-compound is alkylated without isolation. The dimethyl ethers of leuco-derivatives of 1- and 2-acetamido- and 2-benzamidoanthraquinones, and of 1- and 3-chloro-2-acetamidoanthraquinones, are described.

C. HOLLINS.

Condensation product of the perimidone series.



W. MIEG and R. HEIDENREICH, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,955, 11.3.30. Appl., 18.6.28. Ger., 16.12.25).—*N*-Benzoylperimidone is heated at 150—170° with sodium aluminium chloride or is boiled in carbon disulphide with aluminium chloride, to give a compound (annexed formula) or an isomeride. Similar

products are obtained from the *m*-nitrobenzoyl and α -naphthoyl derivatives.

C. HOLLINS.

Manufacture of indoles. IMPERIAL CHEM. INDUSTRIES, LTD., H. A. PIGGOTT, and E. H. RODD (B.P. 330,332, 26.3.29).—Acylated arylamines containing an *o*-methyl or -methylene group are cyclised to 2-substituted indoles by boiling with an alkali metal in an inert solvent (*e.g.*, diethylaniline or tetrahydronaphthalene); a copper catalyst (copper bronze) may be added. The preparation of 2-methylindole from acet-*o*-toluidide; 2-ethylindole, m.p. 35°, b.p. 160—170°/25 mm., from propion-*o*-toluidide; 2:5-dimethylindole, m.p. 114—115°, b.p. 188°/40 mm., from acet-*m*-4-xylylide; 2:7-dimethylindole, m.p. 35—37°, b.p. 146—148°/10 mm., from acet-*m*-5-xylylide; 2-phenylindole, m.p. 185°, from benz-*o*-toluidide; 2:2'-di-indolylmethane, b.p. 152—

153°/15 mm., from malondi-*o*-toluidide; and 2:3-dimethylindole, m.p. 104—105°, b.p. 188—196°/20 mm. (picrate, m.p. 153°), from *o*-ethylacetanilide, is described.

C. HOLLINS.

Iodination of *o*-oxyquinoline-*ana*-[8-hydroxyquinoline-5-]sulphonic acid. UNION CHIM. BELGE SOC. ANON. (B.P. 329,888, 5.7.29. Belg., 7.6.29).—A mixture of aqueous hydrogen peroxide and sulphuric acid is added to a boiling aqueous solution of 8-hydroxyquinoline-5-sulphonic acid, potassium carbonate, and potassium iodide. The iodo-derivative crystallises out on cooling.

C. HOLLINS.

Manufacture of mercaptobenzthiazoles. I. G. FARBERIND. A.-G. (B.P. 306,492, 21.2.29. Ger., 21.2.28).—An *o*-chloronitrobenzene is treated with hot aqueous or alcoholic sodium hydrogen sulphide; the mixture is then cooled, carbon disulphide is added, and reaction completed at 80—100°. The preparation of 2-thiolbenzthiazole and its 5-methyl (m.p. 170—172°), 5-nitro (m.p. 223°), and 5-chloro-derivatives, and of 2:5-dithiolbenzthiazole (from 1-chloro-2:4-dinitrobenzene), is described.

C. HOLLINS.

Gas-generating chemical [lachrymators etc.]. B. C. GOSS, ASSR. to LAKE ERIE CHEM. CO. (U.S.P. 1,750,101, 11.3.30. Appl., 19.1.27).—Chloroacetophenone or other solid irritant is dissolved in methyl or ethyl bromoacetate or other liquid irritant, and the solution is used in shells, bombs, sprays, etc.

C. HOLLINS.

Production of substances which may be used in the textile, leather, and allied industries as wetting, cleaning, foaming, and dispersion agents. H. T. BÖHME A.-G. (B.P. 308,824, 28.3.29. Ger., 30.3.28).—Alcohols corresponding with the higher fatty acids, *e.g.*, oleyl alcohol, $C_8H_{15}\cdot CH:CH\cdot[CH_2]_7\cdot CH_2\cdot OH$, are treated with concentrated or fuming sulphuric acid at 0°, whereby the alcohol group is sulphated and $H-SO_4H$ is added at the double linking.

C. HOLLINS.

Production of water-soluble substances having capillary - active properties [solvents, wetting, emulsifying, and frothing agents]. R. SAJITZ and E. THIEL (CHEM. FABR. POTT & CO.) (B.P. 329,622, 15.1.29. Ger., 28.11.28).—A secondary or tertiary alcohol (C_3 and higher, especially *tert.*-butyl alcohol) is converted by acid agents (concentrated sulphuric acid at 100°) into polymerised hydrocarbons, which are then vigorously sulphonated, *e.g.*, with chlorosulphonic acid in carbon tetrachloride, or with concentrated sulphuric acid in presence of acetic anhydride.

C. HOLLINS.

Manufacture of wetting, cleansing, emulsifying, and dispersing agents and preparations containing the same. I. G. FARBERIND. A.-G. (B.P. 306,116, 29.11.28. Ger., 17.2.28).—Amides or esters (or ester amides) which are useful wetting agents etc. are made by heating together organic carboxylic or sulphonic acids having at least 8 carbon atoms with amines containing at least two hydroxylated radicals. Stearic acid, *e.g.*, is heated with "triethanolamine" at 160—180°, or oleic acid with the product from ethylenediamine and ethylene oxide. The wetting agents may be mixed with soaps, glues, gums, sulphite-cellulose waste liquor, organic solvents, etc. [Stat. ref.]

C. HOLLINS.

Production of organic compounds containing oxygen [higher alcohols]. G. PATART, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,770,165, 8.7.30. Appl., 29.1.26. Fr., 7.2.25).—See F.P. 593,649; B., 1927, 347.

Production of esters. G. F. HORSLEY, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,770,779, 15.7.30. Appl., 9.8.28. U.K., 3.8.27).—See B.P. 301,523; B., 1929, 122.

Manufacture of [poly]sulphides of aromatic [carboxylic acid] compounds. R. EDER, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,769,423, 1.7.30. Appl., 17.5.28. Switz., 27.5.27).—See B.P. 291,100; B., 1929, 889.

Manufacture of [6-]chlorothymol. F. RASCHIG (U.S.P. 1,769,648, 1.7.30. Appl., 2.4.27. Ger., 29.4.26).—See B.P. 270,283; B., 1928, 36.

Products from ethylene and/or its homologues (B.P. 331,186).—See II. Halogenated indanthrones (B.P. 330,217—8). Benzanthrone dyes (B.P. 311,661). Yellow azo-dye intermediate (B.P. 313,927).—See IV. Phosphoric acid esters (B.P. 330,228).—See VII.

IV.—DYESTUFFS.

See A., July, 904, Effect of substituents on shade of derivatives of stilbenesulphonic acids (WAHL and JONICA). 906, Azo dyes of the fluorene series (COURTOT). Substantive dyes derived from 2:7-diaminofluorene (NOVELLI). Colour and constitution (HODGSON). 919, Azo dyes from 2:6-diaceto-resorcinol (ALGAR and BOYLAN). 924, Condensations of ethyl carbamate, phenyl carbamate, and diphenyl carbamate with resorcinol (SEN and MUKHERJI). Methylisopropylthioindigoid dyes from *p*-cymene (HIXSON and CAUWENBERG). 930, Cyanine dyes from quaternary salts (HAMER).

Disintegration. CHWALA.—See I.

PATENTS.

Manufacture of anthraquinone derivatives [wool dyes]. I. G. FARBENIND. A.-G. (B.P. 306,963, 27.2.29. Ger., 29.2.28).— α -Arylamino- β -cyanoanthraquinones are prepared by introduction of either group by suitable reactions, and are then sulphonated to give wool dyes. 3-Bromo-1-*p*-toluidinoanthraquinone is heated with cuprous cyanide in quinoline and the product is sulphonated with weak oleum for a wool red (bluer than the sulphonated starting material). 2-Bromo-1-amino-4-*p*-toluidinoanthraquinone similarly gives a greenish-blue wool dye. 4-Bromo-1-methylamino-2-cyanoanthraquinone is condensed with *p*-toluidine in presence of sodium acetate; sulphonation of the product yields a green wool dye of good shade in artificial light.

C. HOLLINS.

Manufacture of intermediate products of the benzanthrone series and dyes therefrom. Soc. CHEM. IND. IN BASLE (B.P. 311,661, 13.5.29. Switz., 12.5.28).—Benzanthrone is oxidised with manganic sulphate in 100% sulphuric acid at 60–65° to a dihydroxy-3:3'-dibenzanthronyl. This is converted by

alkaline fusion into dihydroxydibenzanthrone, which by alkylation yields grey-blue vat dyes.

C. HOLLINS.

Production of anthraquinone derivatives [halogenated indanthrones]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 330,217, 27.11.28).—3:3'-Dichloro- and dibromo-indanthrones are halogenated; the new halogens enter α -positions and are reactive. 3:3'-Dichloroindanthrone is treated with chlorine in anhydrous nitrobenzene at 15° to give the 3:3':4:4'-tetrachloro- or the 3:3':4-trichloro-compound; 3:3'-dibromoindanthrone gives similar products. The hydrazine may first be converted into the azine and then halogenated; or the azine may be heated with concentrated hydrochloric acid at 160–165°, whereby the hydrogen chloride additive product is converted into 3:3':4-trichloroindanthrone. Chlorination of 3:3':6(7):6'(7')-tetrachloroindanthrone gives a green vat dye.

C. HOLLINS.

Production of anthraquinone derivatives [from halogenated indanthrones]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 330,218, 28.11.28).— α -Halogenated indanthrones are made to react with phenols or with ammonia or primary or secondary amines, particularly with 1-aminoanthraquinone, methylamine, or aniline, with or without diluents, acid-absorbing agents, and/or copper or copper compounds. 3:3':4:4'-Tetrachloroindanthrone (cf. preceding abstract) is condensed with aniline for a blue-green vat dye, with 1-aminoanthraquinone (blue-green), and in the azine form with ammonia (blue-green). 4-Chloro- or 4-bromo-indanthrone with phenol gives a greenish-blue vat dye.

C. HOLLINS.

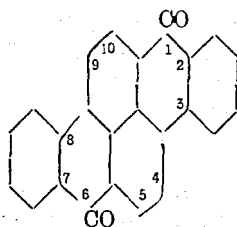
Manufacture of derivatives of pyranthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,357, 17.12.28).—The ethers of hydroxypyranthrone (B.P. 325,250; B., 1930, 453) are halogenated, preferably in chlorosulphonic acid in presence of a carrier or with sulphuryl chloride in nitrobenzene. The bromination of ethoxypyranthrone (bluish-red), dimethoxypyranthrone (orange), β -chloroethoxypyranthrone (bluish-red), diphenoxy- and di-(*o*-methoxyphenoxy)-pyranthrone (reddish-brown), and the chlorination of methoxypyranthrone (brown) are described.

C. HOLLINS.

Manufacture of nitrogen-containing derivatives of the benzanthrone series. I. G. FARBENIND. A.-G. (B.P. 307,926, 13.3.29. Ger., 16.3.28).—Dibenzpyrenequinones are nitrated to give products reducible to amino-compounds which are vat dyes. 2:3:7:8-Dibenzpyrene-1:6-quinone (annexed formula) or its dibromo-derivative gives a violet vat dye, 1:2:7:8-dibenzpyrene-3:6-quinone a greyish-blue (dinitro-) becoming deep brown with hypochlorite, and the methyl derivative (from 4-*p*-toluylbenzanthrone) an olive-green.

C. HOLLINS.

Production of dye derivatives [sulphuric leuco-esters] of the flavanthrone series and their use



R. S. BARNES, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 329,356, 11.12.28).—Red leuco-esters are obtained when flavanthrone or 3:3'-dichloroflavanthrone is treated with pyridine-sulphuric anhydride and copper in presence of an alkaline-reacting salt (sodium acetate).

C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. I. G. FARBENIND. A.-G., E. HOFFA, and E. THOMA (B.P. 329,960, 25.2.29. Addn. to B.P. 286,274; B., 1929, 675).—The process of the prior patent is extended to *p*-aminodiarylaminines in general. Examples are: *p*-aminophenyl- β -naphthylamine \rightarrow 2:3-hydroxynaphthoic anilide (navy-blue); methyl 3-amino-6- β -naphthylaminobenzoate \rightarrow *p*-chloroanilide (reddish-dark blue); 3-chloro- or 3:5-dichloro-4-aminophenyl- β -naphthylamine \rightarrow *p*-chloroanilide (dark blue); 4-amino-4'-phenoxydiphenylamine \rightarrow *o*-toluidide (indigo-blue), etc. 4-Aminodiphenylamines containing a cyano-, nitro-, or acylamino-group in 4'-position, and 2:3-hydroxynaphthoic anilide, *o*-toluidide, *o*- and *p*-anisidides, *p*-chloroanilide, and 4-chloro-*o*-toluidide are specially claimed as components.

C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 306,415, 18.2.29. Ger., 18.2.28).—Diazo compounds of the benzene series containing at least one negative group are coupled in substance or on the fibre with a 2:3-hydroxynaphthoic 4-alkoxy- α -naphthylamide. The 4-methoxy- α -naphthylamide is coupled, *e.g.*, with diazotised 5-nitro-*o*-anisidine (garnet) or 4-chloro-*o*-toluidine (claret).

C. HOLLINS.

Manufacture of azo dyes [ice colours etc.]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 329,353, 16.11.28).—Diazoamino-compounds obtainable by the process of B.P. 324,041 (B., 1930, 364) are coupled in substance or on the fibre with suitable coupling components, the diazoamino-compound being split or the dyes developed by means of acid reagents. The diazoamino-compound derived from 4-chloro-*o*-toluidine (by diazotising and coupling with 4-sulphoanthranilic acid) is dissolved in water, acetic acid and alum are added, and the solution is used for coupling on the fibre with 2:3-hydroxynaphthoic β -naphthylamide for red. The diazoamino-compound from 4-chloro-*o*-anisidine is mixed with a solution of the *o*-anisidine in the calculated amount of alkali, and the dry mixture after evaporation is made into a printing paste; the printed colour is steamed and developed with dilute formic or acetic acid. The diazoamino-compound may be pasted with hot water containing thiodiglycol ($\beta\beta$ -dihydroxydiethyl sulphide) and alkali and thickening agent added to form the printing paste; the print is steamed or hung, and developed in dilute acetic, formic, or oxalic acid. Amongst the coupling components mentioned are: 2:3-hydroxynaphthoic α - and β -naphthylamides, *o*-anisidide, *o*-toluidide, *o*-phenetide, 2:5-dimethoxyanilide; 2-hydroxycarbazole-*o*-carboxylic *o*-anisidide, *o*-toluidide, acetoacetic toluidide; phenyl 4-hydroxy- α -naphthyl ketone; 2-hydroxy- β -naphthacarbazole; acylamino-naphthols; etc.

C. HOLLINS.

Manufacture of azo dyes and their application to the dyeing of regenerated cellulose materials.

IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 329,961, 25.2.29).—Level shades on viscose are obtained with disazo dyes of the type: non-phenolic *m*-nitro- or *m*-acylamino-arylamine \rightarrow B, reduced or hydrolysed, \rightarrow C, in which B is a phenol or naphthol, with or without carboxyl or sulphonic substituents, or an *N*-arylamino-naphtholsulphonic acid, whilst C is a *m*-phenylenediamine or an *N*-aryl- or *N*-aroyl-2:5- or -2:8-aminonaphtholsulphonic acid, or a 1:8- or 1:5:7-aminonaphtholsulphonic acid; the *N*-substituents must be free from sulphonic groups. Examples are: *m*-nitroaniline \rightarrow salicylic acid, reduced \rightarrow phenyl- γ -acid (brown); 4-nitro-*o*-anisidine \rightarrow phenyl- γ -acid, reduced \rightarrow H-acid (blue); *m*-aminoacetanilide \rightarrow salicylic acid, hydrolysed, \rightarrow benzoyl-J-acid (orange); *m*-nitroaniline \rightarrow phenyl- γ -acid, reduced, \rightarrow *m*-phenylenediamine (yellowish-brown).

C. HOLLINS.

Manufacture of yellow monoazo dyes [for chrome leather], material dyed therewith, and an intermediate product therefor. I. G. FARBENIND. A.-G. (B.P. 313,927, 19.6.29. Ger., 19.6.28).—2-Chloro-*p*-toluidine-6-sulphonic acid, obtained by sulphonation and reduction of *o*-chloro-*p*-nitrotoluene, is diazotised and coupled with resorcinol (1 mol.) to give an intense orange-yellow dye for chrome leather, equal to Phosphine in purity of shade. Substituted resorcinols may be used.

C. HOLLINS.

Manufacture of azo dyes containing chromium. I. G. FARBENIND. A.-G. (B.P. 306,843, 25.2.29. Ger., 25.2.28).—The azo dyes of B.P. 245,765 (B., 1926, 734), containing as coupling components pyrazolones derived from aminoarylsulphonylsalicylic acids, are boiled with chromium formate (etc.). Examples are *o*-chloro-*p*-toluidine or metanilic acid \rightarrow phenylmethylpyrazolone from 2-amino-4'-hydroxy-4-sulphodiphenylsulphone-3'-carboxylic acid (greenish-yellow).

C. HOLLINS.

Manufacture of sulphide dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and M. WYLER (B.P. 329,716, 26.2.29).—Aceto-*p*-xylylide is nitrated with mixed acid, and the nitro-compound, or the monoacetyldiamino-*p*-xylene, m.p. 141–142°, obtained on reduction, is heated with benzidine and sulphur to give an orange sulphide dye.

C. HOLLINS.

Manufacture of vat dyes [from naphthalene-1:4:5:8-tetracarboxylic dianhydride and *o*-diamines]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,364, 19.1.29).—The red vat dye from naphthalene-1:4:5:8-tetracarboxylic dianhydride and *o*-phenylenediamine is converted by acid oxidants (chromic acid; permanganate and sulphuric acid) into bright orange vat dyes.

C. HOLLINS.

Manufacture of vat dyes of the violanthrone series. H. SIEBENBÜRGER, ASST. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,770,815, 15.7.30. Appl., 21.7.25. Switz., 9.8.24).—See B.P. 238,225; B., 1925, 876.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of small quantities of copper in materials [fabrics]. P. KLICKOW and SIEBNER (Kautschuk, 1930, 6, 161–162).—The area of 10–20 g. of the fabric is measured in sq. cm. The material is then ashed, treated with 10 c.c. of nitric acid (*d* 1.4),

and evaporated with 5 c.c. of sulphuric acid until fumes of the latter are evolved. The treatment with nitric and sulphuric acids is repeated once or twice, and the solution containing 3–5 c.c. of concentrated sulphuric acid per 100 c.c. is precipitated with hydrogen sulphide. The precipitated copper sulphide is filtered off, care being taken to exclude air as far as possible. The precipitate is washed with as little water as possible into a colorimetric tube, and after warming with a few drops of nitric acid and addition of excess of ammonia, and standardisation of the volume, the copper is determined colorimetrically by comparison with a solution of known copper content. The suggested permissible maximum for fabrics intended to be proofed with rubber is 0.002 g. Cu/m.² D. F. TWISS.

Mechanism of the plasticising of natural cellulose fibres. K. HESS (Z. angew. Chem., 1930, 43, 471–481).—The behaviour of natural cellulose fibres from various sources when subjected to the action of cuprammine solutions has been followed röntgenographically. The results indicate that, besides diffusion phenomena, two compounds are formed between the cellulose and the copper ammine which subsequently undergo hydration to an extent which depends on the alkali concentration. During the latter process the fibres assume a spiral structure, the micelles lose their regular orientation, and the fibres swell by osmosis and become plastic and elastic. Similar tests made on alkali-cellulose in carbon disulphide appear to indicate that a compound is formed, but the reaction is much more complex than in the case of cuprammine solutions. Cellulose acetate and nitrate in various organic solvents, e.g., chloroform-methyl alcohol, cyclohexanone, camphor, give röntgenograms which show lines apparently due to the formation of complexes, the presence of which causes mechanical changes to take place in the micelles of the fibre structure; liquid is drawn into the fibre and serves as a lubricant allowing the micelle series to slip over one another, thus producing the characteristic plastic properties.

A. R. POWELL.

Bamboo. I. Fine structure of the bamboo fibre. K. SISIDO (J. Cellulose Inst., Tokyo, 1930, 6, 148–150).—A theoretical discussion of the results of previous workers.

Composition of *Cassia siamea*, L. Y. SHINODA (J. Cellulose Inst., Tokyo, 1930, 6, 155–156).—The following percentage composition has been found. Moisture content 11.2, alcohol-benzene extract 18.2, lignin (König) 37.3 (on the extracted, dry product), ash content 0.3, impure cellulose content 40.3, hydrolysis value (Ost) 92.8%, and pentosan content (Tollens-Krüger) 15.6. Before chlorination the wood meal was steeped in 10% sodium hydroxide solution for 48 hrs. and its cellulose content redetermined, when the value 33.8%, of purity 100%, was found. Xylose and mannose were found in the hydrolysis liquor, but no galactose was present.

B. P. RIDGE.

Factors during spinning which influence the physical properties of rayon. P. C. SCHERER, JUN., and R. E. HUSSEY (Ind. Eng. Chem., 1930, 22, 594–596).—Complete regeneration has been attained on the spool by the adhering liquor at a relatively

low temperature. The effects of time of contact in the acid bath and of the ripeness of viscose on the properties of rayon are recorded. Complete regeneration by control of the time of contact with the bath is desirable in existing processes. L. S. THEOBALP.

Removal of hemicelluloses from wood by sodium hydroxide. O. HORN (Cellulosechem., 1930, 11, 151–152).—Beech-wood meal, previously extracted with 1:1 benzene-alcohol, is treated 4 times for 36–48 hrs. with 5% sodium hydroxide solution. Determinations of the lignin and methoxyl content show that a portion of the lignin together with the hemicellulose have been removed during treatment with the soda. (Cf. Friedrich and Diwald, B., 1925, 151.) T. H. MORTON.

Determination of the acetate content of cellulose acetate. D. KRUEGER (Farben-Ztg., 1930, 35, 2032–2033).—The methods which have been proposed for this determination can be divided into two classes involving acid and alkaline hydrolysis. Accurate determinations can be achieved only by choice of a method whereby adsorption of the hydrolysing agent and decomposition of the cellulose are kept at a minimum. The methods advocated involve the careful standardisation of the conditions of hydrolysis. J. O. CUTTER.

Nitrates of cellulose benzoate. M. SENDO and J. KONDO (J. Cellulose Inst., Tokyo, 1930, 6, 150–155).—Cellulose monobenzoate was treated at 18–20° with mixed acids of the following percentage composition in, respectively, sulphuric, nitric, nitrous acid, and water: (1) 71.14, 20.81, 0.13, 7.92; (2) 65.6, 18.1, 0.15, 16.15; and (3) 59.58, 21.39, 0.23, 18.8, when cellulose dinitrate mononitrobenzoate [$C_6H_7O_2(NO_3)_2 \cdot CO_2 \cdot C_6H_4 \cdot NO_2$] was obtained by means of acids (1) and (2) and the mononitrate [$C_6H_8O_3(NO_3) \cdot CO_2 \cdot C_6H_4 \cdot NO_2$] by acid (3). The explosive properties of these nitrates are similar to those of the normal cellulose nitrates, but, compared with equally nitrated normal cellulose, these mixed esters are more stable, less hygroscopic, less soluble in ether-alcohol, and burn at a slower rate.

B. P. RIDGE.

Cellulose oxalates. G. VON FRANK and W. CARO (Ber., 1930, 63, 1532–1543).—Alkyl cellulose oxalates are readily prepared by the action of alkoxalyl chlorides on cellulose (paper, ramie fibre, or Girard's hydrocellulose) in presence of pyridine and nitrobenzene. Provided that the degree of esterification is uniform, increase in the weight of the alkyl group is accompanied by diminution in density, increase in solubility in non-polar solvents, and decrease in polar media, diminution in softening temperature, increase in plasticity of films, and decrease in their tenacity. The products obtained are mixtures of the di- and tri-esters. When hydrolysed they yield hydrocellulose. The following substances are prepared by the action of oxalyl chloride, purified from phosphorus trichloride by distillation, on the requisite alcohol; in the case of solid alcohols, a solution in chloroform is used: *allyloxalyl chloride*, b.p. 50–51°/12 mm.; *cyclohexoxalyl chloride*, b.p. 120–122°/34 mm.; *octoxalyl chloride*, b.p. 104–106°/9 mm.; *cetoxalyl chloride*, b.p. 155–160°/0.01 mm.; *nitrobenzoyloxalyl chloride*, b.p. 183°/11–12 mm. H. WREN.

Beating of [paper] pulp. XI. A characteristic of vegetable fibres as papermaking materials. M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 144—147; cf. B., 1930, 608).—Wool, silk, asbestos, and vegetable fibres were beaten, and their paper-forming properties compared. Wool cannot be split longitudinally, but is reduced to short lengths showing no fibrillation, from which it is impossible to form a sheet. Silk and asbestos split up into fibrils, but the sheets formed from the products are weak owing to the slippage of the fibres. It follows, therefore, that fibrillation is not necessarily accompanied by the development of the adhesive properties which are essential for the production of satisfactory sheets. Only vegetable fibres have these characteristic properties, which are due to the colloidal state of the surface of the fibrils, and their felting capacity is of secondary importance.

B. P. RIDGE.

Distribution of loading in paper. H. SCHILDE (Papier-Fabr., 1930, 28, 409—415, 423—427, 439—444).—The factors controlling the concentration of loading material in a sheet of paper, particularly the change in concentration from the "top-side" to the "under-side," have been investigated. It is found that the particles of loading do not fall to the under-side according to the usual laws, but are affected by the state of the fibres, the substance of the sheet, the speed of the paper-machine, and the mode of removal of water by mechanical means from the wire-part. The amount of loading lost through the wire almost invariably exceeds that retained in the sheet, and the loss is increased at high machine speeds and by the use of vacuum couch rolls. No rules are formulated, but the effects of the various factors are illustrated by a series of solid diagrams.

T. T. POTTS.

Adhesives and adhesion. LEE.—See I. Conversion of cellulose into sugar. LÜERS.—See XVII.

See also A., July, 1940, Silk fibroin (GOLDSCHMIDT and STRAUSS). 968, X-Ray analysis of cotton fibres (CLARK and others). Destruction of coniferous wood by the larva of the common beetle (FALCK).

PATENTS.

Treatment of coconut fibre. E. ELÖD (B.P. 308,720, 15.3.29. Ger., 27.3.28).—The fibres are softened without loss of strength and rendered suitable for spinning if after removal of the embedding material they are digested for about 40 min. at 120—150° with dilute solutions of alkalis. After washing and drying, the fibres may be treated with hygroscopic agents, oils, etc.

D. J. NORMAN.

Treatment of threads for receiving sound records. W. F. CROLL (B.P. 330,679, 3.4.29).—Suitable thread, e.g., artificial horse hair, cotton, silk, or soft copper wire coated with cellulose, is impregnated with a compound containing water, glycerin, soap, and wax and is then passed through dies of successively varying diameter.

D. J. NORMAN.

Preparation of artificial silk. COMP. DE PROD. CHIM. ET ELECTROMÉTALL. ALAIS, FROGES, ET CAMARGUE (B.P. 305,468, 29.1.29. Fr., 4.2.28).—Artificial filaments are made from products obtained by condensing

urea or its derivatives and/or cyanamide or its derivatives with formaldehyde, the condensation being conducted in an aqueous medium until the product, when poured into water, neither dissolves nor flocculates, but collects into a homogeneous viscous mass. The solution is simultaneously or subsequently concentrated until it has a viscosity suitable for spinning. The condensation reaction is better controlled if carried out in neutral solution; when condensation has reached the desired stage the solution should be stabilised before concentration by adding an alkali or alkaline-reacting salt such as an acetate or phosphate. A suitable coagulating bath comprises an acidified saturated solution of, e.g., magnesium, sodium, or aluminium sulphate.

D. J. NORMAN.

Producing [from animal proteins] solutions adapted to be spun. HEBERLEIN & Co. A.-G. (B.P. 306,464, 20.2.29. Ger., 20.2.28).—Animal proteins of sparing solubility and high mol. wt., e.g., horn, skin, flesh, waste wool, or silk, are made to swell by treatment with hot water, if necessary under pressure, or with an acid solution which does not cause degradation of the protein, and are simultaneously or subsequently dissolved in liquid phenols or their homologues or derivatives.

F. R. ENNOS.

Preventing the evolution of disagreeable smelling gases in the viscose artificial silk manufacture. BRIT. ENKA ARTIFICIAL SILK Co., LTD. (B.P. 316,971, 20.7.29. Holl., 7.8.28).—A small quantity of alkali nitrite is added to the viscose or to the lye used for dissolving the cellulose xanthate, and the nitrous vapours formed therefrom during spinning are mixed in the air-discharge pipes of the spinning room with oxides of nitrogen from another source, so that the noxious gases are oxidised.

F. R. ENNOS.

Manufacture of threads or filaments of cellulose derivatives. BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 331,229, 20.12.28).—The extruded filaments of organic derivatives of cellulose while traversing a countercurrent of evaporative medium in the spinning cell are subjected to a controlled and uniform amount of stretch by passing around two or more rollers, the first receiving the filaments while they are still plastic and contain 20—40% of residual solvent, and the subsequent rollers drawing the filaments at a greater speed than the first.

F. R. ENNOS.

Production of artificial materials from organic esters of cellulose. BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 330,950, 18.1. and 12.3.29).—Threads of the cellulose derivative, while still in a swollen condition, are partially hydrolysed by passing through an aqueous or alcoholic solution of caustic alkali or ammonia; the desired swollen condition is produced in the case of the wet-spinning process by incorporation of a high-boiling solvent with the spinning solution, the coagulating bath, or a subsequent treatment bath, and in the case of the dry-spinning process by use of a high-boiling solvent in the spinning solution.

F. R. ENNOS.

Manufacture of artificial silk from viscose. W. W. TRIGGS. FROM SPINNSTOFFFABR. ZEHLENDORF

GES.M.B.H. (B.P. 331,097, 28.6.29).—The thread is drawn from the spinning nozzle by means of a roller and is thence wound on to the bobbin, the circumferential velocity of the roller being greater than that of the bobbin, so that uniform shrinking and structure of the thread is ensured. F. R. ENNOS.

Manufacture of opaque films. NON-INFLAMMABLE FILM Co., LTD., and D. J. P. PHILLIPS (B.P. 331,214, 25.3.29).—Two films of regenerated cellulose or cellulose esters or ethers are superimposed, the one containing finely-divided metallic powder, *e.g.*, gold or aluminium, and the other ordinary fillers with or without pigments, *e.g.*, zinc oxide. F. R. ENNOS.

Manufacture of viscose products. I. G. FARBENIND. A.-G. (B.P. 307,848, 14.3.29. Ger., 14.3.28).—In order to remove heavy-metal (copper or iron) compounds, viscose products, after coagulation and washing, are treated with oxalic, lactic, tartaric, or citric acids or their salts. F. R. ENNOS.

Manufacture of soluble cellulose esters and ethers. I. G. FARBENIND. A.-G. (B.P. 305,947, 11.2.29. Ger., 11.2.28. Addn. to B.P. 283,181 and 297,766; B., 1928, 637; 1929, 51).—Cellulose is heated, above or below 100° and in the presence of a base such as pyridine, with a number of higher fatty acid halides, either in admixture or successively, which may also be replaced wholly or in part by a halide of a cyclic-substituted fatty acid (phenylacetic acid) or of an alicyclic carboxylic acid, *e.g.*, cyclohexanecarboxylic acid; where esterification takes place below 100°, the reaction mixture is afterwards heated above 100° until the desired solubility of the cellulose ester is attained. F. R. ENNOS.

Manufacture of readily soluble acylcelluloses yielding clear solutions. I. G. FARBENIND. A.-G. (B.P. 305,674, 8.2.29. Ger., 9.2.28).—Cellulose is acetylated below 50° in the presence of a relatively small amount of catalyst (sulphuric acid, less than 2% by wt. of the cellulose), part of the fatty acid normally present being replaced by monochloroacetic acid as a diluent. F. R. ENNOS.

Manufacture of finely-comminuted masses from cellulose derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,957, 16.3.29).—Cellulose derivatives are subjected to mechanical tearing by friction by repeated passage through a friction roller, in the presence of a resin and/or a non-solvent moistening agent (water, alcohol, hydrocarbons) which may contain small amounts of gelatinising agents insufficient to dissolve the cellulose derivatives, and the mass so obtained is afterwards ground in a ball- or hopper-mill. Addition may also be made during either process of dispersing agents, protective colloids, or emulsifying agents, together with dyes and fillers which have no chemical action on the cellulose derivatives. F. R. ENNOS.

Manufacture of artificial sponges [from viscose]. I. G. FARBENIND. A.-G., Assees of R. SKUTEZKY (B.P. 308,838, 28.3.29. Austr., 30.3.28).—Viscose, to which about 10% of its weight of fibrous material (cotton, hemp, flax, etc.) may be added if desired, is mixed with pore-forming fillers in the form of neutral salts

with or without water of crystallisation, *e.g.*, sodium sulphate, and salts reacting with alkali (acid or ammonium salts), moulded to the required shape, heated for $\frac{1}{2}$ —3 hrs. at 180° under pressure in a moist atmosphere, and washed with hot water. F. R. ENNOS.

Controlling the moisture content of paper. F. E. P. KLAGES (B.P. 330,596, 8.3.29).—Air is continuously withdrawn from the surface of the partly dried paper, preferably at the beginning of the last nest of dryers, and is passed over a wet-bulb thermostat which controls the steam supply to the drying cylinders. D. J. NORMAN.

Manufacture of pulp [boards]. S. D. WELLS, ASSR. to PAPER MILL LABORATORIES, INC. (U.S.P. 1,746,451, 11.2.30. Appl., 7.7.26).—Compact and waterproof products are obtained from cereal straws or other fibrous vegetable material by digesting the raw material with, *e.g.*, lime and then treating the unwashed or partly-washed pulp in a beating engine with acids or acid salts to neutralise the cooking agent, at the same time converting it, if desired, into a filler, *e.g.*, calcium sulphate, and to coagulate colloidal organic matter. When a waterproof product is required aluminium sulphate should be used to complete the acidification of the mass. Further quantities of colloidal matter may be coagulated if, after the liquor has been acidified, it is again rendered slightly alkaline. D. J. NORMAN.

[Rubber]-coated paper. G. J. ESSELEN, JUN., and R. P. ROSE, ASSRS. to GEN. RUBBER Co. (U.S.P. 1,746,888, 11.2.30. Appl., 8.4.25).—A coating mixture comprises rubber latex, a filler (clay), and a resinous adhesive (shellac), which has been dispersed in an aqueous solution of, *e.g.*, borax. Preferably the amount of rubber solids, together with that of any other binders present, should exceed 12% of the weight of the filler. Vulcanising agents may be added, and vulcanisation effected at below 93°. D. J. NORMAN.

Treatment of artificial silk. B. BORZYKOWSKI (U.S.P. 1,770,729, 15.7.30. Appl., 29.10.28. Ger., 12.3.27).—See B.P. 287,073; B., 1929, 677.

[Sliver]-drying apparatus. W. PRINCE-SMITH and D. WATERHOUSE (B.P. 331,403, 15.7.29).

Production of pulp [from wood or other fibrous material]. (SIR) G. C. MARKS. From BAUER BROS. Co. (B.P. 331,567, 5.3.29).

Concentration of paper pulp (B.P. 307,300).—See I. Soluble wood ethers (B.P. 330,011).—See III. Laminated glass (B.P. 306,891, 324,898, 325,048, and 331,030). Safety glass (B.P. 315,667).—See VIII. Celluloid from scrap films (B.P. 331,006).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Plant for bleaching. R. WEISS (Sealed Notes Nos. 1723 and 1730, 23.3. and 2.4.07. Bull. Soc. Ind. Mulhouse, 1930, 96, 284—286). Report by J. LÉONHART (*Ibid.*, 286—287).—The apparatus comprises two boilers, a circulating pump, a tubular heater, and a compensating vessel. The bleaching liquid is pumped through the heater to the bottom of one of the boilers, and up

through the textiles therein; it passes then through the compensating vessel, wherein it is freed from air, to the top of the second boiler, and is sprayed on to the textiles therein; from the bottom of the second boiler it returns to the pump. The direction of circulation is periodically reversed so that the materials in both boilers receive identical treatment. Uniformity of operation, avoidance of the formation of oxycellulose, and an economy of steam and chemicals is claimed. LÉONHART reports favourably on the plant.

A. B. MANNING.

Dyeing of artificial silks. W. WELTZIEN (Kolloid-Z., 1930, 52, 110—116).—A review embracing recent work on the dyeing of cellulose and acetate silks.

E. S. HEDGES.

PATENTS.

Cleansing composition [for fabrics]. A. R. JENNINGS (B.P. 306,119, 22.12.28. U.S., 17.2.28).—A mixture of a powdered mineral absorbent, such as talc, with eucalyptus oil.

A. J. HALL.

Production of coloured reserve effects on textile materials. I. G. FARBENIND. A.-G. (B.P. 311,740, 15.5.29. Ger., 15.5.28).—Coloured reserves on aniline-black are obtained by using a vat-dye paste thickened with cellulose esters or ethers (or other thickening agents precipitated by alkali), to which a reserving agent (sodium acetate, sodium hydrogen sulphite, zinc oxide) is added.

C. HOLLINS.

Production of coloured discharge effects on dyed goods. DURAND & HUGUENIN A.-G. (B.P. 310,893, 2.5.29. Ger., 2.5.28).—The gallocyanines obtainable according to B.P. 301,329 (B., 1930, 502) from *m*-substituted alkylanilines (ethyl-*m*-toluidine, *m*-chlorodimethylaniline, dimethyl-*m*-toluidine) give vivid coloured discharges by the usual hyposulphite reduction method.

C. HOLLINS.

Production of effects on vegetable yarns and fabrics. HEBERLEIN & Co. A.-G. (B.P. 313,616, 14.6.29. Ger., 15.6.28).—Vegetable fibrous materials are esterified superficially so that they retain their strength and gain a woolly handle and resistance to wetting and direct dyes. The material is treated with alkali and esterified by means of an acyl halide in an indifferent solvent in the proportion of 0.5, 2.0, or 3.0 mols. of the halide per mol. of $C_6H_{10}O_5$ (cf. Karrer and Zega, B., 1923, 1125 A).

A. J. HALL.

Manufacture of coloured fabrics. J. MORTON, J. E. G. HARRIS, and MORTON SUNDOWN FABRICS, LTD. (B.P. 329,334, 13.11. and 28.12.28).—Yarn is treated with oxidants (potassium chlorate, ammonium thiocyanate) suitable for developing leuco-vat-dye esters of the Soledon or Indigosol type, and is then woven with untreated yarn. The fabric is printed with a leuco-ester printing paste preferably containing a catalyst (ammonium vanadate). It is advantageous to pass the printed steamed fabric through a dilute alkaline bath to prevent development of the leuco-ester on untreated portions during the rinsing operations.

C. HOLLINS.

Printing with aniline-black etc. I. G. FARBENIND. A.-G. (B.P. 311,779, 16.5.29. Ger., 16.5.28).—A vat dye

can be printed simultaneously with aniline-black if a cellulose ester or ether or other thickening agent precipitated by alkali is used. The aniline-black is developed, and the material is then padded in an alkaline bath containing hyposulphite and a wetting agent, the vat dye being then fixed by steaming without intermediate drying.

C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND. A.-G. (B.P. 314,904, 4.7.29. Ger., 4.7.28. Addn. to B.P. 279,864; B., 1929, 15).—Cotton is printed with a paste containing vat dye, a stable reducing agent (glucose), and a cellulose ether or ester as thickener (cellulose methyl ether), then padded in a caustic alkali bath, and without drying steamed, washed, oxidised, and soaped.

C. HOLLINS.

Fixation of insoluble metal compounds on textile materials [weighting or mordanting of acetate silk etc.]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 329,659, 16.11.28).—Acetate silk or other textile is impregnated, *e.g.*, in a padding mangle, with a soluble metal salt (stannic chloride, ferric chloride, chromium chloride, etc.), and, after drying, a precipitant (particularly trisodium phosphate) is similarly applied.

C. HOLLINS.

Ornamentation of textile fabrics etc. I. G. FARBENIND. A.-G. (B.P. 311,741, 15.5.29. Ger., 15.5.28).—A flexible support such as paper is treated with a removable double layer of pliable materials pigmented as desired, *e.g.*, cellulose derivatives or resins, the layer in contact with the support being of higher m.p. than the other, which latter softens and becomes sticky at raised temperatures. The prepared sheet is laid on the fabric, and ironed hot, when the layer of lower m.p. is absorbed by the fabric, after which the paper support is removed to expose the layer of higher m.p.

F. R. ENNOS.

Dyeing of cellulose acetate threads. O. SPENGLER and W. MÜLLER, Assis. to GEN. ANILINE WORKS, INC. (U.S.P. 1,770,714, 15.7.30. Appl., 14.8.26. Ger., 15.8.25).—See F.P. 619,329; B., 1928, 86.

Delustring of artificial silk fibres or rayon. NYANZA COLOR & CHEM. CO., INC. (B.P. 315,432, 13.7.29. U.S., 14.7.28).—See U.S.P. 1,705,490; B., 1929, 640.

Apparatus for bleaching and dyeing etc. TISSAGE DEWITTE-LIETAER (B.P. 319,672, 25.9.29. Belg., 25.9.28).

Machine for dyeing, bleaching, and the like of loose yarns or bobbins. F. G. SCHUMACHER and J. J. MÜLLER (B.P. 331,706, 20.7.29).

Dyeing machines. C. F. KRANEY, and C. HEINZE MASCHINENFABR. A.-G. (B.P. 331,520, 28.12.28).

Dyeing of fibre laps. C. OBERMAIER (OBERMAIER & Co.) (B.P. 316,182, 16.7.29. Ger., 24.7.28).

Methods and apparatus for coating fabrics [with rubber etc.]. NAUGATUCK CHEM. CO., Assees. of J. MCGAVACK (B.P. 319,726, 5.7.29. U.S., 27.9.28).

Wetting etc. agents (B.P. 306,116, 308,824, and 329,622).—See III. Sulphuric leuco-esters (B.P. 329,356). **Dyeing of regenerated cellulose** (B.P. 329,961).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Volumetric determination of phosphoric acid.

W. H. ROSS (J. Assoc. Off. Agric. Chem., 1930, 13, 203—207).—The accuracy of the official methods for the volumetric determination of phosphoric acid in the presence of sulphates and in materials high in organic matter has been studied collaboratively, using pure monopotassium phosphate, Tennessee phosphate rock, and a commercial grade of cottonseed meal. The results showed that, in the presence of sulphates, precipitation at 45—50° leads to high values, but precipitation at 25—30° with continuous stirring gives values in good agreement with those obtained in the absence of sulphates. Dissolution of the sample in dilute sulphuric acid gave good results for the material high in organic matter, provided precipitation was performed at 25—30° with continuous stirring.

H. J. DOWDEN.

Causticising. W. E. PIPER (Chem. Met. Eng., 1930, 37, 362—365).—The evolution of the Solvay process into a modern continuous system (based on identical reactions), utilising agitators, thickeners, and vacuum filters, is indicated and typical flow sheets of continuous and batch systems are given. The method of slaking the lime is vital as determining the size of the plant because of the effect it has on the settling rate of the carbonate residue. When milk of lime was prepared by slaking with all the water needed to make the soda solution it yielded a carbonate precipitate that settled at an initial rate of about 0.2 ft./hr.; if slaked dry with water and then creamed with the remainder of the solution the rate was 1.5; and when slaked dry with dilute caustic solution and then creamed the rate was 8 ft./hr. The use of a raised temperature increases both the settling rate and the percentage conversion (causticity), but there is no advantage in exceeding 85°; the conversion will then be about 95% when making a 10—15% caustic soda solution. The heating steam should be in a coil and the vessels well lagged to preserve the heat and prevent convection currents. It is advisable to avoid either too rapid or too prolonged agitation; 90 min. at twice the normal speed halved the rate of settling compared with 30 min. at normal speed. Excess of lime does not seem to have much effect on the percentage conversion of the soda (which depends on the concentration of solution made), but it increases the speed of reaction. It is recommended, in the continuous process, to have an additional causticising agitator between the first and second thickeners, to which a portion of the more concentrated sodium carbonate solution is added, the primary agitators giving a rapid and high conversion of the soda with excess of lime and the secondary an incomplete conversion of soda, but complete conversion of lime. Of the dilute caustic carbonate solution from the second thickener part is used to make milk of lime and the remainder to dissolve soda ash; the fractions unite in a drag classifier which is used to remove grit etc.

B. M. VENABLES.

Aluminium sulphate. G. KULLERUD and A. D. HORLÜCK (Dansk Tidsskr. Farm., 1930, 4, 157—171).—Commercial aluminium sulphate frequently contains basic salt, and it is difficult to prepare a pure sulphate

from such material by repeated recrystallisation unless sufficient sulphuric acid be previously added to transform the basic salt present into the normal sulphate. A number of p_H determinations have been carried out by various methods on aluminium sulphate solutions, and the results are tabulated; when using the quinhydrone electrode, several hours are necessary before a constant value is attained. It is recommended that aluminium sulphate should be employed, for pharmaceutical purposes, in the form of a solution, which must always be tested to see that it contains alumina and sulphuric acid in equivalent amounts; this may be conveniently ascertained by making a colorimetric p_H determination, using *p*-sulphobenzeneazobenzylaniline. The above method will detect the presence of 0.15% of free acid in the salt, and the presence of basic salt is also indicated, which is not the case when the usual thio-sulphate method is employed.

H. F. HARWOOD.

Sources of error in the determination of hydrogen in gases.

H. R. AMBLER (Analyst, 1930, 55, 436—443).—In determining hydrogen by the explosion method, error introduced by the oxidation of nitrogen is negligible for mixtures containing less than 20% of hydrogen, but above this the amount of nitrogen peroxide increases rapidly with increase of hydrogen content. With the slow-combustion method the oxidation is negligible. Completeness of combustion in the explosion method varies; in mixtures containing 10—20% of hydrogen at least 99% of the hydrogen is oxidised, but for poorer mixtures combustion may be incomplete even when there is a definitely visible explosion, the error even extending to 40% on the total hydrogen when less than 10% of hydrogen is present. The degree of excess of oxygen makes no apparent difference to the completeness of combustion. Even when 6.5% of methane is present the amount of unburnt hydrogen is not increased.

D. G. HEWER.

Decolorisation of oils. CHOWDHURY and DAS.—See II. Purifying gases. HEALY.—See XI. Coloured-light recipes. VANINO.—See XXII.

See also A., July, 861, System K_2CO_3 - $Ca(OH)_2$ - KOH - $CaCO_3$ (USANOVITSCH and BOROVIK). 867, Catalytic reactions at high pressures (MORGAN; APPLEBEY). 868, Electrochemical production of ammonium persulphate (IZGARISCHEV and GRUZDEVA). 872, Production of potassium phosphates (ASKENASY and NESSLER). 875, Metal carbonyls (HIEBER and others).

PATENTS.

Production of phosphoric acid and hydrogen. BAYERISCHE STICKSTOFF-WERKE A.-G. (B.P. 308,599, 19.3.29. Ger., 24.3.28. Addn. to B.P. 308,598; B., 1930, 660).—In the process described in the prior patent a certain amount of phosphorous acid is produced; this is converted into phosphoric acid and hydrogen by heating under about 600 atm. at below 600°.

A. R. POWELL.

Heating of gases [for ammonia synthesis]. GAS-VERARBEITUNGSGES. M.B.H. (B.P. 307,027, 28.2.29. Ger., 1.3.28).—The gas is first preheated to 400° in a heat exchanger by the hot gases from the ammonia catalysis

chambers, and then heated to the desired temperature by passing it countercurrent to a stream of nitrogen heated above 500° by passing it through a metal coil immersed in molten lead. Alternatively, or in addition, the gas mixture may be preheated during the purification process by adding small quantities of oxygen or of carbon monoxide and passing the mixture over heated catalysts comprising metals of group VIII.

A. R. POWELL.

Preheating of ammonia gas in the execution of reactions therewith at elevated temperatures.

I. G. FARBENIND. A.-G. (B.P. 307,843, 13.3.29. Ger., 14.3.28).—The preheater consists of coils of copper, silver, aluminium, or magnesium, or alloys of these metals with one another or with silicon, *e.g.*, 10% silicon-copper. Mixtures of ammonia and carbon monoxide may be preheated at 590–620° without appreciable dissociation of the ammonia taking place.

A. R. POWELL.

Production of ammonium sulphate. W. G. ADAM, D. G. MURDOCH, and GAS LIGHT & COKE Co. (B.P. 330,945 and 330,947, 20.3.29).—A solution of ammonium sulphate produced (A) by the interaction of ammonia, carbon dioxide, and calcium sulphate, or (B) by saturation of sulphuric acid with ammonia, is evaporated until it contains 45% of ammonium sulphate; ferric sulphate equivalent to 0.003% Fe on the amount of ammonium sulphate present and sulphuric acid equal to 0.2% of the solution are added, and the solution is boiled at constant temperature to give a controlled rate of crystallisation. In this way a loose crystalline mass is obtained which does not cake.

A. R. POWELL.

Manufacture of carbamates and conversion products thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,737, 11.3.29).—Ammonia and carbon dioxide are made to react in an inert liquid in presence of metal salts, or of alkaline-earth oxides or hydroxides, soluble to some extent in the liquid, the latter being one in which the metal carbamate is substantially insoluble. *E.g.*, carbon dioxide and ammonia are passed at ordinary temperature and pressure into methyl-alcoholic calcium chloride solution, or carbon dioxide into a suspension of calcium hydroxide in alcohol saturated with ammonia. Calcium, lead, or sodium nitrate in methyl alcohol, lead acetate in ethyl alcohol, or calcium nitrate and chloride in formamide may also be used. The metal carbamate gives the cyanamide when heated in ammonia gas.

C. HOLLINS.

Electrolysis of fused halogen salts. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,791, 1.7.29).—Electrolysis is effected at a temperature below red heat, using a tungsten anode or a metal or graphite anode coated with tungsten. Pure beryllium may thus be obtained from a bath containing 73% BeCl₂ and 27% NaCl at 420–450°, and pure aluminium from a bath containing 82% AlCl₃ and 18% NaCl at 200°.

A. R. POWELL.

Production of potassium nitrate with simultaneous obtention of dimagnesium phosphate. CHEMIEVERFAHREN-GES.M.B.H. (B.P. 329,939, 11.10.29. Ger., 28.11.28).—Crude phosphate is decomposed with nitric acid and potassium sulphate, according to the

known process. A solution containing potassium nitrate and phosphoric acid results; the bulk of the phosphoric acid is separated out from this as dimagnesium phosphate by neutralisation with oxide, hydroxide, or carbonate of magnesium (or a mixture or compound thereof). The phosphoric acid remaining in the solution (preferably to the extent of about 1%) is then removed by neutralisation, preferably by being precipitated as magnesium orthophosphate, and the potassium nitrate is separated out by crystallisation or evaporation. The magnesium orthophosphate obtained is used to assist in the precipitation of magnesium diposphate from fresh initial materials.

S. K. TWEEDY.

Manufacture of crystalline phosphates. VICTOR CHEM. WORKS, ASSEES. OF N. C. LINDBERG and G. A. McDONALD (B.P. 316,136, 9.7.29. U.S., 23.7.28).—The aluminium and iron phosphates present in phosphate crystals obtained by neutralising concentrated phosphoric acid are removed by washing or flotation. Any liquid which does not impair the crystals may be used, preferably the filtered mother-liquor or a saturated solution of the desired phosphate. The wash-liquor may be passed through oxidising tanks in order to precipitate any ferrous phosphate as ferric phosphate; it may then be added to the phosphoric acid before neutralisation.

S. K. TWEEDY.

Production of diammonium phosphate. FEDERAL PHOSPHORUS Co., ASSEES. OF B. G. KLUGH and W. R. SEYFRIED (B.P. 315,700, 20.6.29. U.S., 16.7.28).—A mixture of 5–6 pts. of phosphoric acid (as a 70% solution) and 1 pt. of diammonium phosphate (as a cold saturated solution) is treated in a heat-insulated vessel with ammonia gas until the composition of the salts in solution corresponds with a 5:1 mixture of mono- and diammonium phosphates. The hot, highly concentrated solution is run into a closed vessel containing about half its volume of a cold saturated solution of the diammonium salt, whereby the temperature of the mixture falls to 80°. An atmosphere of ammonia is maintained above the solution, which is agitated until all the phosphate is converted into the di-salt. The ammonia is then replaced by air and the mixture cooled with agitation at the rate of 10°/hr. The crystals of di-salt are separated and about one third of the mother-liquor is returned to the first saturator and the remainder to the second saturator.

A. R. POWELL.

Manufacture of triple superphosphates. F. C. PALAZZO (B.P. 310,479, 26.4.29. It., 26.4.28).—Substantially pure precipitated dicalcium phosphate of high phosphoric anhydride content (*e.g.*, 38%) is mixed with phosphoric acid, preferably concentrated, in quantity not exceeding that necessary for the formation of monocalcium phosphate. When the reaction mixture corresponds to that represented by the equation: $H_3PO_4 + CaHPO_4 \cdot 2H_2O = Ca(H_2PO_4)_2 \cdot H_2O + H_2O$, the products contain at least 50% of water-soluble P₂O₅. Preferably, the reaction components are mixed, then dried after a few hours at about 50°, and crushed.

S. K. TWEEDY.

Manufacture of phosphoric acid esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,228,

31.1.29).—Phosphorus oxychloride is allowed to react with an aliphatic or aromatic alcohol or mixtures thereof at 0–5°, the mixture is then heated at about 40°, the hydrochloric acid formed removed by washing with water and then alkali, and the ester purified by distillation under reduced pressure. The preparation of the following esters of phosphoric acid is described: tributyl, *n*-butyl diisooamyl (b.p. 145–146°/4.5 mm.), diisooamyl cyclohexyl (b.p. 142°/0.5 mm.), and phenyl dibutyl (b.p. 183–185°/15 mm.).
A. R. POWELL.

Production of calcined borax. AMER. POTASH & CHEM. CORP. (B.P. 330,146, 14.6.29. U.S., 11.3.29).—Borax is calcined while retaining its shape and size by subjecting it in stages to a stream of air, or other dry gas, having a temperature at each stage such that the borax cannot melt or dissolve in its water of crystallisation and having a humidity such that water of crystallisation is picked up at each stage. Five stages may be employed, the temperature and humidity (in lb. of water vapour per lb. of air) for each being: (1) 50–100°, 0.08; (2) 65–150°, 0.06–0.08; (3) 125–200°, 0.04–0.06; (4) 175–260°, 0.02–0.04; (5) 225–325°, 0.0–0.02. Two mols. of water are lost in each stage except the last, where only one is lost, the final product being the monohydrate having an apparent sp. gr. of 0.5–0.9. Borax containing 8–12% H₂O, or having 1–3 mols. of water, is most advantageously produced by the process.
S. K. TWEEDY.

Borax product and its production. AMER. POTASH & CHEM. CORP. (B.P. 330,453, 2.8.29. U.S., 9.4.29. Addn. to B.P. 330,146; preceding).—Borax is heated at 50–100° in a current of warm air containing less than 10% of moisture until 20% of the combined water is removed; the temperature of the air is raised to 65–150° and its humidity decreased below 8% to remove a further 20% of water, then to 125–200° with less than 6% humidity, and finally to 175–260° with less than 4% of moisture, whereby a cellular, dense product, Na₂B₄O₇·3H₂O, is obtained which is readily soluble in water.
A. R. POWELL.

Production of [crystalline] aluminium oxide. VEREIN. ALUMINIUM-WERKE, A.-G. (B.P. 313,409, 10.6.29. Ger., 9.6.28).—A mixture of bauxite and sulphur-containing materials (pyrites) is subjected to a reducing smelting treatment, and the molten product, consisting to a large extent of aluminium sulphide, is chilled, e.g., by pouring on to a cooled surface, or by pouring into water or into molten aluminium.
S. K. TWEEDY.

Simultaneous preparation of alumina and pure carbon dioxide. C. D'ASSEEV (B.P. 318,976, 13.9.29. Belg., 13.9.28).—A solution of crude aluminium sulphate containing iron and obtained by treating aluminium ores with sulphuric acid is treated cold with magnesium hydrogen carbonate, whereby basic iron and aluminium carbonates are precipitated, carbon dioxide is evolved, and a solution of magnesium sulphate is obtained. The precipitate is separated from the mother-liquor and heated at 90–100° to obtain pure carbon dioxide; the residual hydroxides are digested with 5% sodium hydroxide solution to obtain ferric hydroxide for use in gas purification and a solution of sodium aluminate from

which the alumina is reprecipitated in a pure form by treatment with the carbon dioxide evolved in the first stage of the process or by agitation with alumina.

A. R. POWELL.

Preparation of alumina. W. J. MÜLLER and H. HILLER (B.P. 330,661, 19.3.29).—Bauxite is heated under pressure at above 190° with a solution of sodium hydroxide of *d* 1.16–1.18. After removal of the insoluble red slime the aluminate solution so obtained may be directly inoculated with aluminium hydroxide as in the Bayer process and, after separation of the alumina, the alkaline liquor may be returned to the autoclaves without being concentrated further.
A. R. POWELL.

Removal of one or more metals of the iron group from solutions containing salts of one or more of these metals [e.g., iron from aluminium salts]. K. W. PALMAER (B.P. 318,149, 12.8.29. Appl., 28.8.28).—Iron is removed from solutions of aluminium sulphate obtained by the acid decomposition of aluminium ores by reducing any ferric salt to the ferrous state, neutralising the solution, and electrolysing it between a lead anode and a mercury cathode which is kept continuously in movement. The iron is thus deposited as an amalgam and a pure solution of aluminium sulphate is obtained.
A. R. POWELL.

Production of magnesium oxide from kieserite and other salts or salt mixtures containing magnesium. KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 330,715, 7.5.29. Ger., 12.1.29).—Kieserite, schönite, or other natural or artificial sulphates containing magnesium, potassium, and sulphate are made into a slurry with water and the latter is saturated with ammonia, whereby ammonium sulphate is formed in solution and an insoluble residue of crystalline anhydrite and/or potassium sulphate and magnesium hydroxide is obtained. By continuous decantation of the mother-liquor the magnesium hydroxide may be separated from the other insoluble constituents. After filtering off the magnesia the mother-liquor is returned to the saturator for use again.
A. R. POWELL.

Separating magnesium chloride from its aqueous solutions and obtaining anhydrous magnesium chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,650, 15.2.29. Addn. to B.P. 329,032; B., 1930, 644).—A 30% solution of magnesium chloride is treated with 64% of its weight of ammonium chloride, saturated with ammonia, and cooled to –30°. The crystals of MgCl₂·6NH₃ which separate are collected, washed with liquid ammonia, and heated at 500° to remove the ammonia for use again; the residue consists of anhydrous magnesium chloride.
A. R. POWELL.

Obtaining hydrogen peroxide by distillation. DEUTS. GOLD-UND SILBER-SCHNEIDANSTALT VORM. ROESSLER, G. BAUM, and OESTERR. CHEM. WERKE GES.M.B.H. (B.P. 330,255, 20.2.29).—About 200–250 litres/hr. of dilute sulphuric acid containing 25% of persulphuric acid are drawn by suction under 650–700 mm. of vacuum through a lead pipe, 60 m. long and 75 mm. internal diam., wound into 15–25 coils and immersed in a steam jacket. The hydrogen peroxide evolved is recovered by fractional condensation of the vapours evolved.
A. R. POWELL.

Manufacture of manganese dioxide. Y. KATO and K. YAMAMOTO (B.P. 330,257, 26.2.29).—Lower oxides of manganese or materials containing them are heated with 3–5% of an alkali hydroxide or carbonate in the presence of air or oxygen at 500°. The product is cooled, ground, and leached, first with water and then with dilute acid. [Stat. ref.] A. R. POWELL.

Production of zirconium oxide [from zirconium mineral]. A. KARL (B.P. 314,526, 18.6.29. Belg., 30.6.28).—The mineral is fused with sodium carbonate. The product (sodium zirconate), after removing the sodium silicate by washing, is dissolved in excess sulphuric acid and the iron and titanium sulphates contained in the resulting solution are reduced (*e.g.*, by means of nascent hydrogen, sulphur dioxide, or sodium hyposulphite). Pure zirconia is then precipitated out as the result of hydrolysis by neutralising the solution, *e.g.*, with sodium carbonate. The initial fusion is conveniently effected in a furnace lined with zircon, the melt being allowed to run straight into water. The precipitated sodium zirconate is preferably dissolved at 100–400° in sulphuric acid having a concentration between that of fuming acid and acid of *d* 1.71.

S. K. TWEEDY.

Production of hydrogen peroxide. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 309,602, 11.4.29. Fr., 14.4.28).—Electrolytic hydrogen containing 4% O₂ is passed over a platinum catalyst at 500–600° under 400 atm. and thence through a condenser maintained at 0°. The condensate is a 2.9% solution of hydrogen peroxide. Combustion of oxygen under high pressure in an atmosphere of hydrogen under pressure also affords a dilute solution of hydrogen peroxide. A. R. POWELL.

Removal of free chlorine and bromine from fluid mixtures. J. B. PAYMAN and H. A. PIGGOTT, ASSRS. TO BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,770,010, 8.7.30. Appl., 23.4.28. U.K., 27.4.27).—See B.P. 292,307; B., 1928, 604.

Processes and apparatus for liquefying solid carbon dioxide. MASCHINENFABR. ESSLINGEN, and J. STOFFELS (B.P. 331,750, 3.9.29).

Gas-analysis apparatus (B.P. 330,799).—See I. [Hydrogen from] brown coal (G.P. 462,388). Separation of ammonia from coal gas (G.P. 462,186).—See II. Activation of chemical reactions (B.P. 330,226). Treatment of mica (B.P. 329,878).—See XI. Lakes or pigments (B.P. 307,436).—See XIII. Compressed iodine (B.P. 331,118).—See XX.

VIII.—GLASS; CERAMICS.

Effects of additions of zinc oxide and cadmium oxide on simple glasses. C. W. PARMELEE and R. G. EHMAN (J. Amer. Ceram. Soc., 1930, 13, 475–488).—The small batches of glass were melted and fired in a vacuum electric furnace, which is fully described. A simple Na₂O–PbO–SiO₂ glass was used, the Na₂O content varying from 5% to 20% in steps of 5%. This constituent content was replaced by additions of zinc and cadmium oxides, and the effect of such variations on thermal expansion, refractive index, acid-resistance,

and solubility was determined. The difference in expansion indicated occurs only in the higher temperature ranges. Glasses containing above 10% ZnO (or CdO) showed a greatly increased refractoriness and viscosity. These oxides are stated to impart desirable properties to enamels, and additions of up to 10% act as fluxes, decrease the viscosity, increase the acid-resistance, and improve the appearance. R. J. CARTLIDGE.

Effect of additions of calcium and magnesium oxides on enamel glasses. C. W. PARMELEE and D. T. H. SHAW (J. Amer. Ceram. Soc., 1930, 13, 498–508).—A simple glass containing SiO₂ 58%, PbO 22%, and Na₂O 20% was used, and various amounts of calcium and magnesium oxides were added. The tests consisted of measurements of the thermal expansion, refractoriness, compressive strength, modulus of elasticity, solubility, and index of refraction of the enamels produced. A glass consisting of SiO₂ 58%, PbO 22%, CaO (or MgO) 5–10%, Na₂O remainder gave the best enamel. R. J. CARTLIDGE.

Systematic method for the investigation of sheet-iron enamels. A. I. ANDREWS (J. Amer. Ceram. Soc., 1930, 13, 489–497).—Selected compositions of 24 sheet-iron ground coats and 24 cover enamels are considered. Tables and triaxial diagrams are given to simplify the study of relative amounts of felspar, borax, and flint. A description of the utility of the three most common triaxial diagrams is appended.

R. J. CARTLIDGE.

Reactivity test for determining the value of barium carbonate as a scum preventive [on glazed ware]. A. L. BENNETT and H. R. GOODRICH (J. Amer. Ceram. Soc., 1930, 13, 461–469).—A discussion of the causes of scumming and glaze peeling is followed by a description of the Howat and Williams reactivity test for barium carbonate (Bull. Amer. Ceram. Soc., 1923, 2, 161) and a method for determining soluble sulphates in clays. Tables given indicate the reactivities of various barium carbonates, and comparative effects of barium carbonate and barium chloride in a terra-cotta body. A reactivity value of 22–23% is stated to be desirable. R. J. CARTLIDGE.

Rate of vitrification of porcelain. C. W. PARMELEE and A. E. BADGER (Ind. Eng. Chem., 1930, 22, 781–785).—The rate of vitrification of a typical porcelain has been followed by determining the changes in volume and in porosity brought about by different conditions of heating in an electric furnace. At 890° the initial increase in volume is maintained at a constant value, whilst at 995° it is followed by a gradual decrease in volume as the time of heating is increased. At higher temperatures, 1090° and 1200°, this decrease is greater and sets in more rapidly. The porosity varies in a similar manner, the decrease being the greater the higher is the temperature (up to 1200°) at which the sample is heated. These changes are in accord with the transformations which are known to take place when a porcelain body is fired. L. S. THEOBALD.

Thermal expansion of silica brick and mortars. S. S. COLE (J. Amer. Ceram. Soc., 1930, 13, 437–446).—A more complete study of the thermal expansion of

American and European silica brick and mortars is presented. Data are given in the form of curves showing values for the expansion of silica brick varying between 1.15% and 1.30% at 950° and of mortars between 1.30% to 1.52%, depending on the content of clay, quartzite, and bats. Apparent sp. gr. and porosity values are also given; porosity appears to have little effect on expansion. R. J. CARTLIDGE.

Special refractories for electric furnace linings.

(a) **Silica, corundum, and carborundum bricks.** O. KUKLA (Stahl u. Eisen, 1930, 50, 800—803).

(b) **Sillimanite bricks.** F. SOMMER (*Ibid.*, 804).—

(a) Experiments with a 7-ton Héroult-Lindenburg electric steel furnace show that carborundum bricks behave well as a refractory in the roof of the furnace in a reducing atmosphere, but are not as good as silica bricks for the body of the furnace in contact with the molten charge. In this position the carborundum is rapidly burned at the surface of the bricks, thus causing rapid wear. Corundum bricks are unsatisfactory owing to their great tendency to spalling.

(b) Sillimanite bricks behave no better than good silica bricks in electric steel furnaces, and are four times as expensive. A. R. POWELL.

Clays. BERRIDGE.—See IX.

See also A., July, 847, **Fusion diagrams of highly refractive oxides** (VON WARTENBERG and WERTH). 857, **Production of kaolin and kaolinite** (ENK). 874, **Silvering of glass** (VON WARTENBERG). 885, **Determination of coefficients of expansion of glasses and ceramic materials** (THULENIUS and HOLZMANN). 968, **Testing of ampoule glass for alkalinity** (STICH).

PATENTS.

Stratified bodies such as strengthened glass.

C. R. CROSS, C. J. G. FOX, L. G. S. HEBBS, and TRIPLEX SAFETY GLASS CO., LTD. (B.P. 324,898, 3.11.28).—Sheets of cellulose acetate may serve to unite one or more sheets of glass if diacetone alcohol is used as the medium, the coating being applied to the nitrocellulose enamel with which the gelatin-coated glass surface is normally covered. In place of diacetone alcohol, "acetone oil," the monomethyl ether of ethylene glycol, or the monoethyl ether of diethylene glycol may be used. M. PARKIN.

Manufacture of safety glass. I. G. FARBENIND. A.-G. (B.P. 315,667, 15.7.29. Ger., 14.7.28).—The middle layer in safety glass is made of the cellulose ester of a fatty acid containing more than 3 carbon atoms, *e.g.*, laurate, acetate-butyrate, butyrate-laurate, these esters being fast to light and water. Adhesion may be increased by addition of resin, such as polymerised vinyl acetate, or other softener, *e.g.*, resorcinol monoacetate; or a sticky layer, such as cellulose naphthenate with vinyl acetate resin and resorcinol monoacetate, may be spread on the two glass plates, which are then applied to a film of the cellulose ester. C. HOLLINS.

Making compound transparent sheets [laminated glass]. L. BARTELSTONE (B.P. 331,030, 27.4.29. U.S., 13.3.29).—A sheet of celluloid, conditioned by baking for 5 days in a current of warm air at about 37°, is cleaned while "earthed" to prevent electrification,

resurfaced by spraying with a free-flowing celluloid solution, and the residual solvent from this removed by baking for 30 min. at 65—71°. Two plates of cleaned glass are then given a thick coating of castor oil on their upper faces, the celluloid sheet is laid on one, and the other quickly turned over on to it, the process being completed by subjecting to suitable temperature and pressure. M. PARKIN.

Manufacture of [laminated] safety glass. DU PONT VISCOLOID CO. (B.P. 306,891, 14.1.29. U.S., 27.2.28).—Laminated glass possessing better adherence and not so liable to discoloration is produced by using a cellulose ester plastic from the sheeting machine without polishing, coating it with a cement (*e.g.*, gelatin) to give a film not greater than 0.0005 in., placing it between the polished glass sheets and subjecting the sandwich to hot water (110—115°) under pressure (150—500 lb./in.²). M. PARKIN.

Luminous enamels and their application. A. ANDRÉ (B.P. 325,072, 10.1.29).—An aluminous flux (2 pts.), for which three preferred compositions are given, is finely powdered after melting at 900—1100°, and mixed with 1 pt. of phosphorescent material, such as zinc sulphide, together with 6—7% of white clay (*e.g.*, kaolin), the mixture being made into a paste with water. The clay should be pretreated with potassium permanganate solution to destroy organic matter. Surfaces of metal, porcelain, or ceramic bodies may be decorated by applying this paste and, when dry, firing as usual. Oxidisable metal parts should be covered with a protective coating, preferably having a cobalt base. M. PARKIN.

Production of hollow bodies from silica and other refractory materials. W. C. HERAEUS GES.M.B.H., and A. KRÖNER (B.P. 330,943, 20.3.29).—Fused silica tubes etc. are produced by feeding pulverulent quartz into a furnace rotating about a horizontal axis, which carries a mould either of water-cooled metal or of refractory material readily detachable from the product afterwards, or the lining may consist of granules of the material itself. A carbon arc is struck to play along the mould, and the fused quartz particles are spread by centrifugal force (100 r.p.m. for a tube 100 mm. in diam.) against the sides of the mould. M. PARKIN.

Manufacture of objects [glass, enamel] from non-conducting materials. A. UHLMANN, ASSR. to A. T. OTTO & SONS (U.S.P. 1,768,869, 1.7.30. Appl., 14.7.26. Ger., 18.7.25).—See B.P. 255,497; B., 1928, 264.

Forming ceramic articles and the like. T. S. CURTIS, ASSR. to VITREFRAX CORP. (U.S.P. 1,768,546, 1.7.30. Appl., 16.11.26).—See B.P. 291,523; B., 1928, 605.

Weatherproof laminated glass. L. BARTELSTONE (B.P. 325,048, 13.12.28).

IX.—BUILDING MATERIALS.

Colloidal nature and water content of clays. H. BERRIDGE (Engineering, 1930, 130, 5—7, 61—63).—Whilst most investigations of clay have been carried out from the ceramic point of view, a study of clay has now been made so far as it affects the foundations of engineering and architectural structures. Data

obtained by the author and by other workers for the water content, volume weight, tensile strength, and compressive strength of various clays in different states are listed. The volume weight, or density of the total system, affords for engineering purposes a valuable means of classification which has so far not been adopted. When dried, clay shrinks until its water content has fallen to about 12.5%; its volume then ceases to diminish, although evaporation of water proceeds until the porous mass contains about 5% of water, which can be driven off at 110°. If the temperature is kept below 415° the resulting "argil" re-absorbs moisture, but not if heated above that temperature. The effect of absorption of water on the mechanical properties and volume changes of clay is discussed in relation to the digging of trenches for foundations and the construction of clay embankments, and the most efficient and economical courses are indicated. Dried clay expands when wetted, even when loaded to above 5 tons per sq. ft. If left exposed to the atmosphere, the clay absorbs about 6% of water without volume change. E. S. HEDGES.

Corrosion of structural steel and steel reinforcing rods encased in concrete. E. A. CROSS (Engineering, 1930, 130, 30).—An engine-room floor composed of continuous concrete 6 in. thick supported on 18-in. steel joists (covered with concrete) was originally provided with reinforcement in the lower part of the slabs only, *i.e.*, no steel was provided to take tensional stresses in the upper half of the floor, where it passed over the joists. The floor carried a brine tank, which for some years had been leaking; there were external signs of severe corrosion, especially along the joists where tension cracks were noticed. A large area of floor was removed, and the joists and rods were found to have corroded to vanishing point in places. A minor contributory cause was considered to be condensation around ventilating holes, which had been left in the floor, for the room below was at a much higher temperature. A method of repair, which provides a dense and impervious concrete with adequate reinforcement, is described. B. M. VENABLES.

Corrosion of steel by breeze and clinker concretes. F. L. BRADY (Dept. Sci. Ind. Res., Building Res., Rep. No. 15, 1930, 15 pp.).—Breeze and clinker aggregates promote the rusting of steel even under dry indoor conditions, the rate of corrosion of steel in contact with these aggregates being roughly proportional to their sulphur content. The deleterious effect of breeze and clinker in reinforced concrete is due to their high porosity, which permits the ready diffusion of oxygen and moisture into the mass with consequent rapid oxidation of the sulphur compounds present. The substitution of sand for the finer portion of coal residue aggregates reduces rusting owing to the diminution in the amount of accessible sulphur compounds, and not to the reduced porosity. When clinker low in sulphur and free from fines is used together with sand in concrete aggregates, only very slow rusting of embedded steel reinforcement occurs; if, however, the fines are not removed, fairly rapid corrosion occurs. These results indicate that coal residues are unsuitable for the

preparation of concrete which is to come into contact with steel.

A. R. POWELL.

Action of salts, acids, and organic substances on cement and concrete. R. GRÜN (Z. angew. Chem., 1930, 43, 496—500).—Addition of calcium, barium, aluminium, ferric, or hydrogen chloride to the water used in making cement or concrete mixtures increases appreciably the rate of setting and the tensile and compressive strengths, probably owing to the formation of calcium chloride which acts as an accelerator. Formaldehyde, sugars, and similar organic compounds, even in small quantities, retard or almost completely prevent the hardening of cement. Oxalic acid tends to stabilise cement mixtures by combining with the free lime, and has no action whatever on hardened cements. Phosphoric acid, on the other hand, slowly disintegrates cement structures by forming soluble acid phosphates, but its action in low concentrations is exceedingly slow.

A. R. POWELL.

See also A., July, 872, **Hydrated calcium aluminates** (TRAVERS and SEHNOUTKA). 967, **Drying of timber** (CAMPBELL and BOOTH). 968, **Destruction of coniferous wood by beetle larva** (FALCK).

Tar-asphalt mixtures. SCHLÄPFER.—See II. **Thermal expansion of mortars.** COLE.—See VIII.

PATENTS.

Piston pump for raising viscous mixtures such as concrete, mortar, etc. M. GIESE and F. HELL (B.P. 319,347, 30.5.29. Ger., 21.9.28).

Pulp-board (U.S.P. 1,746,451).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Potentiometric determination and separation of chromium, vanadium, and molybdenum, applied to steel analysis. W. TRZEBIATOWSKI (Rocz. Chem., 1930, 10, 411—436).—Steel containing chromium, vanadium, molybdenum, and tungsten is dissolved in concentrated hydrochloric acid to which one third of its volume of water has been added, insoluble tungsten being then dissolved by the addition of nitric acid. Tungstic acid is precipitated by dilution and is removed by filtration. The filtrate is concentrated to a syrup, which is transferred to a crucible containing excess of sodium hydroxide, ignited, fused with excess of sodium peroxide, and extracted with boiling 2*N*-sodium hydroxide containing 2—3 c.c. of hydrogen peroxide. The precipitate of hydroxides is filtered off, and the filtrate is concentrated to about 80 c.c., neutralised with 50% sulphuric acid, and the volume made up to 200 c.c. with 25% sulphuric acid. 50 C.c. of the solution are now titrated with stannous chloride solution, in an inert atmosphere, to the first abrupt fall in potential, corresponding to reduction of sexa- to ter-valent chromium, and of quinque- to quadri-valent vanadium. Ferrous ammonium sulphate and concentrated sulphuric acid to a final concentration of 50% are now added, the system is warmed at 90—100°, and stannous chloride is added to the second potential fall, corresponding to reduction of quadri- to ter-valent vanadium, and of sexa- to quadri-valent molybdenum. The solution is

now diluted to 100 c.c., and to 50 c.c. 20 c.c. of 25% ammonium sulphate and 50 c.c. of 4% potassium bromate solutions are added; the solution is then diluted so that for every 5 c.c. of concentrated sulphuric acid present there are 60 c.c. of solution. The solution is then heated for 10 min. at 60° and for 5 min. at boiling, a stream of carbon dioxide being passed in in order to remove bromine. After cooling to 40–50°, 25 c.c. of concentrated sulphuric acid are added and the solution is titrated at 60° with stannous chloride to the third potential fall, corresponding to reduction of quinquo- to quadri-valent vanadium. The percentage chromium, vanadium, and molybdenum contents are then given, respectively, by $346 \cdot 6M(b - 2cM'/M)/mi$, $2040M'c/mi$, and $1920M(a - 2cM'/M)/mi$, where m represents the weight of steel taken, a , b , and c the number of c.c. of stannous chloride solution used for the first, second, and third potential falls, i the number of c.c. taken for titration, and M and M' are the titres of the stannous chloride solution determined alone and under conditions analogous to those used in the experiment. Molybdenum catalyses the reduction of quadri- to ter-valent vanadium; should it be absent from the solution it should, therefore, be added.

R. TRUSZKOWSKI.

Pressure-ageing of duralumin. L. PESSEL (Ind. Eng. Chem., 1930, 22, 776–777).—The application of pressure to duralumin during the ageing period reduces its susceptibility to intergranular corrosion. Tensile strength and hardness are negligibly decreased by this treatment, but elongation is increased. After exposure to corrosion, however, the decrease in tensile strength and in elongation is much smaller in duralumin which has been aged under pressure. L. S. THEOBALD.

Resistance of nickel to corrosion. R. KRULLA (Chem.-Ztg., 1930, 54, 429–431).—Curves showing the rate of dissolution of nickel in various corroding media at room temperature are given and the effect of various alloying elements on the resistance of nickel to corrosion is discussed. Up to 4% Fe decreases greatly the rate of dissolution in nitric acid, carbon increases the resistance to attack by molten alkalis, and chromium and molybdenum improve the resistance towards acetic and hydrochloric acids, respectively. A nickel alloy with 20% Mo loses only a few cg./hr. in hot concentrated hydrochloric acid. In contact with reducing flames or hot gases containing sulphur compounds nickel rapidly becomes brittle due to absorption of carbon or sulphur.

A. R. POWELL.

Composition of deposit forming on zinc immersed in cupric sulphate solution. I. A. GALECKI and J. TOMASZEWSKI (Rocz. Chem., 1930, 10, 437–471).—The coherence of the deposit forming on zinc rods immersed in cupric sulphate solution increases with stirring and with the concentration and acidity of the solutions; at the same time the colour of the deposit becomes lighter. The deposit contains two constituents: (a) a metallic, flexible, red, yellow, or white coating, possessing either a dull or a polished surface; and (b) a dark brown to black powdery or flocculent precipitate. The former consists of metallic copper and zinc in various proportions, probably both as a mixture

and as alloys, whilst the latter contains oxides of the two metals, as well as basic sulphates and the free metals.

R. TRUSZKOWSKI.

Technical preparation of rhenium. W. FEIT (Z. angew. Chem., 1930, 43, 459–462).—An account is given of researches made on the extraction of rhenium from a complex sulphide slime obtained as a by-product in an unstated metallurgical process. The slime had weathered in the air for many months and on extraction with water yielded a brownish-green solution from which nickel and copper sulphates were obtained by evaporation. By the addition of regulated quantities of ammonium sulphate most of the copper, nickel, and zinc were separated as double ammonium sulphates. The almost black mother-liquor had $d > 2$, and with more ammonium sulphate yielded dark violet crystals of the ammonium salt of a hetero-poly-acid containing molybdenum, vanadium, and phosphorus; the pale yellow mother-liquor yielded crystals of potassium perrhenate on addition of potassium chloride. Large quantities of the slime are being worked up as follows: it is heated at 100° with constant rabbling for 1 month, whereby almost complete oxidation is effected; the residue is extracted with water and the solution evaporated in stages to remove zinc, copper, nickel, and ferrous sulphates. When traces of potassium perrhenate begin to separate the solution is diluted with mother-liquor from a previous crop of crystals and treated with a large excess of potassium chloride. The grey, impure perrhenate is collected, washed with cold water, and dissolved in hot dilute sodium hydroxide; on filtering off the precipitated hydroxides and cooling the solution relatively pure potassium perrhenate is obtained and may be further purified from traces of molybdenum and vanadium by repeating the recrystallisation twice. In this way a product with 64·6% Re and less than 0·01% of impurities has been obtained at the rate of 10 kg. per month at a cost of 10 s. per g. A. R. POWELL.

Resistance of metals to the system urea-water-ammonium carbamate. J. G. THOMPSON, H. J. KRASE, and K. G. CLARK (Ind. Eng. Chem., 1930, 22, 735–736; cf. Krase and co-workers, B., 1930, 408).—The corrosion of 36 metals, mainly alloys, by this system at 142° under pressure has been investigated.

L. S. THEOBALD.

See also A., July, 846, **Recrystallisation of lead** (GARRE and MÜLLER). 863, **Electrode potentials of silver in cyanide solutions** (FINK and HOGABOOM). 870, **Electrodeposition of metals from their liquid ammonia solutions** (TAFT and BARHAM). 874, **Silvering of glass** (VON WARTENBERG). 882, **Determination of metals by electrodeposition** (TUTUNDŽIĆ). **Test for tin** (MEISSNER).

Adhesives and adhesion. LEE.—See I. **Sheet-iron enamels**. ANDREWS.—See VIII. **Corrosion of structural steel**. CROSS. **Corrosion of steel by concrete**. BRADY.—See IX.

PATENTS.

Siemens-Martin furnaces. H. WADE. From "TERNI" SOC. PER L'IND. E L'ELETTRICITA (B.P. 329,176, 24.5.29).—The furnace has superimposed air and gas

ducts, the lower wall of the upper or air duct being downwardly inclined towards the gas duct, and the upper and lower walls of the air duct diverging towards the mixing and combustion chambers, so that the angle at which the air and gas streams meet can be varied by altering the supply pressures. A. R. POWELL.

Electrical devices for heating a metal melt within a ladle. F. KRUPP A.-G. (B.P. 312,063, 8.5.29. Ger., 18.5.28).—The device comprises a hollow cylinder of refractory material in which is embedded a water-cooled copper coil through which a high-frequency current is passed. The apparatus is immersed in molten steel in the ladle so as to keep it hot by inductive heating until the slag has separated. A. R. POWELL.

Furnace ladles. F. KRUPP A.-G. (B.P. 312,064, 8.5.29. Ger., 18.5.28).—The ladle comprises a shell built up of annular rings of non-magnetic chromium-nickel-iron alloy insulated from one another and provided with holes to prevent generation of eddy currents. The shell is lined with a refractory material in which is embedded a water-cooled copper coil, by the aid of which the ladle contents may be heated by induction. A. R. POWELL.

Crucible furnaces for melting metals. H. BAILEY (B.P. 329,365, 13.2.29).—A crucible furnace comprises a heating chamber tapering inwards towards the top and provided with fire-bars at the bottom on which the crucibles and fuel are placed. Air under pressure is admitted into the ash-pit below the fire-bars and is there preheated before passing into the fuel chamber; the ashes are sprayed with water to damp them down and to provide steam to aid the combustion of the fuel. A. R. POWELL.

Case-hardening furnace with charging device of worm type. H. LINDHORST (B.P. 330,188, 22.8.29).—The case-hardening chamber consists of a horizontal rotating drum closed at one end with a cap carrying a tube inside which is a worm-charging device, the whole rotating round a pyrometer tube through the longitudinal axis of the worm. At the end of the worm-tube outside the drum is a hopper through which more of the case-hardening mixture is fed into the drum during use, so that the case-hardening gases are generated regularly throughout the operation. A. R. POWELL.

Manufacture of tools of homogeneous alloys of great hardness. G. BREWER. From F. KRUPP A.-G. (B.P. 331,121, 25.7.29).—Carbides, borides, or silicides of tungsten, molybdenum, chromium, uranium, titanium, or vanadium are obtained by melting a finely-powdered intimate mixture of the constituents in an atmosphere of hydrogen and casting in a water-cooled copper mould. The fine-textured alloy is then crushed to a fine powder, which is pressed into rods or shaped bodies, and these are heated at 1750° in hydrogen until completely sintered. A. R. POWELL.

[Nickel] alloys. A.C. SPARK PLUG Co. (B.P. 330,401, 7.6.29. U.S., 1.4.29).—An alloy for use as an electrode in sparking plugs comprises 88–94% Ni, 1.5–2% Mn, 4–10% Cr, 0.4–0.6% Ba. A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 330,289, 5.3.29. U.S., 1.10.28).—A bath for cadmium plating comprises a solution of 1–6 oz. of sulphuric acid, 2–8 oz. of aluminium sulphate crystals, 10–20 oz. of sodium sulphate crystals, 0–8 oz. of ammonium sulphate, 2–4 oz. of cadmium oxide, and 10 g. of glue in 1 gal. of water. A. R. POWELL.

Anti-friction metals and bearings thereof. F. L. BALL, F. MOULTON, and L. W. TOOLS Co., LTD. (B.P. 330,577, 11.3.29).—A mixture of powdered or granulated lead, tin, copper, and antimony together with 3–6% of graphite is compressed into a suitable mould and heated at such a temperature that the metals melt. A. R. POWELL.

Soldering [tungsten, molybdenum, or tantalum]. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 330,403, 10.6.29).—The parts to be joined are sheathed with zirconium and the joint is heated electrically until the zirconium melts. A. R. POWELL.

Blast-furnace tuyères and prevention of burning thereof. E. H. HOLZWORTH (B.P. 331,464, 30.10.29).—See U.S.P. 1,738,901; B., 1930, 244.

Preparation of silicon-steel sheets for electrical purposes. A. F. MURPHY and W. JONES (B.P. 331,511, 2.4.29).—See U.S.P. 1,714,038; B., 1929, 603.

Treatment of nickeliferous material [South African copper-nickel-platinum concentrates]. S. C. SMITH (U.S.P. 1,770,866, 15.7.30. Appl., 18.4.29. U.K., 18.4.28).—See B.P. 311,909; B., 1929, 562.

[Electric] furnace for production of aluminium. E. R. LAUBER (U.S.P. 1,769,298, 1.7.30. Appl., 28.6.26. Ger., 30.7.25).—See B.P. 256,193; B., 1927, 449.

Roasting of [tin] ores and the like. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING, Assrs. to ELECTRO METALLURGICAL ORE REDUCTION, LTD. (U.S.P. 1,769,405, 1.7.30. Appl., 29.3.28. U.K., 6.4.27).—See B.P. 291,568; B., 1928, 574.

Production of aluminothermic mixture and the like. R. W. HYDE (B.P. 319,775, 19.9.29. U.S., 29.9.28).—See U.S.P. 1,751,550; B., 1930, 719.

[Bell-pump] gas-suction apparatus for mineral furnaces. E. SOMOZA (B.P. 317,727, 17.8.29. Spain, 20.8.28).

Apparatus for heating billets etc. H. A. DREFFEIN (B.P. 302,651, 17.11.28. U.S., 19.12.27).

Furnaces particularly adapted for heat-treatment of railway tyres and similar articles. J. BAKER & Co. (ROTHERHAM), 1920, LTD., and S. E. BAKER (B.P. 330,844, 27.9.29).

Removing iron from aluminium salts (B.P. 318,149).—See VII. Luminous enamels (B.P. 325,072).—See VIII. Electrolytic processes (B.P. 329,308).—See XI. Silver from scrap films (B.P. 331,006).—See XXI.

XI.—ELECTROTECHNICS.

Drying of electrolytic pastes in pocket-lamp dry batteries. H. GRIMM (Z. Elektrochem., 1930, 36, 427–428).—In studying the drying of such pastes an impression of the surface is taken by impressing a filter-paper

coated with carbon black thereon, the surface thus coated being re-impressed on a clean filter-paper. Illustrations of results are given. H. I. DOWNES.

Purifying [pyrites] burner gases with electrostatic precipitators. J. J. HEALY, JUN. (Chem. Met. Eng., 1930, 37, 366—368).—Operating and constructional details are given of an installation of both dry and wet Cottrell cleaners used for purifying the gases (10% SO_2) before they pass through a platinum catalyst. The hot or dry precipitators are provided with nickel-steel wire discharge electrodes and steel-mesh collecting electrodes; they operate at between 440° and 340° and collect about 24 cwt. of dust per day from 28 tons of ore. The cold or wet cleaners have lead-covered copper wire and lead-sheet electrodes; they operate at about 30° and deliver an acid drip (d 1.16) containing 9 g. of As_2O_3 per litre from the first stage and 10 g. of H_2SO_4 and 0.05 g. of As_2O_3 per litre from the third and final stage, the arsenious oxide in the cleaned gas being 0.0013 mg./cub. ft., and after passing through a long flue about half that, an amount which is supposed to be at the danger point, though no ill-effects have been noticed. B. M. VENABLES.

See also A., July, 863, **Electrode potentials of silver in cyanide solutions** (FINK and HOGABOOM). 868, **Electrolytic manufacture of ammonium persulphate** (IZGARISCHEV and GRUZDEVA). 870, **Electrodeposition of metals from their liquid ammonia solutions** (TAFT and BARHAM). 882, **Determination of metals by electrodeposition** (TUTUNDŽIĆ). 883, **Thermoregulators** (BEATTIE and JACOBUS; HIBBEN; WESSON; EGERTON). 919, **Preparation of tri- and tetrachloro-*p*-benzoquinones and *s*-trichloroaniline** (ERDÉLYI).

Heat transfer from tube walls. EAGLE and FERGUSON.—See I. **Ageing of mineral oils.** EVERS and SCHMIDT.—See II. **Refractory furnace linings.** KUKLA; SOMMER.—See VIII. **Steel analysis.** TRZEBIA-TOWSKI.—See X. **Polymerised oils.** HOCK and NOTTE-BOHM.—See XII. **Colour of tanning extracts.** DE LA BRUÈRE.—See XV.

PATENTS.

Electrical induction furnaces. N. R. DAVIS, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 331,324, 8.5.29).—In induction furnaces of the type described in B.P. 323,561 (B., 1930, 334), an earthed, hollow, electrically-conducting screen or grid, cooled by circulating fluid and insulated from the inductor coil, is arranged between this coil and the charge. J. S. G. THOMAS.

Electrolytic processes and baths therefor. M. SCHLÖTTER (B.P. 329,308, 14.2.29).—Metals are electrolytically deposited evenly and a satisfactory current-path dispersion is secured by using a plating bath containing acid sulphonates of the metals, *e.g.*, stannous dihydrogen naphthalenetetrasulphonate, or copper hydrogen benzene-*p*-disulphonate. Gelatin and tannin and copper sulphate are preferably also present for copper-plating. C. HOLLINS.

[Electrical] activation of chemical reactions. L. Tocco (B.P. 330,226, 29.1.29. Fr., 7.11.28).—Certain reactions, *e.g.*, the oxidation of sulphur dioxide or

hydrogen sulphide to sulphuric acid, are said to be promoted by the presence of an electric field produced by an A.C. of 25,000—150,000 volts at 25—500 periods.

A. R. POWELL.

Dip for dry-cell cores. BURGESS BATTERY Co., Assees. of J. M. HENDERSON (B.P. 331,467, 31.10.29. U.S., 2.11.28).—Cathodes are dipped in a mixture composed of wood pulp (10—30 pts.) and powdered graphite (100—400 pts.) suspended in water (1000—2500 pts.) containing gelatinised starch (25—200 pts.).

J. S. G. THOMAS.

Manufacture of [porous] electrodes for accumulators. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,080, 14.6.29).—Powdered metal mixed with at least 5% of removable material is sintered, if desired under pressure, and the latter material is removed either partially or wholly. Thus, *e.g.*, a slab containing 70% of nickel powder produced from the carbonyl and 30% of powdered ammonium carbonate is heated for 4 hrs. at 600° , then for 1 hr. at 800° in hydrogen. The resulting plate is impregnated with nickel nitrate.

J. S. G. THOMAS.

[Grids, frames, etc. of] electric accumulators. I. G. FARBENIND. A.-G. (B.P. 311,401, 26.4.29. Ger., 12.5.28).—A hollow body composed of an alloy of lead with an alkali or alkaline-earth metal is completely filled with hard lead worked through an extrusion press. [Stat. ref.] J. S. G. THOMAS.

Treatment of mica, or artificial mica, or substances resembling mica. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,878, 28.6.29).—Single sheets of mica for electrical purposes are covered with mixtures of unhardened resinous condensation products of polyhydric alcohols (*e.g.*, glycols, sorbitol, erythritol), with one or more polybasic organic acids (*e.g.*, phthalic, succinic, or maleic acid) and one or more organic or inorganic vapour-absorbing fillers (such as asbestos, iron oxide, zinc oxide, kieselguhr, talc), after dilution, if desired, with an organic solvent (*e.g.*, acetone). The whole is then hardened by prolonged heating.

S. K. TWEEDY.

Manufacture of thermionic cathodes. SOC. ANON. DES ÉTABL. IND. DE E. C. GRAMMONT ET DE A. GRAMMONT (B.P. 308,649, 25.3.29. Fr., 24.3.28).—Gold-coated or gold wire, after passing through a bath of mercury, is transferred to an electrolytic bath composed, *e.g.*, of a saturated solution of chloride(s) of alkali or alkaline-earth metal(s), the anode being formed of precious metal(s), *e.g.*, platinum or iridium. The wire, thus coated with amalgam of alkali or alkaline-earth metal, is then heated electrically in hydrogen or other gas until the mercury completely evaporates.

J. S. G. THOMAS.

Protection of [iodised starch] pictures [for wireless-transmission purposes] from the effect of light. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 331,154, 9.10.29).—Pictures are covered with or enveloped in protective material consisting of regenerated cellulose soaked in an aqueous solution of *æsculin* or of an alkali salt of naphthionic acid.

J. S. G. THOMAS.

Induction furnace. E. F. RUSS (U.S.P. 1,770,662, 15.7.30. Appl., 24.5.28. Ger., 21.8.26).—See B.P. 305,388; B., 1929, 360.

[Mounting of ribbon heating-units in] electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF R. M. CHERRY (B.P. 307,031, 1.3.29. U.S., 1.3.28).

Processes and casting moulds for manufacturing zinc containers for galvanic elements, batteries, etc. A. GASSAN and P. RUCK (B.P. 331,650, 22.5.29).

Production of thin insulating coatings on wires. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 304,192 and Addn. B.P. 306,122, 8.1.29. Ger., [A] 16.1.28, [B] 16.2.28).

[Tubular] electric incandescence lamps. GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN (B.P. 331,796, 27.11.29. Ger., 30.11.28).

Testing incandescence lamps etc. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF W. L. KUBACK (B.P. 310,339, 23.4.29. U.S., 23.4.28).

Electric-discharge tubes. S. G. S. DICKER. FROM N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 331,318, 6.5.29).

Electron-discharge device. RAYTHEON, INC. (B.P. 317,398, 6.8.29. U.S., 15.8.28).

Electric-discharge lamps. PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H., ASSEES. OF DEUTS. LEUCHTRÖHREN-GES.M.B.H. (B.P. 317,851, 15.8.29. Ger., 25.8.29).

Cathodes for electron-discharge devices. S. LOEWE (B.P. 306,513, 22.2.29. Ger., 22.2.28).

[Exhausting] thermionic valves. F. A. GOULDING and S. F. GAME (B.P. 331,231, 23.7.29).

[Panel system for] electric heating [of buildings etc.]. F. A. L. BILLERY (B.P. 331,237, 25.3.29).

X-Ray tubes. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRICITÄTS GES. (B.P. 315,430, 13.7.29. Ger., 14.7.28).

Oil-immersed X-ray apparatus. VICTOR X-RAY CORP., ASSEES. OF J. B. WANTZ (B.P. 313,568, 13.6.29. U.S., 14.6.28).

Measurement of the rate of flow of fluids by means of electric generators. L. MELLERSH-JACKSON. FROM SIEMENS & HALSKE A.-G. (B.P. 331,040, 8.5.29).

Removing soot from gases (B.P. 306,043). Energy production from brown coal (G.P. 462,388). Cracking oils (U.S.P. 1,751,148).—See II. Electrolysis of fused halogen salts (B.P. 330,791). Removing iron from aluminium salts (B.P. 318,149).—See VII. Silica bodies (B.P. 330,943).—See VIII. Heating metal melts (B.P. 312,063). Nickel alloys (B.P. 330,401). Cadmium plating (B.P. 330,289). Furnace ladles (B.P. 312,064).—See X. Porous sheets (B.P. 331,197 and 331,227).—See XIV. Treatment of tobacco (B.P. 331,026).—See XX.

XII.—FATS; OILS; WAXES.

Spectrographic analysis of the varying fluorescence of olive oil as observed with ultra-violet light. H. MARCELET and H. DEBONO (Compt. rend., 1930, 190, 1552—1553).—The emission and absorption spectra of various North African olive oils, exhibiting very varying fluorescence, have been examined by the method previously described (cf. Marcelet, B., 1930, 674). The emission spectra show a band at 5700—6900 Å. with sometimes a maximum at 6650—6900 Å.; the absorption spectra bands at 4380—4750 and 5640—6900 Å., with a maximum in some cases towards 6900 Å. Slight differences in the spectra seem to be connected with the varying fluorescence, but are not sufficient to modify sensibly the spectra. C. A. SILBERRAD.

Analysis of Philippine lumbang oil. A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1930, 42, 251—257).—A refined, cold-pressed lumbang oil had: d_4^{20} 0.9170, n_D^{20} 1.4740, saponif. value 191.7, iodine value (Wijs, 6 hrs.) 152.7, unsaponifiable matter 0.34%, oxyacids 0.13%, free fatty acids (as oleic) 0.05%, Wesson colorimeter readings 0.1 red and 1.5 yellow. The composition of the oil is given as: essential oil (cf. Santiago and West, B., 1927, 304) 0.3%; unsaponifiable matter 0.3%; glycerides: of saturated acids 2.1%, of oxidised acids 0.1%, of unsaturated acids [α]linolenic 7.7%, α -linoleic 14.3%, β -linoleic 4.5%, γ -linoleic 15%, δ -linoleic 27.9% (cf. *loc. cit.*); and of oleic 40.4%.

E. LEWKOWITSCH.

Shea nuts from Nigeria. ANON. (Bull. Imp. Inst., 1930, 28, 123—131).—Botanical specimens from Nigerian shea trees (*Butyrospermum Parkii*; no distinct morphological varieties were observed) and 14 samples of nuts from the provinces of Kabba and Sokoto were examined, the proportion of kernel (61.3—84.5%, 57.6—75.5% from the respective districts), the oil content (35.5—44.2%, 33.9—50.2%, on moisture-free kernels), and the unsaponifiable matter in the oil (7.7—11.8%, 5.1—11.0%) being determined. From the results it appears that the percentage of unsaponifiable matter in the oil is the greater, the lower are the percentage of kernels and the yield of oil, although it is unconnected with the average weight of the kernels or the shape or colour of the nuts. It would seem, therefore, that the riper are the nuts, the lower is the amount of unsaponifiable matter in the oil. E. LEWKOWITSCH.

Vitamins in [fatty] oils. F. S. GERONA (Ann. Falsif., 1930, 23, 284—290).—The presence of vitamins in oils can be determined by their absorption spectra. Olive oil has a greater vitamin content than cod-liver oil, and contains vitamin-B as well; it loses its activity when heated to 200°. Natural grape-seed oil is also rich in vitamins, but the refined product, owing to heat-treatment, is inactive. B. W. TOWN.

Drying oils. XIII. Changes in linseed oil, lipase, and other constituents of the flax seed as it matures (1929). E. R. TREIS, J. S. LONG, and G. F. BEAL (Ind. Eng. Chem., 1930, 22, 768—771; cf. B., 1930, 109).—Oils from seeds of various degrees of maturity were analysed, the content of the individual

saturated and unsaturated (Kaufmann-Keller thiocyanometric method; cf. B., 1929, 401) acids being determined. The rapid production of oil in the early stages of growth of the seed (some 17 days) is accompanied by a marked decrease in the activity of the enzymes present and by desaturation of the first-formed glycerides. The oils from the 1929 North-west (mature) crop (Minnesota) showed an abnormally low iodine value (170—175) and high oleic acid content (15—20%), probably owing to the unusually hot, dry weather in the latter part of the season.

E. LEWKOWITSCH.

Drying tests on soya-bean oil. E. MARKOWICZ (Farben-Ztg., 1930, 35, 2078—2079).—Experiments on the drying of soya boiled oils made with various driers are described. Resinate driers cause slow drying and tend to produce re-softening of the film; manganese driers are also unsuitable as they yield turbid or deposit-forming oils. By the use of cobalt or lead cobalt linoleates better boiled oils can be produced which dry in 6—8 hrs., forming films which scarcely re-soften and have good adhesion, but abrade more easily than linseed oil films. The drying properties are much improved by bodying the soya-bean oil to a stand oil in the absence of air.

E. LEWKOWITSCH.

Physico-chemical investigations on electrically polymerised oils (Voltol process). (Heat of dissolution of caoutchouc.) L. HOCK and C. L. NOTTEBOHM (Kolloidchem. Beih., 1930, 31, 185—221).—There are analogies between caoutchouc and multimolecular oils and a study of the polymerisation of linseed oil under the influence of the silent electric discharge has been made. The final product of this treatment is a solid gelatinous product which is insoluble in all the usual solvents and can be purified from other oils by washing with light petroleum. This substance contained about 70% C and 11% H, but a mol. wt. determination was not possible; X-ray examination indicated an amorphous structure. The course of the polymerisation was followed by measurements of refractive index, viscosity, iodine value, and mol. wt. in various solvents. The heats of dissolution in benzene and in menthol were also measured.

E. S. HEDGES.

See also A., July, 856, Systems soaps-cresol-water (ANGELESCU and POPESCU). 867—8, Catalytic reactions at high pressures (MORGAN; DONNAN). 891, Linoleic acids and their oxidation by peracids (SMIT). [Oil from] seeds of *Cesalpinia Bonducella* (KATTI; KATTI and PUNTAMBEKAR). Crystalline coloured wax (KUHN and others).

Decolorisation of oils. CHOWDHURY and DAS.—See II.

PATENTS.

Isolation of fatty acids obtained by the oxidation of waxes. J. Y. JOHNSON. From I.G. FARBENIND. A.-G. (B.P. 330,921, 14.3.29).—The oxidation products of waxes are treated with hot concentrated acetic acid, and the insoluble unsaponifiable matter which separates on cooling to not below 15° is removed, the fatty acids being recovered by freezing or by distilling the solution.

E. LEWKOWITSCH.

Refining vegetable and animal fats and oils by removing the free fatty acids therefrom by distil-

lation. METALLGES. A.-G. (B.P. 307,750, 7.3.29. Ger., 12.3.28).—The oil is treated with dilute mineral acid alone, before distilling off the high-boiling fatty acids, which are recovered in a high degree of purity.

E. LEWKOWITSCH.

Manufacture of edible fatty products with a butter-like aroma. N.V. NEDERL. N.V. FRANSCH-HOLLANDSCHE OLIEFABR. NOUVEAUX ETABL. CALVÉ-DELFT (B.P. 301,750, 3.12.28. Holl., 3.12.27).—A minute amount (4 pts. per million) of diacetyl or other diketone is incorporated directly in the fatty product during any stage of the manufacture, or indirectly, by promoting the formation of butylene glycol, acetyl-methylcarbinol, etc. during the souring of milk and oxidising these compounds to the corresponding diketones.

E. LEWKOWITSCH.

Manufacture of oils which are soluble in mineral oils from castor oil. I. G. FARBENIND. A.-G. (B.P. 317,391, 14.3.29. Ger., 15.8.28).—Castor oil, or a mixture thereof with mineral oil, is heated at about 250—320° in the presence of an inert catalyst, e.g., oxides of aluminium, tungsten, titanium, etc., or sulphates, phosphates, silicates, etc., which split off water; the volatile products are preferably removed during the reaction, e.g., by a stream of carbon dioxide.

E. LEWKOWITSCH.

Detergent composition of oils. H. MENZ, W. STEFFEN, and E. JAAKS-MÜNCHENBURG (B.P. 331,034, 30.4.29).—The composition consists of a sulphonated vegetable oil (e.g., castor oil) mixed with turpentine oil, a hydrogenated hydrocarbon, and pine-needle oil.

H. ROYAL-DAWSON.

Preparation of fine granules of soap. COLGATE-PALMOLIVE-PEET Co. (B.P. 314,483, 18.2.29. U.S., 28.6.28).—Fine soap particles from a shredder are dried by falling through a counter-current of hot air (cf. B.P. 314,482; B., 1930, 725), deposition on the shredder being prevented by a fan suitably arranged to reverse the air current locally. At the lower end of the drying chamber the air inlet comprises a series of ports which are adapted to impart a centrifugal motion to the entering hot air, and are kept clear by a rotary cleaning device.

E. LEWKOWITSCH.

Converting liquid soap into a rapidly soluble, handy form. A. WELTER (B.P. 331,268, 26.3.29).—The dry powder produced by atomising a liquid soap (containing soda, silicate, etc., as required) is rendered homogeneous in roller mills and shaped either to threads by extruding presses or to soap-wool, flakes, etc. by rollers in the usual way.

E. LEWKOWITSCH.

Detergent alcoholic soap or cream. G. MAZZETTI (B.P. 317,819, 6.4.29. It., 23.8.28).—A mixture of alcohol with motor spirit or benzene, light petroleum, turpentine, etc., is added to a potash-coconut oil soap containing additional glycerin.

E. LEWKOWITSCH.

Recovery of grease or fat [from wool-scouring liquors]. F. E. BAILEY (U.S.P. 1,770,476, 15.7.30. Appl., 22.8.23. Austral., 19.9.22).—See B.P. 204,326; B., 1925, 325.

Manufacture of margarine. A. K. EPSTEIN (B.P. 331,562, 5.2.29).—See U.S.P. 1,707,800; B., 1929, 482.

Water-soluble substances (B.P. 329,622).—See III. Treatment of non-fibrous materials (B.P. 330,649).—See V. Deodorising etc. of butter fat (B.P. 330,662). Production of plastic substances (B.P. 330,887). Salad cream (B.P. 330,708).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Measurement of colour-tone. W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1930, 35, 1824—1825).—In examining the use of the Zeiss step-photometer for colour-tone determinations in pigments and varnishes, dry mixtures of lead-white and lampblack are made up into a series of grey varnishes and the tones obtained are analysed. Addition of 0.1 g. of lampblack to 100 g. of lead-white, *e.g.*, gives a grey containing 70% black tone, 0.7 g. 90%, 2.1 g. 94.5%, and 7.0 g. 97.5%. The standard tones in railway colour charts used for testing colour-blindness are found to have 1—5% colour content (green, blue, etc.), and this varies according to the source of the pigments employed. The use of the Zeiss photometer for the standardisation of pigments, especially those intended for greys, by manufacturers is recommended. C. HOLLINS.

Yellowing of interior gloss paints and enamels. S. WERTHAN, A. C. ELM, and R. H. WIEN (Ind. Eng. Chem., 1930, 22, 772—776).—A preliminary report. Moisture and a temperature above 0° are necessary for the reactions which lead to the formation of yellow compounds, but yellowing is independent of atmosphere since the same degree of discoloration occurred in ozonised air, in hydrogen, and in nitrogen. Red light produces a greater discoloration than green or blue, and infra-red radiations have the greatest effect of all. Purification of the oil does not eliminate yellowing, which is also independent of the free acid content. The most unsaturated oil shows the greatest discoloration. L. S. THEOBALD.

Lac industry in India. (Miss) D. NORRIS (J. Oil Col. Chem. Assoc., 1930, 13, 195—199).

See also A., July, 883, Protection of ventilating fans (DREIFUSS and STAAB). 913, *Cannabis Indica* resin (CAHN). 920, Nitration of abietic acid (GOLD-BLATT).

Disintegration. CHWALA. Adhesives and adhesion. LEE.—See I. Drying tests on soya-bean oil. MARKOWICZ.—See XII.

PATENTS.

Manufacture of oil paint. M. W. SAUNDERS and J. J. HYNES (B.P. 330,584, 6.3.29).—An oil paint containing heavy-metal soaps as driers (*e.g.*, lead linoleate and/or resinate) is heated to about 100° and mixed with a hot, aqueous, saline solution (*e.g.*, 4 oz. of sodium or magnesium chloride per gal.) which will form an emulsifier by double decomposition with the drier. E. LEWKOWITSCH.

Production of dull-decoration paints ready for painting. A. RÖHRMANN (B.P. 330,673, 25.3.29).—A matrix consisting of 75 pts. of a mixture of waxes, 125 pts. of a copal varnish-wood oil solution (41 pts. of copal, 20 pts. of wood oil, 39 pts. of solvent), 300 pts. of a linseed oil-wood oil solution (similar proportions,

linseed oil replacing the copal), 485 pts. of solvent (*e.g.*, turpentine), 10 pts. of driers, and 5 pts. of glycerin is mixed with pigments at about 100°.

E. LEWKOWITSCH.

[Dehydration of] pigments. E. C. DE STUBNER (B.P. 330,917, 13.12.28. Cf. B.P. 277,949; B., 1929, 365).—Wet pigments, other than those which have been precipitated in aqueous media, are washed with a non-aqueous liquid miscible with water until the liquid has completely replaced the water. For example, ethyl alcohol, acetone or pyridine, and alcohol or acetone are used for pigments for the manufacture of lacquers, printers' inks, and cellulose acetate, respectively. L. A. COLES.

Manufacture of [aluminium] lakes or pigments.

I. G. FARBENIND. A.-G. (B.P. 307,436, 7.3.29. Ger., 7.3.28).—Colour lakes are obtained by boiling the absorption complex of a basic or acid dye on aluminium hydroxide, silicate, or phosphate with the reduction products of complex phosphotungstomolybdic acids obtained by the process described in B.P. 292,253 (B., 1928, 603). A. R. POWELL.

Transfer and marking composition therefor.

KAUMAGRAPH CO. (B.P. 331,135, 5.9.29. U.S., 19.4.29).—The composition comprises a mixture (*e.g.*, "Amberol G 8") of a phenol-formaldehyde condensation product with a more fusible resin, together with a fusible wax (*e.g.*, beeswax) and a blown oil (*e.g.*, blown rapeseed oil). L. A. COLES.

Manufacture of pearl essence. J. PAISSEAU (B.P. 308,969, 26.3.29. Ger., 2.4.28).—Natatory bladders are pickled in an acid medium, *e.g.*, water containing 1—5% of tartaric or formic acid, in the presence of sufficient common or other neutral salt to prevent the decomposition of the conjunctive tissues. A detergent material, *e.g.*, saponin, is added, the temperature is raised to 30—60° or above for a suitable period, after which a proteolytic or lipolytic ferment is added at 45° or less. The unattacked pearl essence crystals are removed by settling, centrifuging, etc. S. S. WOOLF.

Production of lac [lacquer] products. J. SCHEIBER (B.P. 316,538, 13.2.29. Ger., 30.7.28).—Ricinoleic acid or elaidoricinoleic acid is distilled in a vacuum until 70—80% has passed over in the form of octadecadienoic acid. The residual viscous liquid or its glyceryl ester is a quick-drying varnish, and may usefully be combined with glyceryl octadecadienoate to give a product similar in properties to boiled wood oil; for this purpose the residue and distillate may be mixed and esterified together with glycerol. The residue may also be incorporated with cellulose esters. C. HOLLINS.

Manufacture of stoving lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,954, 22.11.28).—Residues from the distillation of oxidised paraffin waxes, especially residues from products of B.P. 322,437 (B., 1930, 155) in which hydroxycarboxylic acids are present and acids up to C₈ have been distilled off, are dispersed or dissolved in organic diluents, with or without other suitable film-forming substances, and applied as stoving lacquers. Preferably the residues are first heated alone or with a polymerising agent, such as chlorine or sulphur. C. HOLLINS.

Manufacture of lacquers, filling or priming compositions, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,910, 14.3.29. Cf. B.P. 322,537; B., 1930, 157).—Coating compositions are claimed comprising nitrocellulose and soluble resinous condensation products of aromatic dibasic acids, *e.g.*, phthalic acid, with polyhydric alcohols containing 4–6 hydroxyl groups, *e.g.*, sorbitol or pentaerythritol, incompletely esterified by organic monobasic acids of natural origin and of non-drying character, *e.g.*, lauric, ricinoleic, or abietic acid, and solvents; softeners, fillers, and colouring materials may be added. S. S. WOOLF.

Manufacture of lacquers, filling or priming compositions, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,909, 14.3.29. Addn. to B.P. 322,466; B., 1930, 157).—Coating compositions are claimed which comprise nitrocellulose and soluble resinous condensation products of aliphatic dibasic acids, *e.g.*, succinic or diglycolic acid, with polyhydric alcohols, *e.g.*, glycerol or glycols, incompletely esterified by high-molecular, aliphatic, monobasic acids of non-drying character, *e.g.*, lauric, ricinoleic, or abietic acid; and solvents; softeners, fillers, and colouring materials may also be added. S. S. WOOLF.

Preparation of coatings. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,897, 16.3.29).—Water-insoluble cellulose derivatives are finely dispersed, mechanically, in water and mixed with aqueous emulsions of assistants (dissolving, softening, or gelatinising agents, or mixtures of these, in amounts which are insufficient for homogenising the mixture). The assistant emulsions are preferably prepared with the use of protective colloids, *e.g.*, gelatin or methylcellulose, or of wetting or emulsifying agents, *e.g.*, alkyl-naphthalene-sulphonic acid salts, resin soaps, etc. Colouring materials and/or rubber latices may be added. The coating material thus formed may be sprayed on to substrata and subjected, if desired, to after-treatment. S. S. WOOLF.

Organic liquid coating compositions [pyroxylin varnishes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,735, 17.5.29).—The varnishes contain at least one organic film-forming substance, *e.g.*, pyroxylin, resin esters, linseed oil, and a solvent containing a ketone or mixture of ketones containing an aliphatic branched-chain connected with the carbonyl group, such as are obtained by the process described in B.P. 310,438 (B., 1929, 635). *E.g.*, the varnish may consist of 50 pts. of pyroxylin, 10 pts. of butyl phthalate, and 30 pts. of castor oil dissolved in 500 pts. of the ketones having b.p. 120–170°, 300 pts. of toluene, and 100 pts. of light petroleum. A. R. POWELL.

Plastic or coating compositions. SELDEN CO., Assees. of A. O. JAEGER (B.P. 312,636, 6.5.29. U.S., 29.5.28).—Phthalide, hydrogenated phthalides, their alkylated, arylated, or alkylidene derivatives are used as plasticisers for synthetic resins of all types. C. HOLLINS.

Preparation of solutions or plastic masses. BRIT. CELANESE, LTD. (B.P. 303,152, 14.12.28. U.S., 29.12.27).—The solid constituent is treated with the

vapour of a solvent (or solute) before mixing with a liquid solvent (or solute), which is the same as, or easily miscible with, that used in the vapour treatment; the process is particularly applicable to the production of solutions etc. of cellulose derivatives. E. LEWKOWITSCH.

Synthetic resin varnishes and synthetic resins. IMPERIAL CHEM. INDUSTRIES, LTD., H. H. MORGAN, and A. A. DRUMMOND (B.P. 329,335, 13.11.28).—Partly esterified or etherified polyhydric alcohols (glycol, glycerol, or erythritol) are condensed with a polybasic acid (phthalic) anhydride in presence of a non-hydroxylated fatty ester of glycerol (preferably a drying oil) and in presence or absence of a solvent. Phthalic anhydride, *e.g.*, is heated at 220–230° with glycerol monobenzoate and wood oil, with α -chlorohydrin and wood oil, or with glycerol monobenzoate, tung oil, and tetrahydronaphthalene. C. HOLLINS.

Production of shaped articles from urea or its derivatives, solid polymerised aldehydes, and fillers. BAKELITE GES.M.B.H. (B.P. 305,211, 28.1.29. Ger., 3.2.28).—The components are mixed with one another in the absence of water or volatile liquids and without heating, and the mixture is hot-pressed with no intermediate treatment. Natural or artificial resins, aldehyde-binding substances, *e.g.*, *p*-toluenesulphonamide, or water-binding substances, *e.g.*, burnt gypsum, dyes, fire-resistant materials, etc., may be incorporated if desired. S. S. WOOLF.

Moulding mixtures [comprising phenol-formaldehyde condensation products]. H. WADE. From BAKELITE CORP. (B.P. 330,968, 20.3.29).—A phenolic resin is kneaded, *e.g.*, at 100°, with a mixture of a filler and a high-boiling aldehyde (*e.g.*, furfuraldehyde), with the addition, when a non-reactive resin is used, of a methylene compound (*e.g.*, hexamethylenetetramine). L. A. COLES.

Manufacture of (A) shellac-like product and (B) artificial shellac. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,770,875–6, 15.7.30. Appl., [A] 20.12.26, [B] 2.1.28. U.K., [A, B] 6.10.27).—See B.P. 309,384; B., 1929, 651.

Compositions for use in marking lines in roads and tennis courts. W. T. BELL and J. F. BENNETT (B.P. 331,207, 23.3.29).

[Flotation method of] surfacing articles with films comprising pigments, varnishes, glazes, enamels, lacquers, etc. G. M. BROWN and H. T. WYSE (B.P. 331,201, 19.2. and 21.5.29).

Azo pigments (B.P. 306,415 and 329,960).—See IV. **Soluble acylcelluloses** (B.P. 305,674). **Artificial silk** (B.P. 305,468). **Treatment of non-fibrous material** (B.P. 330,649).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Structural changes during the processing of rubber. E. A. GRENQUIST (Ind. Eng. Chem., 1930, 22, 759–765).—Plasticity values of raw rubber based on flow and recovery, *e.g.*, in a Williams plastometer, are probably regulated mainly by the state of aggregation of the rubber and the degree of breakdown and fusion

of the rubber globules. Cold milling causes disaggregation of the hydrocarbon and breakdown and fusion of the globules, the resulting softness being permanent. Heating in air causes partial breakdown and fusion of the globules together with thermal disaggregation and oxidation. At temperatures up to 164° the presence of oxygen appears to be necessary for breakdown and fusion of the globules; heating in steam or carbon dioxide leaves the globular structure almost unaltered, and, as the plastic changes are more or less reversible so long as the globular structure remains, the effect of such heating is only temporary, re-aggregation occurring gradually. In the absence of air, rubber exhibits a maximum degree of disaggregation at any temperature. Ultra-violet light concurrently affects the plastic and also the elastic properties of the rubber, both plastic flow and recovery being increased. There exists a correlation between elasticity and double refraction of rubber, the anisotropy at 300% elongation falling parallel to the recovery values after deformation in the plastometer. No agency in this investigation caused such far-reaching and profound destruction of the elastic properties of rubber as prolonged mastication.

D. F. TWISS.

Vulcanisation of rubber. I. Thermochemistry of vulcanisation of rubber. II. Vulcanisation of rubber with nitro-compounds. III. Kinetics of vulcanisation of rubber with sulphur and selenium.

J. T. BLAKE. IV. Theory of vulcanisation of rubber. C. R. BOGGS and J. T. BLAKE (Ind. Eng. Chem., 1930, 22, 737—740, 740—744, 744—747, 748—755).—I. By careful measurement of the heat of combustion of a rubber-sulphur mixture before and after vulcanisation and taking precautions to avoid premature atmospheric oxidation of the vulcanised material, it is found that combination of rubber with sulphur up to about 6% occurs without any appreciable energy change in the system. From this point onwards, however, the combination with sulphur is accompanied by liberation of heat, complete formation of vulcanite being attended by a maximum liberation of 442 g.-cal. per g. of rubber. With the additional presence of diphenylguanidine as vulcanisation accelerator, together with zinc oxide, heat evolution is observable even at approx. 4% of combined sulphur. The formation of soft vulcanised rubber and of vulcanite are distinct processes, only the latter involving thermochemical change. Vulcanisation with *m*-dinitrobenzene or selenium does not involve any heat evolution.

II. Vulcanisation of rubber with di- or tri-nitrobenzene involves a definite chemical change, a change in density occurring during the process and a proportion of the vulcanising agent becoming insoluble in acetone. The reaction is one of the first order, and probably comprises a rapid primary combination of rubber and nitro-compound to form a loose molecular compound which undergoes subsequent slower conversion into the vulcanised product. The equivalent weight of rubber per nitro-group in the final product from either di- or tri-nitrobenzene is approx. 1315.

III. Vulcanisation of rubber by selenium is a reaction of the first order. The maximum coefficient of vulcanisation obtainable is about 5.1%, corresponding

with a sulphur coefficient of 2.04%, and with an at. wt. of selenium per 1560 pts. of rubber. No vulcanite product was obtainable from rubber and selenium even on prolonged heating or with the aid of an organic accelerator. The second stage of the combination between rubber and sulphur, which gives rise to vulcanite, is a reaction of the second order and, in any one molecule, occurs after the reaction yielding soft vulcanised rubber. Vulcanisation accelerators expedite the formation of the soft vulcanised product, but have no appreciable influence on the subsequent reaction yielding vulcanite.]

IV. All the phenomena observed in vulcanisation are explained by the theory that vulcanisation is an additive chemical reaction capable of yielding two stable products, viz., soft vulcanised rubber and hard vulcanite. The formation of the two products occurs successively in any one molecule during exhaustive vulcanisation by sulphur. Certain vulcanising agents effect only the first stage of the process, and the influence of accelerators is substantially limited to this stage. There are two types of chemical unsaturation in the rubber molecule; after satisfying the valencies responsible for the production of soft vulcanised rubber, further addition of sulphur at first gives rise to an unstable type of material which may be described as "intermediate vulcanite"; the deterioration of ordinary soft vulcanised rubber with age is due to the presence in it of a proportion of molecules of the "intermediate vulcanite" type. There is a definite relation between the physical properties and the proportion of combined sulphur only when "intermediate vulcanite" is not present extensively. The electrical properties of vulcanised rubber are in accord with a view that in the formation of vulcanite the sulphur adds progressively from one end of the rubber molecule to the other, thus giving rise to dipoles which have a maximum effect when the reaction is about half completed. In the "reclaiming" of rubber, the vulcanised rubber molecule undergoes fission into two portions, viz., an insoluble fraction containing practically all the combined sulphur, and a soluble, sulphur-free fraction. The physical properties of ordinary soft vulcanised rubber are determined by the relative proportions of pure soft rubber and "intermediate vulcanite" present, the best product being obtained when the latter is at a minimum. Vulcanisation accelerators, by affecting mainly the reaction yielding the soft rubber constituent, lead to relative reduction of the proportion of "intermediate vulcanite," and so result in a product of superior physical properties, but with a lower total proportion of combined sulphur. For the production of pure soft rubber, the proportion of combined sulphur necessary would be approx. 0.5%, which corresponds with a composition $(C_5H_8)_{200}S_2$.

D. F. TWISS.

Determination of copper in fabrics. KLICKOW and SIEBNER.—See V. **Heat of dissolution of caoutchouc.** HOCK and NOTTEBOHM.—See XII.

PATENTS.

Manufacture of rubber. SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 330,520, 6.3.29. Addn. to B.P. 284,608; B., 1929, 612).—The addition of an aromatic disubstituted guanidine, e.g., *s*-diphenylguanidine,

renders latex capable of thickening and of coagulation in regions where localised heating, *e.g.*, to 70–95°, is applied. Small quantities of other substances such as ammonium salts, or salts of bi- or ter-valent metals may expedite the thickening and coagulating process. The rubber layer or article produced, even in the presence of sulphur, is unvulcanised, but may be vulcanised subsequently.

D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., C. HAYES, E. W. MADGE, and F. H. LANE (B.P. 330,536, 8.3.29).—The addition of excess of formaldehyde, to natural or artificial latex of rubber or similar materials, in excess of that necessary to neutralise the free alkalinity such as ammonia, leads to the formation of a continuous coagulum or gel in a definite and controllable time. The higher the ammonia content of the latex the greater is the necessary excess of formaldehyde to produce a continuous coagulum in a given time at room temperature. For treatment of a non-compounded latex containing more than 0.05% of ammonia and concentrated by a centrifuging process, it is necessary also to introduce a small proportion of a sensitising agent such as ammonium sulphate, zinc sulphate, or an ammonium salt of an organic acid, *e.g.*, oxalic, tartaric, or citric acid. Emulsions or dispersions so treated are applied to manufacturing purposes by such processes as spreading, dipping, spraying, or moulding.

D. F. TWISS.

Manufacture of rubber-like products. I. G. FARBENIND. A.-G. (B.P. 307,937—8, 15.3.29. Ger., [A], 16.3.28, [B] 17.3.28).—(A) Rubber-like products are obtained by polymerisation of diolefines in the form of an emulsion which has previously been forced through a homogeniser. (B) An aqueous solution of a material promoting emulsification, *e.g.*, sodium oleate, and a solution in a diolefine of an emulsion-promoting material, *e.g.*, magnesium oleate, are emulsified together. Emulsification is thereby facilitated and the yield of rubber-like product improved.

D. F. TWISS.

Polymerisation of hydrocarbons of the butadiene series. I. G. FARBENIND. A.-G. (B.P. 315,356, 1.7.29. Ger., 12.7.28).—Polymerisation of butadiene hydrocarbons with the aid of sodium hydride is slower than with metallic sodium, which sometimes induces polymerisation with almost explosive violence, and the product is more homogeneous. The process is conveniently performed in the presence of an indifferent solvent, such as benzene, with agitation.

D. F. TWISS.

Improving synthetic rubber-like substances. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,922, 16.3.29).—Polymerisation of butadiene, its analogues or homologues sometimes leads to rubber-like products which, however, are insoluble in benzene and do not readily become plastic when milled. These undesirable features can be removed by treating with an organic nitro-compound, *e.g.*, mono-, di-, or tri-nitrobenzene, advantageously in the presence of a solvent or diluent, such as benzene and at an elevated temperature, *e.g.*, 140–150°.

D. F. TWISS.

Manufacture of rubber solutions. F. KEMPTER

(B.P. 330,953, 22.2.29).—The duration of the process of preparing rubber solution is shortened by stirring the rubber with a solvent in a trough having an irregularly surfaced wall and one or more stirrers, desirably also provided with irregularities.

D. F. TWISS.

Vulcanisation of rubber. RUBBER SERVICE LABS. Co., Assees. of W. A. MOORE (B.P. 330,981, 22.3.29. U.S., 12.11.28).—Rubber stock containing all the desired compounding ingredients with the exception of the vulcanisation accelerator is vulcanised by immersion in a hot aqueous dispersion (*e.g.*, at 95–100°) of a water-insoluble ultra-accelerator, such as the carbon disulphide derivative of the reaction product of piperidine and formaldehyde, the dispersion being stabilised by the presence of an alkali salt or "triethanolamine" salt of a fatty acid, gum arabic, starch, or saponin.

D. F. TWISS.

Manufacture of resistant porous sheets. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,197 and 331,227, 24.1.29).—(A) Chemically-resistant porous sheets suitable for diaphragms in electrolytic cells are produced by incorporating a non-distillable polymerisation product of butadiene, or a mixture thereof with india-rubber, with a porous substance such as coarse kieselguhr; compounding ingredients of the customary types, including fillers, vulcanising agents, and anti-oxidants, may also be added, and the sheets may be vulcanised. If desired, the sheets may be reinforced, *e.g.*, with porous fabrics or metal gauze. (B) Instead of using a porous substance as in (A), a substance capable of forming pores by dissociation into gases or vapours, *e.g.*, ammonium carbonate, or by dissolution in an inert solvent, *e.g.*, common salt, is incorporated in the mixture, the treatment otherwise being as described.

D. F. TWISS.

Manufacture of articles from aqueous dispersions containing rubber, gutta-percha, balata, and similar resins. DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 330,970, 21.3.29).—In the manufacture of articles from aqueous dispersions of rubber and similar materials by such processes as dipping, spreading, or spraying, removal of the product from the mould or former is facilitated by providing the surface of the latter with a film of a dehydrating and setting liquid (*cf.* B.P. 303,544; B., 1929, 220), prior to introduction into or contacting with the aqueous dispersions. The process of dipping in the dispersions may be repeated, if desired, with alternate dipping into the dehydrating and setting fluid. Alternatively, or finally, the deposit or deposits may be treated to develop a wrinkled or roughened surface, or may be simply dried and vulcanised.

D. F. TWISS.

Electrodeposition of rubber. W. A. WILLIAMS (U.S.P. 1,769,659, 1.7.30. Appl., 12.5.27. U.K., 28.5.27).—See B.P. 293,095; B., 1928, 649.

Thickening and treatment of latex. S. D. SUTTON, Assr. to VEEDIP, LTD. (U.S.P. 1,770,092, 8.7.30. Appl., 27.3.28. U.K., 2.1.28).—See B.P. 312,947; B., 1929, 652.

Mercaptobenzthiazoles (B.P. 306,492).—See III. Coated paper (U.S.P. 1,746,888).—See V.

XV.—LEATHER; GLUE.

Influence of neutral salts on [raw] hide. M. BERGMANN (J. Soc. Leather Trades' Chem., 1930, 14, 307—312).—The volume of water forced through raw skin in unit time under a pressure of 1 atm. was determined and is termed the "primary water permeability." Salt solutions of different concentrations (0.01—0.4*N*) were similarly forced through the same pieces of skin to determine the "salt permeability," after which water was again forced through and the "secondary water permeability" determined. The salt and secondary water permeabilities expressed as percentages of the primary water permeability are found to depend on the salt concentration used. The salt permeability was less than the primary water permeability, due to the shrinkage of the pores by the action of the salt. Except for 0.01*N*-sodium chloride, the secondary water permeabilities were less than the primary. Similar tests with other chlorides did not show any gradation in accordance with the Hofmeister series. Tests with sodium salts of different anions showed diminished permeabilities which did not recover on elimination of the salt from the pelt. The original permeability of pelt which had been treated with 0.00625*N*-solutions was restored by eliminating the salt. It was found that the permeability of salt solutions decreased much more when the salt was forced through from the grain than from the flesh. The reverse effect was obtained for potassium chloride. This effect was not due to the grain layer, since it was repeated on pelt from which the grain layer had been removed.

D. WOODROFFE.

Qualitative analysis of tannins. L. POLLAK (J. Soc. Leather Trades' Chem., 1930, 14, 299—307).—Solutions of tanning extracts are prepared in ammonia and ammonium sulphhydrate of definite concentrations and examined spectroscopically with the instrument arranged vertically over a 50-c.p. Philips lamp. A cell containing the tannin solution is fixed between the instrument and the lamp and wave-length measurements are taken at every 5 mm. in depth until total extinction is obtained. The wave-length-depth of liquid curves obtained are characteristic for the different vegetable tanning materials. Magnesium sulphate must be removed from extracts before treating them with ammonia, but they cannot be examined in ammonium sulphhydrate solution. It has been shown that a mixture of a pyrocatechol tannin and 25% of sulphited quebracho extract or 75% of sulphite-cellulose extract fails to give a definite reaction in the Stiasny formaldehyde-hydrochloric acid test. By adding 1 g. of urea, however, a definite reaction was obtained for pyrocatechol tannins even in the presence of 10% of sulphited quebracho extract or 90% of sulphite-cellulose extract. A high percentage of precipitate was obtained when this modified formaldehyde-hydrochloric acid test was applied to Neradol D, but not with other synthetic tannins. D. WOODROFFE.

Measurement of colour of tanning extracts. II. Photocolorimeter method. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 315—316; cf. B., 1929, 140).—In using the Toussaint photoelectric colorimeter, not only the voltage supplied to the lamp but

that of the accumulators connected to the photoelectric cell must be kept constant. Electrical leaks must be avoided. Photocolorimetric measurements of different tannin extracts are recorded. The error caused by calculating tintometer measurements of tannin solutions of analytical concentration to 0.5% of tannin has been confirmed.

D. WOODROFFE.

Effect of the particle size of kaolin and P.H.A.S. on the filtration of tannin solutions by the contact method. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 313—314).—The insoluble matter and soluble tans were determined in a number of different extracts, using kaolins and "P.H.A.S." of different particle size, respectively. No variation in the results was observed, thus disagreeing with Jamet and Girard's results (B., 1928, 721).

D. WOODROFFE.

Water-resisting properties of glue and casein glue. I, II. T. ARAKI and T. MATSUNO (J. Soc. Chem. Ind., Japan, 1929, 32, 183—184 B, 184 B).—I. An automatic recording device has been employed for determining the time after which a glued joint yields when immersed in water at constant temperature (about 20°) and subjected to a constant load. In general, glues of good quality are more resistant to the action of water than are commercial casein glues, although the results obtained for the former group vary more widely among themselves; liquid glues are considerably inferior as regards their power of resisting the action of water.

II. The resistance of glue to water is considerably increased by treatment with a mixture of paraldehyde and oxalic acid, or if the material to be joined is first treated with formalin; casein glues are improved in like manner by admixture with a small quantity of a solution of a copper salt.

H. F. GILLBE.

Diffusion of oakwood and chestnut extracts into a gelatin jelly. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 296—298).—Test-tubes partially filled with a 2% gelatin jelly were covered with the solution of the extract and kept at 15°. The chestnut extracts penetrated further than did the oakwood, and showed a marbled effect, whilst the oakwood diffused with a brownish appearance. Diffusion tests made on solutions at different p_H values showed that the marbled effect was produced with oakwood extracts at low p_H values, and was no longer shown by chestnut extracts at higher p_H values.

D. WOODROFFE.

Wetting etc. agents (B.P. 308,824). Water-soluble substances (B.P. 329,622).—See III. Azo dyes for chrome leather (B.P. 313,927).—See IV. Solutions of animal proteins (B.P. 306,464).—See V.

XVI.—AGRICULTURE.

Properties of organic hardpan soils with special reference to their formation. L. A. RICHARDSON (Soil Sci., 1930, 29, 481—488).—Less clay and silt occurs in hardpan soils than in non-hardpan soils. The reactions were almost identical. In the hardpan horizon there is a definite accumulation of aluminium, but not of calcium or iron. The C/N ratio in non-hardpan soils decreased from the surface downward, but was almost constant in the hardpan layer. In

Florida the formation of an organic hardpan is favoured by a loose sandy soil, low in silt and clay and free of electrolytes, and with a water-table near the surface during part of the year. A. G. POLLARD.

Podsolis and moor soils of Russia and the Baltic states. H. VON RATHLEF (*Z. Pflanz. Düng.*, 1930, 17A, 201—226).—The formation, flora, and chemical and physical properties of certain Russian soils are described. A. G. POLLARD.

Correlations between the specific conductivities of soil extracts, nitric nitrogen, and soluble calcium. C. H. WRIGHT (8th Ann. Bull. Agric. Dept., Nigeria, 1929, 104—109).—Periodical sampling and analysis of cropped soils show that the calcium and nitrate contents of 1:5 soil-water extracts are closely correlated with their specific conductivities. Increased conductivity is evidence of biological activity in the soil. A. G. POLLARD.

X-Ray and microscopical examinations of soil colloids. S. B. HENDRICKS and W. H. FRY (*Soil Sci.*, 1930, 29, 457—479).—The finer fractions of soils contain crystalline materials. Samples give characteristic powder diffraction patterns which can be identified as arising from one of the clay minerals. Montmorillonite-beidellite, Ordovician bentonite, and halloysite are common constituents of soil colloids. Bauxite was also found. The finer fractions from specific soil types in different localities give the same type of diffraction pattern. A. G. POLLARD.

Can the laboratory examination of a soil give sufficiently definite information as to its lime requirement? A. GEHRING (*Z. Pflanz. Düng.*, 1930, 9B, 241—245).—Laboratory determinations of the lime requirement of soils give satisfactory information for practical purposes and, moreover, save much time. Mitscherlich's plea for field trials is criticised. A. G. POLLARD.

Equilibrium between soil and electrolytes and its influence on some lime requirement methods. N. A. CLARK and E. R. COLLINS (*Soil Sci.*, 1930, 29, 417—427).—Comparative electrometric titrations were made of soil suspensions in water and in calcium chloride solution with lime water and in sodium chloride solutions with sodium hydroxide. Equilibrium is attained after varying periods with different soils, but little change occurs after 90 hrs. In general, equilibrium is reached more rapidly in the salt solutions. The lime requirement measured after 90 hrs. is much higher in salt suspensions (2—5 times) than in aqueous suspensions. The bearing of these results on Christensen and Jensen's measurements (lime requirement = $3 \times$ direct lime titration; cf. B., 1927, 309) is discussed. The suggested instantaneous neutralisation occurring in lime titrations (cf. Saint, B., 1927, 309) is not confirmed. A. G. POLLARD.

Chemistry of mineral soil acidity. II. Chemistry of exchange acidity in soils. III. M. TRÉNEL and J. WUNSCHIK (*Z. Pflanz. Düng.*, 1930, 17A, 257—296, 296—305; cf. B., 1930, 254).—II. Permutit was treated with a number of acids of varying concentration and the extent of the removal of bases and of the decomposition of the permutit was examined. With the progressive

removal of bases the permutit is gradually decomposed into its constituents. Probably no "permutit acid" exists. When bases are partially removed, permutit behaves as a mixed gel of the hydrates of the sesquioxides and of silica. Silicic acid remains after complete removal of the bases. The action of acids on permutit depends on the nature of the acid, its concentration, and the temperature; 0.1*N*-inorganic acids completely decompose permutit. With lower concentrations decomposition of the permutit begins when about half of the bases have been removed. Solutions of aluminium chloride and acetic acid remove bases from permutit to a similar extent to inorganic acids of corresponding concentrations, but differ in having much weaker decomposing effects. Oxalic acid is distinguished by its ability to remove large amounts of silica from permutit. Water containing carbon dioxide removes bases from permutit, but cannot decompose it. During electrodialysis permutit is similarly decomposed and an acid-reacting silicic acid sol appears in the middle chamber. Exchange acidity depends on the degree of decomposition of the permutit, on the dryness of the residual substance, and on the temperature and concentration of the neutral salt solution. It is apparent in *N*-potassium chloride at room temperature when about half of the bases have been removed. Completely decomposed permutit (essentially silica) shows small exchange acidity. At higher temperatures (85°) the acidity is increased and the silica and aluminium hydroxide are peptised. Drying the gel mixture at 150° after removal of bases largely reduces its reactivity. Methyl-red indicates smaller acidity values than phenolphthalein, either because two dissociating substances are present in permutit, or because exchange acidity is not a single-factor phenomenon. Probably peptised silica is titrated when phenolphthalein is used. The amount of iron and aluminium oxides in potassium chloride extracts of permutit is practically the same as that in acid extracts. Potassium chloride appears to peptise the gel. The term "exchange" acidity is inappropriate since the phenomenon is not a direct exchange either of hydrogen or of aluminium ions. Exhausted permutit residues have no absorbed acids to which "exchange" acidity might be attributed. Examination of soil gave results similar to the above. The acidity of potassium chloride extracts is always associated with the presence of silicic acid and sesquioxides.

III. The observed acidity produced in potassium chloride solutions by silica gel depends on the peptisation of the gel which does not exhibit any "neutral salt decomposition." Alumina and ferric oxide gels, after purification by electrodialysis, produce an alkaline reaction in potassium or ammonium chloride solutions. This alkalinity decreases with rising temperature, decreases in the presence of silica gel, and in the latter case is uninfluenced by rise of temperature. The mixed gel of the "silicates" of iron and aluminium produced by precipitation with water-glass exhibits towards potassium chloride solutions exchange acidity depending to some extent on the concentration of the salt solution and the temperature. The gel produced by the removal of bases from sodium-permutit exhibits similar properties. In both cases potassium chloride

causes dissolution of silica and sesquioxides, but there is no equivalence between the titratable acidity and the amount of alumina in solution. The increased peptisation of silica produced by warming causes higher "phenolphthalein" acidity. Exchange acidity represents equilibrium points of the reaction $\text{Al}(\text{OH})_3 + 3\text{KCl} \rightleftharpoons \text{AlCl}_3 + 3\text{KOH}$. "Mineral" acidity in soils is explained on similar lines.

A. G. POLLARD.

To what extent can ammonium salts cause acidity in soils, by the preferential absorption of ammonium cations compared with anions? G. HAGER and W. STOLLENWERK (*Z. Pflanz. Düng.*, 1930, 17A, 129—165).—The physiological acidity of ammonium salts is discussed in relation to ion hydration and the permeability of root-cell membranes. Examination of plant growth in sand cultures shows that the physiological acidity of the ammonium salts increases in the order of the Hofmeister series: nitrate, chloride, sulphate, phosphate. With the sulphate and chloride the majority of the ammonia enters the plant as free ammonia. From the nitrate in faintly acid media the NH_4 ion is absorbed to a greater extent than the NO_3 ion. In very acid media the proportion of NO_3 absorbed is increased. The indirect acidity of the ammonium salts varies in the same order as above. Soil acidity results not only from the actual nitrification of the ammonium salts, but also from the preferential absorption of non-hydrated NH_4OH molecules as compared with that of the anions associated with them.

A. G. POLLARD.

Effect and velocity of action of calcium carbonate in the field on the indexes of acidity in soils. S. GOY [with P. MÜLLER and O. ROOS] (*Z. Pflanz. Düng.*, 1930, 17A, 165—176; cf. B., 1930, 254).—The action of marl and of chalk on soils differs widely with the soil type. The rate of reaction of chalk with a particular soil differs from that of quicklime. Chalk does not completely neutralise soil acidity, even when applied in large excess, presumably because the residual carbon dioxide gives indications of weak hydrolytic acidity. An increased calcium content in the soil solution after liming is of great significance. A general standard of liming, based only on laboratory determinations, is not recommended. Correct applications of lime can only be determined with reference to soil conditions. The authors' system of soil classification affords a satisfactory basis of consideration for this purpose.

A. G. POLLARD.

Effect of different fertilisers on the reaction of strongly acid soils. W. SCHMIDT (*Z. Pflanz. Düng.*, 1930, 9B, 272—275).—Field trials are recorded, which serve to emphasise the importance of considerations of soil reaction and optimum condition of growth for individual crops, in relation to fertiliser practice.

A. G. POLLARD.

Fixation of phosphoric acid in soils. T. GAARDER, (Medd. Vestlandets Forstl. Forsøksstat., No. 14, 1930, 140 pp.).—The solubility of the phosphates of iron, aluminium, calcium, and magnesium in dilute solutions of varying reaction and base content was examined. For each base there is a definite p_{H} range within which the phosphates are relatively insoluble. The variable

effects of soil treatments on the solubility of phosphates are attributed to differences in the quantity and nature of the bases present, either in the adsorbed or soluble condition. Within the reaction range of normal soils the phosphates of iron and aluminium are of prime importance in controlling phosphate solubility. The two maxima observed in p_{H} -growth curves of plants (Arrhenius) may be explained by variations in the solubility of phosphates with soil reaction. In soils rich in "active" sesquioxides, within the entire range p_{H} 4.0—7.5, the phosphate is largely insoluble and liming does not always effect an improvement. In humid and semi-humid soils containing humus the solubility of the phosphates depends mainly on the relative proportions of "active" sesquioxides and humus present, the former tending to decrease and the latter to increase their solubility. From such soils having only moderate sesquioxide contents and p_{H} 4.5—6.5 organically combined phosphorus may be leached sufficiently rapidly to bring about a phosphate deficiency. Where lime did not increase the solubility of phosphates in soils rich in sesquioxides, treatment with sodium silicate proved effective, presumably by the precipitation or "deactivation" of the sesquioxides.

A. G. POLLARD.

Determination of plant-assimilable nutrients in soils. M. VON WRANGELL (*Landw. Jahrb.*, 1930, 71, 149—169).—To determine the assimilable phosphate content, 1 g. of soil is shaken with 100 c.c. of water for 5 hrs. and the suspension is centrifuged. The residue is similarly treated with a further 100 c.c. of water. The total assimilable phosphate is calculated as $a^2/(a-b)$, where a and b are the phosphate contents of the first and second extracts, respectively. Results agree well both with Neubauer values and field trials. The method is suitable for determining the relative availability of phosphatic fertilisers. A similar process is used for determining assimilable potash in soils, but the exact significance of the results is less clearly defined. In water-culture experiments the use of very dilute solutions corresponding in concentration to soil solutions is preferable to the common introduction of relatively concentrated solutions.

A. G. POLLARD.

Home management [of soils] in agriculture. H. KAPPEN (*Z. Pflanz. Düng.*, 1930, 9B, 245—249).—A criticism of Mitscherlich's advice to farmers to carry out their own examination of lime and fertiliser requirements.

A. G. POLLARD.

Effect on crops and acidity condition of soils of amounts of lime calculated from the exchange acidity and from titration curves. L. FRESSENIUS (*Z. Pflanz. Düng.*, 1930, 17A, 176—193).—Applications of lime calculated by Daikuhara's method or from titration curves were insufficient to prevent injury to barley in acid soils. The grain : straw ratio increased with the amount of lime used. Corresponding trials with oats gave similar relative crop yields and grain : straw ratios. Under acid conditions oats utilised existing nutrients more easily than did barley. A particular degree of acidity affected plant growth to a greater extent on sandy soils than on clays. Lime requirements calculated by the two methods examined showed no proportionality for soils of varying types. Plant injury due to excessive

liming occurred only on light sandy soils, and did not persist for a second season. In comparative tests of ammonium sulphate and nitrate on variously limed soils, crop yields with the nitrate were consistently higher than with the sulphate, the differences decreasing with increasing amounts of lime used. Differences in the effects of the two fertilisers were greater in sandy than in clay soils. These crop yields were paralleled by differences in p_H values of the soils, except in light soils treated with ammonium sulphate, where, in some cases, the fertilising effect predominated over the ill effects of increased acidity. A. G. POLLARD.

Yield law and *Aspergillus niger*. R. MEYER (Z. Pflanz. Düng., 1930, 17A, 199—201).—A discussion of some aspects of the yield-law theory based on observations with *A. niger*. A. G. POLLARD.

Nature and course of the nutrient intake of various vegetables. T. REMY and F. WEISKE (Landw. Jahrb., 1930, 71, 315—331).—Cultural trials are recorded designed to show the rate of intake of nutrients by a number of vegetables. The significance of this and of the length of the growing period in the consideration of appropriate fertiliser treatment is discussed. A. G. POLLARD.

Fixation of the nitrogen of liquid manure by lignin and humus-lignite. F. HONCAMP and H. WIESSMANN (Z. Pflanz. Düng., 1930, 17A, 194—199).—Liquid manure absorbed by lignin is an effective nitrogenous fertiliser. Lignin alone has no effect on plant growth, and the efficiency of liquid manure use alone was small. Used in a similar manner, a humus-lignite proved slightly better than lignin. A. G. POLLARD.

Action and value of hot-fermented stall manure. F. LÖHNIS (Z. Pflanz. Düng., 1930, 9B, 268—272).—A reply to recent criticisms (Gerlach, B., 1930, 73; Ehrenberg, *ibid.*, 209). A. G. POLLARD.

Boron as an essential element for healthy growth of citrus. A. R. C. HAAS (Bot. Gaz., 1930, 84, 410—413).—Citrus plants grown in culture solutions from which boron was carefully excluded gave every indication of malnutrition. Addition of borax produced normal healthy growth within a week. Plants appear to obtain sufficient boron for normal needs from the glaze of earthenware culture vessels. A. G. POLLARD.

Control of ragwort and other weeds by spraying. J. W. DEEM (N. Zealand J. Agric., 1930, 40, 291—294).—Sodium and calcium chlorates killed ragwort in full flower and also other weeds. Spraying with 2½—5% solutions is recommended, and quantities ranging from 2 lb. per acre (for scattered plants) up to 22 lb. per acre were used. Little damage was done to grass by 5% chlorate solutions. Dry sodium chlorate was equally successful if applied directly to the weeds, but such applications are more costly. A. G. POLLARD.

Insecticidal action of some derivatives of pyridine and pyrrolidine and of some aliphatic amines. C. H. RICHARDSON and H. H. SHEPARD (J. Agric. Res., 1930, 40, 1007—1015).—Some 33 nitrogenous organic

compounds, mostly related structurally to nicotine, have been tested as contact insecticides against *Aphis rumicis*, L., colonised on nasturtium plants, with the object of finding a product which could be synthesised to replace nicotine. Metanicoline and nicotyrine were the only substances tested which showed a toxicity of the same order as that of nicotine. Benzylpyridine appears to show promise of useful application where its oily properties are advantageous. E. HOLMES.

Test to determine toxicity of pyrethrum vapours to honeybees. J. M. GINSBURG (J. Agric. Res., 1930, 40, 1053—1057).—The essential oils of pyrethrum either exerted no toxic effect on bees or were not sufficiently volatile to produce a lethal concentration in the present experiments. It appears that the toxicity of ground pyrethrum flowers is primarily due to the non-volatile substances, pyrethrins I and II. E. HOLMES.

See also A., July, 872, **Utilisation of potassium phosphates** (ASKENASY and NESSLER). 881, **Determination of calcium in the presence of iron and aluminium** (WILEY). 964, **Soil reaction and growth** (REIFENBERG and FRANKENTHAL). 965, **Rôle of magnesium in the ageing of plants** (LUTMAN and WALBRIDGE). Selection characters as correlated with percentage of sucrose, weight, and sucrose content of sugar beet (PACK). 967, **Action of ultra-violet rays on germination of seeds and growth of plants** (MEZZADROLI and VARETON).

Iodine content of waste water from sewage. KÖHLER.—See XXIII.

PATENTS.

Manufacture of organic mercury compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 329,987, 26.2. and 22.7.29).—Organic mercuri-hydroxides are more reactive than the mercuri-halides and may be condensed with phenols, thiophenols, or mercaptans to give compounds, Ar·Hg·O(or S)·R, useful for seed disinfection. Phenylmercuri-hydroxide [hydroxymercuri-benzene], m.p. 216°, reacts in hot water or alcohol with salicylic acid (product, m.p. 159°), α -hydroxynaphthoic acid (product, m.p. 171°), *o*-chlorophenol (product, m.p. 104°), *m*-hydroxybenzaldehyde (product, m.p. 159°), thiosalicylic acid [product, m.p. 226° (decomp.)], thio-*p*-cresol (product, m.p. 107°), thiophenol (product, m.p. 101·5°), thioglycolic acid [product, m.p. 114° (decomp.)], and 2-thiolbenzthiazole (product, m.p. 147°); ethylmercuri-hydroxide [hydroxymercuriethane] with thio-*p*-cresol (product, m.p. 162°), and 2-thiolbenzthiazole (product, m.p. 86·5°). C. HOLLINS.

Preparation of organic mercury compounds and dust disinfectants containing them. (SIR) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 329,672, 19.1.29).—Organo-derivatives of metals replaceable by mercury by double decomposition, e.g., the, alkyl, aryl, or aralkyl compounds of lead, tin, bismuth, or magnesium, are treated with a mercury salt (chloride, acetate, etc.) in absence of liquid media. E.g., lead tetraethyl is milled with mercuric acetate and kaolin

to give directly a dusting powder for use as a seed disinfectant. A water-soluble diluent (sodium sulphate) may be used in place of kaolin.

C. HOLLINS.

XVII.—SUGARS; STARCHES; GUMS.

Scholler's process for the conversion of cellulose into sugar. H. LÜERS (Z. angew. Chem., 1930, 43, 455—458).—The mathematical principles governing the reaction and the development of the process during the last six years are reviewed. A small-scale plant has been in operation for some months with satisfactory results; the cellulose, as wood shavings, is heated in a series of vertical tubes while a countercurrent stream of 1% sulphuric acid under pressure at 185° is passed through the tubes, the end tube of the series being emptied, refilled, and made the first of the series after each leach. The lignin residue obtained is about 30% of the weight of wood used, the total solid in the extract 57%, reducing sugars amount to 46%, and fermentable sugars to 38%. The solution obtained contains about 4% of reducing sugars, but only 0.003% N, and processes have been devised for the manufacture of alcohols and various organic acids by suitable fermentation of this liquor. A. R. POWELL.

Determination of the tenacity of starch. SCHULZ and PARLOW (Z. Spiritusind., 1930, 53, 186—187).—The tenacity is deduced from the viscosity at 20° of a solution of the starch prepared by mixing 3 g. of starch with sufficient distilled water to bring the total weight of the solution to 500 g., and warming the mixture on a steam-bath until the mucilage is formed. The viscosity is determined in a Parlow viscosimeter which has a special arrangement at the exit end of the capillary to exclude errors due to surface tension. The moisture content of the starch must also be determined. A determination is made of the concentration of the aqueous glycerin mixture which has the same time of flow as that of the solution of unknown starch, and the amount of Wolff's standard starch to which the unknown starch is equivalent is calculated from a table giving the relative values of Wolff's standard starch and of the aqueous glycerin mixtures.

C. RANKEN.

See also A., July, **Inulin** (PRINGSHEIM and HENSEL). **Purification of polysaccharides** (REILLY and McSWEENEY). 923, **Sweetening power of 5-benzyl-furfuraldoxime** (GILMAN and DICKEY). 965, **Selection characters as correlated with percentage of sucrose, weight, and sucrose content of sugar beet** (PACK).

PATENTS.

Manufacture of dextrin. K. PERL, and F. STEINITZER, CHEM. TECH. INST. (G.P. 456,841, 6.7.26).—A white dextrin of good solubility and low acidity is obtained by heating starchy matter with acids to which has been added a little manganous chloride, aluminium chloride, cobaltous chloride, platonic chloride, or other metallic chloride or nitrate.

C. HOLLINS.

Nitrated carbohydrates (U.S.P. 1,750,948, 1,751,063—4, 1,750,949, 1,751,367, 1,751,377, 1,751,436—8).—See XXII.

XVIII.—FERMENTATION INDUSTRIES.

Yeast testing. W. H. COOK and J. G. MALLOCH (Cereal Chem., 1930, 7, 133—142).—The method consists in measuring the carbon dioxide produced from an 8% maltose solution by a definite quantity of yeast at 28° in presence of sodium monohydrogen phosphate and of potassium dihydrogen phosphate. Using the above medium, the initial p_H of 5.6 falls to 5.2 in 1 hr. and to 4.7 in 2 hrs. The gas produced in 1 hr. is almost exactly half that produced in 2 hrs.; that produced during the first 30 min. is less than that produced during the second, the subsequent period showing an intermediate value. Within wide limits the volume of gas produced is proportional to the concentration of the yeast. Baking tests show that the relative efficiency in dough of two strains of yeast is not necessarily the same as their relative gas production in the above medium, possibly owing to the stimulative effect of the phosphate.

W. J. BOYD.

Solubility of maize proteins in mashing. II. R. H. HOPKINS (J. Inst. Brew., 1930, 36, 296—304; cf. B., 1930, 212).—The permanently soluble nitrogen in wort which is apparently derived from flaked maize when mashed with malt is not appreciably assimilated and removed by the yeast in a primary fermentation. The nitrogen is ultimately assimilable if the alcohol is boiled off after the first fermentation and the de-alcoholised wort seeded with fresh yeast.

C. RANKEN.

Nitrogen balance in brewery mashes and spent washes. I. Electrolyte precipitation of protein substances in brewery mashes. B. BLEYER and W. DIEMAIR [with K. SICHERT] (Landw. Jahrb., 1930, 71, 1—16).—Examination is made of the changes occurring in the nitrogenous matter during the customary processes of preparation and fermentation of potato mash. The Scherning method for the fractional precipitation of proteins is successfully adapted to the elucidation of protein precipitation during steaming, the activity of proteolytic enzymes during mashing, and the further precipitation and decomposition of proteins during fermentation.

A. G. POLLARD.

Brewing value of hop tannin. A. A. D. COMRIE (J. Inst. Brew., 1930, 36, 307—311).—From a review of the experimental evidence on the brewing value of tannin it is deduced that, although the tannin precipitates very little more of the nitrogen in a wort than does mere boiling alone, it accelerates the precipitation to a marked degree, and in order that the precipitate should be as complete and permanent as possible, the tannin must be converted into phlobaphen. Unchanged tannin by remaining soluble tends to hinder fermentation by clogging the surface of the yeast and to give rise to two kinds of haze in the beer. The first type is due to the slow conversion of the soluble tannin compounds into insoluble phlobaphen compounds, a change accelerated by rise in temperature such as in pasteurisation; the second is due to reduction of the solubility of the tannin compounds by fall in temperature such as occurs in the process of beer-chilling.

C. RANKEN.

Sterilisation of beer by filtration. F. HARDER (Woch. Brau., 1930, 47, 305—309).—Yeast and bacteria

are removed from beer by the E.K. filter, which is built up of from 2 to 80 filter-plates each 30 cm. in diam., and the filtering material of which consists chiefly of asbestos. A pressure of 1.5—3 atm. is required, and from a filter containing 40 plates, 35 hectolitres of beer can be obtained in 6 hrs., provided the beer undergoes a normal filtration before entering the filter. The flavour of the beer is improved slightly, the proteins are not altered, and the colour decreases only at the beginning of filtration. Protective colloids are apparently removed, since the filtered beer is more sensitive to cold after pasteurisation than is the normally filtered beer. A haze appears at -1.5° compared with -3° to -4° with the normally filtered beer. Head retention is affected equally by both types of filtration. The stability of all kinds of beer is improved after being passed through the E.K. filter, provided the beer is filled into specially sterile bottles by a sterile bottling machine. With bottles cleansed in the ordinary manner, the E.K.-filtered beer is almost always less stable than that obtained by normal filtration. This follows from a greater sensitiveness to infection, which is probably due to the removal of antiseptic or protective substances.

C. RANKEN.

Protein-tannin combination [in beer]. B. D. HARTONG (Woch. Brau., 1930, 47, 295—297).—When beers are treated with adsorbents, *e.g.*, norit, hide powder, or alumina, the proportion of protein to tannin in the adsorbate is constant for any one beer although the total amounts adsorbed differ widely with different adsorbents. There are indications that the proportion of silicic acid is also constant. This is considered to indicate the presence of a complex colloid containing these substances, the coagulation of which may be a frequent source of turbidity (cf. B., 1929, 833).

F. E. DAY.

Reduction in white wines. J. DUBAQUIÉ (Ann. Falsif., 1930, 23, 295—301; cf. B., 1925, 821).—The gradual appearance of a turbidity, and finally of a brownish-red precipitate, in wines after bottling is discussed. This precipitate consists of organic matter, together with ferrous or cuprous oxides, or complex basic sulphites of these metals. The affected wines can be cleared by aeration. This reduction to cuprous salts is not due to dextrose, but is caused by the presence of reductases in the wines; it occurs mostly in wines which have not remained long enough in barrels, and the reductase can be destroyed by pasteurisation at 75° , after which the wine will remain clear.

B. W. TOWN.

See also A., July, 1958, **Action of poisons on living yeast, dried yeast, and macerated juice** (KOSTYTSCHEV and BERG). **Effects of monochromatic light on growth of yeast** (HUTCHINSON and NEWTON). 959, **Butyric acid fermentation** (ELIASBERG).

PATENTS.

Manufacture of a spirit beverage [apple gin]. W. B. MELROSE, and MELROSE-DROVER, LTD. (B.P. 330,496, 9.2.29).—Ethyl alcohol, which is obtained by the rectification of diluted grain whisky, is diluted to 34% with water and is distilled with various berries,

herbs, roots, and seeds. A tincture of apples in ethyl alcohol is then added together with citric acid to the distillate.

C. RANKEN.

Treatment of molasses [for use in manufacture of yeast]. R. HAMBURGER and S. KAESZ, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,770,402, 15.7.30. Appl., 12.10.23. Czechoslov., 23.12.22).—See B.P. 209,034; B., 1924, 844.

Drying of pulp (B.P. 330,663).—See I. Salad cream (B.P. 330,708).—See XIX.

XIX.—FOODS.

Determination of ash [of flour] by the direct-weight method. E. O. KLOPFSTEIN (Cereal Chem., 1930, 7, 189—191).—The sample (5 g.) is weighed into an aluminium scoop, and transferred to a crucible which is placed in an electric muffle at 593° , the door being left open. When the flame has died out the furnace is allowed to cool to 420° with the door still open. The door is then closed and the temperature raised slowly to 579° and kept at this till 3 hrs. have elapsed from the beginning of incineration. The temperature is further raised to 593° and kept at this for 1 hr. The crucible is cooled in a desiccator till just warm to the touch, and then the ash is transferred to a counterpoised watch-glass on the balance pan. Vitreosil crucibles of 15-c.c. capacity, 41 mm. in diam., and 25 mm. high are used. The method is found to be quicker and more reliable than the usual method of weighing in the crucible.

W. J. BOYD.

Rapid and simple carbide method for determining moisture in flour. M. J. BLISH and B. D. HILES (Cereal Chem., 1930, 7, 99—107).—The flour (1 g.) is weighed into a dry flask fitted with a rubber stopper carrying a 2-way stopcock, through which the flask can be connected to the air or to an open mercury manometer through a T-piece which carries a second stopcock to give communication between the outside air and the manometer. A bent metal spoon, the end of which is forced into the rubber stopper, hangs down inside the flask. Freshly powdered calcium carbide (2—3 g.) is placed in the bowl of the spoon, the stopper is inserted, and the flask connected with the manometer. The stopcock on the T-piece is opened for an instant, then closed, and the flask is tipped and shaken so as to spill the carbide gradually on the flour. Vigorous shaking at intervals of 1 min. (usually for 3—4 min.) is continued until the manometer reading is constant. The apparatus is calibrated by means of repeated determinations, using weighed quantities of water from 0.01 to 0.1 g., and a graph is constructed from which the weight of water corresponding to a given pressure can be read. All the moisture in the flour does not react with the calcium carbide, but, by comparative determinations on many samples, using the carbide and the oven methods, it was found that the residual moisture was reasonably constant for flours of different types and moisture contents. A correction of 0.0455 g. is therefore added to the value obtained from the manometer reading. Hence the method is not applicable to flours containing less than 4.5% of moisture.

W. J. BOYD.

F.p. of milk as a means of detecting added water. G. D. ELSDON and J. R. STUBBS (Analyst, 1930, 55, 423—432).—Hortvet's cryoscopic method (B., 1921, 274 A) has been used for determining the f.p. of milk. Duplicate readings agreeing within 0.002° are readily obtained, the usual deviation being less than this and almost entirely due to errors of parallax. Tables of f.p. depressions obtained by other workers and by the authors are given. An average of 0.54 is taken for the purpose of calculating added water, but no milk is considered as watered on the evidence of the f.p. of a single sample unless the depression falls below 0.53 . The method is applicable only to fresh milk or to slightly sour samples after correction. D. G. HEWER.

Routine detection of nitrates in milk. A. F. LERRIGO (Analyst, 1930, 55, 433—435).—Nitrates may be detected in milk by adding 6—7 drops of mercury reagent (a solution containing 20% of mercuric chloride, 5% of ammonium chloride, and 20 vol.-% of concentrated hydrochloric acid) to 4 or 5 c.c. of milk in a test-tube previously washed with milk, shaking occasionally for 2 min., and filtering into 2 c.c. of diphenylamine reagent (cf. Elsdon and Sutcliffe; A., 1913, ii, 979). With normal milk the line of demarcation is colourless; with fairly large quantities of nitrate it is dark blue, and with quantities of the order of 0.1 pt. of nitric nitrogen per 100,000 or less the blue colour appears at the bottom of the serum on gently agitating. Reagents should not be stored in the same room as nitric acid. The test applied to all samples of milk containing less than 8.5% of solids-not-fat (102 out of 1172) gave a positive reaction for 50.

D. G. HEWER.

[Effect of] adding sodium bicarbonate to milk. P. CAZENEUVE (Ann. Falsif., 1930, 23, 303—305).—This practice, though common, is to be deprecated as it greatly favours the development of pathogenic bacteria. The adulterant can be detected on the effervescence on acidification, and can be determined by ashing a given volume of milk and titrating the aqueous extract with standard acid, using first phenolphthalein and then helianthin as indicators. The second titration gives a figure for the sodium phosphate which arises by double decomposition from the calcium phosphate of the milk.

B. W. TOWN.

Content of salt in cheese. F. MÜNCHBERG (Milch. Zentr., 1930, 59, 201—202).—The cheese (1—2 g.) is boiled with 10 c.c. of halogen-free nitric acid (d 1.40) and 10 c.c. of 0.1N-silver nitrate, and 10% potassium permanganate solution is gradually added till the liquid is clear. The solution is cooled by addition of 150 c.c. of distilled water, and, after further addition of 2 c.c. of saturated iron ammonium alum solution, excess of silver is titrated with 0.1N-ammonium thiocyanate. (1 C.c. of 0.1N-silver nitrate = 0.005856 g. NaCl.) As the salt content should be reckoned on the dry weight of the cheese, the moisture content is determined by mixing 2—3 g. of sample with sand and drying it to constant weight at 105° with frequent stirring. Values found for the moisture and salt contents of various kinds of cheese are given.

W. J. BOYD.

Determination of egg in ice-cream. N. C. SMITH

(J. Assoc. Off. Agric. Chem., 1930, 13, 272—291).—A method for the determination of egg yolk in ice-cream has been worked out and is based on the lipid phosphorus content. The average lipid phosphorus content of a typical egg-free mixture has been calculated to be 2.49 mg. per 100 g., and this value agrees well with the average found for 21 commercial samples, viz., 2.56 mg. per 100 g. Special mixes containing a high proportion of cream have higher lipid phosphorus contents, approximately proportional to the fat content. Analyses of egg products made by Hertwig (cf. B., 1923, 1040 A) showed the average value of the lipid phosphorus of water-free, commercial egg yolk to be 777.7 mg. per 100 g., and this value has been confirmed by the author. The percentage of egg yolk in ice-cream is then given by $100(A - 2.56)/(777.7 - 2.56)$ or $(A - 2.56) \times 0.129$, where A is the phosphorus content of the sample expressed in mg. per 100 g. To calculate the percentage of whole egg the factor 0.129 becomes 0.166, and for fresh egg yolk the dry egg yolk percentage is doubled (total solids in fresh egg yolk = 50%). The Röse-Gottlieb method for extracting the lipoids was found satisfactory, and the phosphorus content of the residue was determined colorimetrically by a modification of the Briggs method (cf. A., 1922, ii, 718), using a solution of potassium dihydrogen phosphate (0.2193 g./litre) as colour standard. By applying these methods and the formula to egg-free mixtures to which known amounts of egg yolk had subsequently been added, an average recovery of 95.5% of the lipid phosphorus was obtained. The general applicability of the formula has been tested on samples of ice-cream without preservative, stored in a refrigerator for periods up to 1 month, on samples containing formaldehyde as preservative stored at room temperature for 10 days, and on samples pasteurised for 40 min. at 63° . The changes in the phosphorus content were insignificant.

H. J. DOWDEN.

Relative merits of sucrose, dextrose, and lævulose as used in the preservation of eggs by freezing. O. M. URBAIN and J. N. MILLER (Ind. Eng. Chem., 1930, 22, 355—356).—The white of egg was found to undergo no change when frozen at -15° , maintained at this temperature for 72 hrs., and then thawed, but the yolk treated similarly became ropy. The cause of this ropiness was found to be the dehydration and coagulation of a lecitho-protein, and could be prevented most efficaciously by the addition of dextrose or lævulose, in the presence of which less than 1% of the lecithin was coagulated, whilst in the presence of sucrose 98% was coagulated. Dextrose and lævulose were also found to prevent the fermentation and bacterial composition of the batters more efficiently than did sucrose. It is suggested that the differences are due to the greater osmotic pressure of a monosaccharide solution compared with a disaccharide solution of equal concentration. A. SHORE.

Use of lead acetate in the determination of the acidity of fruit products. B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1930, 13, 259—265).—The total free acidity of phosphates is not indicated by direct titration using phenolphthalein as indicator, but when lead acetate is added an equivalent

amount of acetic acid is liberated which gives a true measure of the acidity. In the examination of fruit products, 200 c.c. of the solution (or 25 c.c. of fruit juice diluted to 200 c.c.) are mixed with 20 c.c. of dilute nitric acid (15 c.c. in 1 litre), and to the mixture are added 20 c.c. of lead acetate solution (100 g. in 300 c.c. of water containing 10 c.c. of glacial acetic acid diluted to 1 litre and filtered). After shaking, diluting to 250 c.c., and filtering, lead is removed by adding solid potassium oxalate in slight excess, and after refiltering, the filtrate is titrated with 0.1N-caustic soda against phenolphthalein. The solutions are partially decolorised by precipitation of the lead and a sharp end-point was always obtainable. The results given by the method checked excellently with materials of known composition, but were always higher than those given by direct titration and by electrometric titration.

H. J. DOWDEN.

Determination of caffeine in decaffeinated coffee.

W. F. ALLEN (J. Assoc. Off. Agric. Chem., 1930, 13, 265—272).—In the official (Power-Chesnut) and the tentative (Fendler-Stüber) A.O.A.C. methods of analysis, the caffeine content is based on the weight of the residue after removal of the chloroform, and may be verified by nitrogen determinations on the residue (factor $\times 3.464$). Two commercial brands of decaffeinated coffee were examined by each method, and the purity of the residues was tested by nitrogen determinations and by sublimation at 180—190° for 10 hrs. The results showed that values based on the weight of the residues are quite erroneous since only 20—40% is pure caffeine. Satisfactory results are given by nitrogen determinations either by micro- or macro-Kjeldahl methods (provided the dilute solutions of the former are used for the latter method), and these results are in close agreement with those obtained by sublimation. The average values of 15 samples examined by the Power-Chesnut and Fendler-Stüber methods were: 0.0569 and 0.0487% of caffeine, respectively, by direct weighing; 0.0203 and 0.0183% by nitrogen determination; 0.0208 and 0.0174% by sublimation; and 0.0127 and 0.0151% by nitrogen determination after sublimation.

H. J. DOWDEN.

Vitamins. I. Vitamin content of foods and delicacies. II. Vitamin content of preparations manufactured from fresh vegetables [fruit]. F. V. VON HAHN (Z. Unters. Lebensm., 1930, 59, 4—18, 18—31).—I. A discussion of technique.

II. Results of the examination of commercial preparations from oranges and lemons and of lemon substitutes are detailed.

H. J. DOWDEN.

Control of mineral waters and their salts under the Federal Food and Drugs Act. J. W. SALE (Ind. Eng. Chem., 1930, 22, 332—335).—The application of the Federal Food and Drugs Act to bottled mineral waters is discussed, and suggested methods of analysis are outlined.

A. SHORE.

See also A., July, 872, **Photochemical oxidation of food materials by air** (PALIT and DHAR). 963, **Colorimetric determination of the anti-beri-beri vitamin content of rice** (SPRUYT). **Fat-soluble vitamins of**

milk (CRAWFORD and others). 964, **Biochemistry and histochemistry of fruits and seeds** (NIETHAMMER).

Disintegration. CHWALA.—See I. **Yeast testing.** COOK and MALLOCH.—See XVIII. **Fumigation with ethylene oxide.** RUSS.—See XXIII.

PATENTS.

Deodorising and similar treatment of milk, cream, and other liquids containing butter fat. A. E. WHITE. From PFAUDLER Co. (B.P. 330,662, 19.3.29).—In an apparatus described, the milk etc. heated to above 38° is subjected in a fine state of division to aeration under reduced pressure. By this means the objectionable natural flavours sometimes present in milk and cream may be removed. E. B. HUGHES.

Apparatus for pasteurising milk or other liquids. AKTIEB. RUDELIUS & BOKLUND (B.P. 330,867, 14.11.29. Swed., 28.11.28).—Closed containers for pasteurising milk by the "holding" process are described in which the temperature of the surface of the milk is maintained by heating-tubes just below and also above the surface of the liquid. E. B. HUGHES.

Continuously producing ice-cream and other plastic substances. C. W. VOGT, and VOGT INSTANT FREEZERS, INC. (B.P. 330,887, 18.1.29).—The material, such as ice-cream or vegetable or animal oils, with or without air or other gas, is forced under pressure through a cylindrical space wherein the material is kept in constant agitation. Means are provided for cooling or heating the material during agitation and, in order to maintain a constant plasticity, the effect of the cooling or heating medium is controlled by the consistency of the material passing through. E. B. HUGHES.

Manufacture of salad cream. CROSSE & BLACKWELL, LTD., and W. CLAYTON (B.P. 330,708, 29.4.29).—Salad creams or dressings may be prepared by emulsifying a vegetable oil (olive oil) and milk with egg-yolk, sugar, starch, or other suitable nidus for bacterial growth. Edible organic acids (acetic and lactic) may be added if desired. After emulsifying, a bacterial culture of *Streptococcus lacticus* or *Hollandicus* or *B. Bulgaricus* is added which matures the cream in the bottle, imparting definite aroma and flavour.

E. B. HUGHES.

Preservation of gherkins and the like. F. TARNAWSKI (B.P. 331,161, 17.10.29. Czechoslov., 18.10.28).—Such materials are fermented in 10% salt solution, soaked in water, spices and other vegetables and a 2% unsweetened vinegar solution added, and the product is sterilised in sealed vessels for 5—10 min.

E. B. HUGHES.

Process and apparatus for expressing material, e.g., of the consistency of dough or pasty material. O. KREMMLING (B.P. 331,489, 28.3.29).

Machines for treating fish. GEN. FOODS Co., Assees. of C. BIRDSEYE (B.P. 309,018, 7.1.29. U.S., 3.4.28).

Treatment of non-fibrous material (B.P. 330,649).—See V. **Edible fatty products** (B.P. 301,750).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Porphyroxine test for Indian opium. F. BAMFORD (Analyst, 1930, 55, 445—446).—The purple colour produced in the porphyroxine test for opium has been regarded as characteristic of Indian opium and as a means for differentiating the latter from Turkish or Smyrna opium, but a sample recently seized in the Egyptian Custom House, and known to be Turkish, gave a positive reaction. D. G. HEWER.

Methylene-blue, U.S.P., as precipitant of Irish moss. G. E. EWE (J. Amer. Pharm. Assoc., 1930, 19, 568—570).—Zinc-free methylene-blue is almost quantitatively precipitated from aqueous solution by an extract of Irish moss, 1 g. of dry extractive matter being equivalent to 1.17 g. of dye at the ordinary temperature and to 1.30 g. at 100°. A precipitate is not obtained from methylene-blue with the mineral constituents of the moss, or with other gums, nor from moss extract with 15 other typical dyes. H. E. F. NOTTON.

See also A., July, 897, Local anæsthetics from dialkylaminopropanediols (RIDER). 916, Reactions of 4-hydroxy-3-ethoxybenzaldehyde (KLOTZ). 921, Essential oil of *Backhousia angustifolia* (GIBSON and others). 926, Synthesis of 2-methylquinuclidine (WINTERFELD). 935, Potentiometric measurements of certain homologues of ephedrine (KRAUTZ and HARTUNG). Amine oxides of alkaloids (POLONOVSKI and POLONOVSKI). 936, Strychnine alkaloids (WIELAND and MÜNSTER; LEUCHS and KRÖHNKE). Tetrachloriodides of alkaloids (CHATTAWAY and PARKES). 937, New reaction for codeine and dionine (DE HAAS). Neoarsphenamine (JURIST and CHRISTIANSEN). Arsenic derivatives of pyridine (BINZ and others). Quinoline compounds containing arsenic (SLATER). 938, 10-Chloro-5:10-dihydrophenarsazine and its derivatives (GIBSON and JOHNSON). 960, Purification of anticarbuncle serum (MODERN and WERNICKE). Germicidal power of colloidal silver and of ionic silver on *B. typhosus* (SAMAAAN). 961, Bio-assay of adrenaline-procaine mixtures (MUNCH and DECKERT). Preparation of insulin (KAULBERSZ). 966, Non-volatile constituents of *Mentha piperita* (BRAUN). Essential oil of green violet leaves. Mignonette oil (WALBAUM and ROSENTHAL). Volatile oils in plant economy (MARSH and MAUS). 968, Testing of ampoule glass for alkalinity (STICH).

Aluminium sulphate. KULLERUD and HØRLÜCK.—See VII. Toxicity of pyrethrum vapours. GINSBURG.—See XVI. Control of mineral waters. SALE.—See XIX.

PATENTS.

Treatment of tobacco. DEVELOPMENT OF INDUSTRIES, LTD., and V. A. GAVRILOVITCH (B.P. 331,026, 25.4.29).—Tobacco is subjected to the effects of high-frequency electricity, with or without ultra-violet rays, ozone, and electric sparks. E. H. SHARPLES.

Ephedrine-aldehyde and -ketone compounds and solutions, and their preparation. E. H. STUART, Assr. to E. LILLY & Co. (U.S.P. 1,749,361

and 1,749,452, 4.3.30. Appl., [A] 3.5.28, [B] 20.8.28. Renewed [B] 8.1.30).—(A) An aldehyde is added to an ethereal solution of ephedrine. The products from *l*-ephedrine with acetaldehyde (b.p. 239°), furfuraldehyde (m.p. 48°), citral, heptaldehyde, benzaldehyde (m.p. 73°), anisaldehyde (m.p. 89.5°), *o*-methoxybenzaldehyde (m.p. 103.5°), salicylaldehyde (m.p. 117°), veratraldehyde (m.p. 97°), vanillin, and cinnamaldehyde (m.p. 92.5°), and from *d*- ψ -ephedrine with cinnamaldehyde (m.p. 75°), benzaldehyde (m.p. 67°), salicylaldehyde (m.p. 79—80°), and vanillin (m.p. 124.5—125°) are described. These are soluble in paraffin oil and are used for nasal sprays. (B) Similarly applicable oil-soluble products are made by dissolving ephedrine in liquid (or molten) ketones, reaction being complete in several days or weeks. Compounds described are: from *l*-ephedrine with acetone (m.p. 46°), acetophenone (m.p. 34°), methyl ethyl ketone, menthone (m.p. 35°), and camphor; *d*- ψ -ephedrine with acetone (b.p. 152.5°/12 mm.), acetophenone (m.p. 115°), methyl ethyl ketone, ethyl propyl ketone, and camphor (m.p. 87°).

C. HOLLINS.

Preparation of α -*p*-hydroxyphenyl- β -methylaminopropanol [β -methylamino- α -*p*-hydroxyphenylpropyl alcohol]. I. G. FARBENIND. A.-G. (B.P. 314,448, 27.6.29. Ger., 27.6.28).—This therapeutic compound, m.p. 152—154° (hydrochloride, m.p. 209—211°; oxalate, m.p. 216—218°), is obtained by hydrogenation of the corresponding propiophenone or its *N*-benzyl derivative in presence of a nickel catalyst at 100° or palladium at 15°, with hydrogen at 40 atm.

C. HOLLINS.

Manufacture of therapeutical agents. I. G. FARBENIND. A.-G. (B.P. 306,905, 26.2.29. Ger., 27.2.28).—Salts of ethers of 6:8-dihydroxyquinoline with 5:5-disubstituted barbituric acids are made by double decomposition, e.g., from sodium 5-phenyl-5-ethylbarbiturate and 6:8-diethoxyquinoline hydrochloride, or sodium 5-cyclohexenyl-5-ethylbarbiturate and 6:8-dimethoxyquinoline hydrochloride. They are tasteless and have hypnotic and antispasmodic properties.

C. HOLLINS.

Mercury-bromine derivatives of fluorescein. W. L. VOGEL, Assr. to P. W. PRUTZMAN (U.S.P. 1,749,201, 4.3.30. Appl., 28.5.28).—Fluorescein is monobrominated in acetic acid. The product, m.p. 110°, is boiled with mercuric acetate solution, and the precipitated mercury compound is treated with the calculated amount of alkali to give $C_{20}H_5O_5Br(HgOH)_2Na_4$, a non-irritant antiseptic. C. HOLLINS.

Increasing the therapeutic action and lowering the toxicity of arsenic-containing organic therapeutic substances. J. I. STEINFELD (B.P. 308,600, 19.3.29. Ger., 24.3.28).—Arsenicals such as atoxyl, salvarsan, neosalvarsan, myosalvarsan, etc. are treated with phosphatides (e.g., lecithin, cephalin, and cerebrosides), or with extracts of animal organs rich in phosphatides, such as brain, liver, etc., or suspensions prepared from such organs. Adsorption compounds are obtained which, although having a very small arsenic content, possess the same therapeutic action

as that possessed by the arsenic compounds alone. The preparations are more resistant to atmospheric oxidation and the increased therapeutic action is an inherent property of the adsorption compound and is not obtained by the separate incorporation of the two substances.

E. H. SHARPLES.

Preparation of compressed iodine in the form of rods, pencils, etc. O. and I. BRAUN (B.P. 331,118, 19.7.29. Ger., 18.4.29).—Iodine is mixed with an iodine compound, such as potassium iodide, and small quantities of water, and the resulting mass is converted into a paste with binding agents such as bolus or kaolin and a large proportion of lactose. The plastic masses are dried at a low temperature, mixed with sufficient quantities of talc, and compressed into pencils, rods, etc.

E. H. SHARPLES.

Obtaining gland extracts. PARKE, DAVIS & Co. (B.P. 303,362, 21.12.28. U.S., 31.12.27).—Hormones are separated from glandular substances by treating the material with a lower fatty acid in relatively high concentration (98–100% acetic, 95% propionic, 90% butyric acids, etc.) followed by treatment of the acid extract, which contains the hormones in solution but little foreign protein, with organic solvents such as acetone, sulphuric ether, and light petroleum. The active principles are precipitated. Precipitation may be fractional and fractions having various degrees of physiological activity obtained. The most active precipitates can be redissolved in fatty acid and reprecipitated with solvents. By extraction of the posterior lobe of the pituitary gland, or active extract thereof, with the concentrated fatty acid followed by precipitation with sulphuric ether, a fraction high in the pressor principle (*b*-hypophamine) and low in oxytocic principle (*a*-hypophamine) is obtained. From the filtrate, by addition of light petroleum, a precipitate having high oxytocic and low pressor activity is formed. The fatty acid extract may be treated with acetone to remove foreign proteins prior to the above fractional precipitation. By repeated fractionation, the two principles can be obtained substantially free from each other; pressor and oxytocic principles are thereby obtainable having a potency 80 and 150 times as great, respectively, as that of the U.S.P. standards.

E. H. SHARPLES.

Manufacture of sexual hormones. SCHERING-KARLBAUM A.-G. (B.P. 307,844, 13.3.29. Ger., 14.3.28).—The faeces of mammals is extracted with either a water-soluble organic solvent, the hormone being separated in the customary manner, or with a water-insoluble solvent, the hormone being isolated from the unsaponifiable portion of the extract. The starting material may be dried, or mixed with water and alkali, or with an alkaline earth, or an indifferent porous material may be added previous to extraction.

E. H. SHARPLES.

Medicinal articles. [Coated dried fruit carrying a medicine at the core.] C. W. WALLIS (B.P. 331,321, 7.5.29).

Alkylene-substituted phenols (B.P. 304,727). **Amino-alcohols** (B.P. 307,304). **o-Hydroxycarbox-**

ylic acids of fluorene (B.P. 330,305).—See III. **Organic mercury compounds** (B.P. 329,672).—See XVI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Light filters for the isolation of narrow spectral regions. L. A. JONES (Phot. J., 1930, 70, 337–346).—A series of eight filters of good stability suitable for the isolation of spectral bands in the infra-red, ultra-violet, and visible spectra have been constructed from a combination of Wratten dyed-gelatin filters, various grades of Corning glass, aqueous solutions of nickel and copper sulphates, and distilled water. The components and transmission characteristics obtained by spectrophotometric methods are given for each filter.

J. W. GLASSETT.

See also A., July, 871, **Photochemistry of silver nitrate** (BELENKY and JUSE). **Microtome studies of photographic layers** (SCHAUM and KLEIN). **Photosensitisation by ammonia** (TAYLOR and EMELEUS).

Heat expansion of solids. COHEN.—See I.

PATENTS.

Photographic and kinematographic films. K. BRATRING (B.P. 307,431, 6.3.29. Ger., 7.3.28).—The inflammability of films is greatly reduced by suitably securing to the picture side only or to both sides of the finished film, which may be very thin, a thin layer of a non-inflammable cellulose derivative, such as the hydrate or acetate.

J. W. GLASSETT.

Apparatus and developer for developing [diazotype] photographic prints. F. VAN DER GRINTEN (B.P. 330,625, 13.3.29. Holl., 8.12.28).—Apparatus for developing diazo-types with a solution containing alkaline substances, azo dye components, buffer salts, and reducing agents is claimed.

J. W. GLASSETT.

Photographic printing by imbibition. F. J. TRITTON, and COLOUR SNAPSHOTS (1928), LTD. (B.P. 330,199, 12.12.28).—A process is developed for photographic printing on a layer of silver halide-gelatin emulsion containing an orange or yellow dye, developing, fixing, and tanning the layer, so as to produce the gradations in the image in relief in the gelatin. The latter is dyed and printing carried out by pressing on a layer of gelatin, which takes up the dye by imbibition.

J. W. SMITH.

Colour photography. C. L. FINLAY, and FINLAY PHOTOGRAPHIC PROCESSES, LTD. (B.P. 329,018, 9.2.29).—In the preparation and reproduction of colour transparencies, the colour record is obtained in intimate optical contact with the colour viewing screen by coating the latter with a gelatin layer, which is sensitised with a solution of alkaline potassium dichromate, dried, and exposed, after colour relation has been adjusted, behind a positive colour-record transparency. The screen is removed, bathed in water, and the differentially hardened image is treated with a solution of a black dye (nigrosine), which is absorbed in the unhardened parts, washed, and dried. Any brown or blue tinge must be

neutralised by the addition to the bath of a small quantity of blue or brown dye, respectively, of similar character.

J. W. GLASSETT.

Films for colour photography. KODAK, LTD., Assees. of J. G. CAPSTAFF (B.P. 312,992, 4.5.29. U.S., 4.6.28).—To lessen halation and the consequent degradation of colour values, a dye, preferably bleachable by prolonged exposure to light, is incorporated or applied in a thin layer over the lenticulated support in sufficient concentration to absorb about 20% of the incident light. Kryptocyanine dissolved in a 1:2 mixture of ethyl acetate and ethyl alcohol is recommended for the purpose.

J. W. GLASSETT.

Production of multicoloured transparencies or diapositives, especially applicable to kinematograph films. K. WAHL (B.P. 306,026, 18.1.29. Ger., 14.2.28).—A cellulose film is coated on both sides with a chromated gelatin solution capable of giving differentially hardened images. One side is exposed under a negative and the other, after alinement, under a positive; alternatively, both may be exposed under a negative. The images so formed are then dyed in different colours by imbibition or by tinting after the unhardened gelatin has been removed by hot water.

J. W. GLASSETT.

Production of multicolour pictures. JOS-PE FARBENPHOTO-GES.M.B.H. (B.P. 330,997, 27.3.29. Ger., 28.2.29).—Very flat gelatin reliefs corresponding to the colour-record images are produced by heavily tinting the silver halide emulsion with a colour complementary to the exposure light, exposing through the support, treating after development with metallic salts to give differential hardening, and, finally, after fixation, removing the unhardened gelatin with warm water. These reliefs are then dyed with the appropriate colours, and applied successively in register to a gelatin-coated support.

J. W. GLASSETT.

Recovery of celluloid and silver from scrap films. H. PICK (B.P. 331,006, 8.4.29).—The scrap film is treated first with a warm (30–50°) 1% solution of caustic soda, which serves to free the silver-gelatin layer, and then for at least 2 hrs. with cold *N*-caustic soda, which removes the gelatin substratum. The gelatin and silver are then precipitated by coagulation at the b.p. with aluminium sulphate.

J. W. GLASSETT.

Production of negative or positive printing plates. MEISENBACH, RIFFARTH, & Co. A.-G. (B.P. 310,500, 15.12.28. Ger., 27.4.28. Addn. to B.P. 302,887; B., 1930, 350).—The dried collodion plates may be etched by the process of the prior patent if treated first with a swelling agent (ether) and then with the hygroscopic agent (*e.g.*, glycerin) and the protective varnish.

J. W. GLASSETT.

Manufacture of light-sensitive films. U. DIEM-BERNET (U.S.P. 1,769,940, 8.7.30. Appl., 17.5.27. Switz., 21.5.26).—See B.P. 271,507; B., 1928, 625.

Manufacture of diazo-type sensitive papers and the like. II. D. MURRAY, ASS. to NORTON & GREGORY, LTD. (U.S.P. 1,770,352, 8.7.30. Appl., 28.9.29. U.K., 14.6.28).—See B.P. 320,395; B., 1930, 218.

XXII.—EXPLOSIVES; MATCHES.

Comparison between calculated and experimental explosion pressures. H. MURAOUR and G. AUNIS (Compt. rend., 1930, 190, 1547–1549; cf. A., 1925, ii, 319; 1926, 480).—The pressures attained on explosion of a powder consisting of 52% of guncotton (12.2% N) and 48% of nitroglycerin in tube form (8.80 × 4.25), at five densities of loading (0.062–0.285), as measured by crusher, and corrected by two different methods for the cooling effect of the walls, are compared with calculated results. These assume a temperature of explosion of 3600°, and are based on an extrapolation of the Nernst-Wohl curves for sp. heat, and on gaseous compositions, taking into account the dissociation at different pressures. Agreement is (with one exception) within 3.5%. It is inferred that the correction for cooling at the lower pressures is doubtful.

C. A. SILBERRAD.

Coloured-light recipes. L. VANINO (J. pr. Chem., 1930, [ii], 127, 16–19).—The following coloured phosphorescent mixtures are produced by heating the constituents together for 1 hr., the number of parts being in parenthesis. *Violet-blue*: quicklime (15.0), strontium hydroxide (5.0), sulphur (8.0), magnesia (1.0), sodium carbonate (3.0), lithium sulphate (1.0), and 6 c.c. of 0.3% aqueous colloidal bismuth solution; or, alternatively, quicklime (17.0), strontium hydroxide (5.0), sulphur (8.0), lithium fluoride (1.0), magnesia (1.0), 1.0 c.c. of 1% rubidium nitrate solution, and 6 c.c. of 0.3% colloidal bismuth solution. *Violet*: quicklime (20.0), barium hydroxide (10.0), sulphur (9.0), sodium sulphate (1.0), potassium sulphate (1.0), lithium sulphate (1.5), 2 c.c. of 0.5% alcoholic bismuth nitrate solution, 1 c.c. of 0.5% aqueous thallium sulphate solution, and 0.5 c.c. of 0.5% aqueous thorium sulphate solution (the nitrates may be substituted for thallium and thorium sulphates). *Blue*: strontium hydroxide (15.0), quicklime (5.0), sulphur (8.0), magnesia (1.0), sodium carbonate (3.0), lithium sulphate (1.0), and 6 c.c. of 0.3% colloidal bismuth solution. *Green*: strontium hydroxide (20.0), sulphur (8.0), magnesia (1.0), sodium carbonate (3.0), lithium sulphate (1.0), and 6 c.c. of 0.3% colloidal bismuth solution.

A. I. VOGEL.

See also A., July, 880, **Quantitative analysis of azides** (REITH and BOUWMAN).

Cellulose benzoate nitrates. SENDO and KONDO.—See V.

PATENTS.

Initially detonating explosives and manufacture of detonating caps. E. VON HERZ (B.P. 331,162, 17.10.29. Ger., 18.10.28).—In detonators the main charge consists of a nitric ester of the general formula $C_nH_{n+2}(NO_3)_n$, such as hexanitromannitol or its molecular mixture with nitropentaerythritol. This is covered by a perforated cap and then pressed at 100–400 kg./cm.² The priming charge may consist of a mixture of an oxygen carrier, such as barium nitrate, and aluminium or magnesium powder, together with lead hypophosphite, if desired, and is pressed at the same pressure as the main charge.

W. J. WRIGHT.

Manufacture of nitrated sugar explosives. P. G. WRIGHTSMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,750,948 and 1,751,063, 18.3.30. Appl., [A] 17.9.27, [B] 7.12.27).—(A) Sugar is nitrated in the form of a suspension in a mixture of glycerin and ethylene glycol, the sugar particles being of such fineness as to pass 100-mesh, and the amount of sugar being greater than 25%. (B) A mixture containing more than 5% of a monosaccharide and about 25% of a disaccharide is dissolved in a polyhydric alcohol, or mixture of polyhydric alcohols of lower viscosity than that of glycerin, and nitrated. A suitable composition consists of dextrose 10–30%, sucrose 20–25%, and ethylene glycol 45–70% (or ethylene glycol 20–60% with glycerin 20–50%). W. J. WRIGHT.

Manufacture of nitrated carbohydrate explosive. P. G. WRIGHTSMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,750,949 and 1,751,377, 18.3.30. Appl., [A] 17.8.28, [B] 7.11.27).—(A) Mixtures of carbohydrates and polyhydric alcohols having 4–6 carbon atoms are nitrated in a liquid polyhydric alcohol medium, the maximum amount of carbohydrate being thus held in solution. A suitable mixture consists of sucrose 20–25% and sorbitol 10–50%, the remainder being a mixture of glycerin and ethylene glycol. (B) Sugar is nitrated at 10° in suspension in monochlorohydrin, or in a mixture of monochlorohydrin and glycerin, the particles of sugar being of such fineness as to pass 100-mesh, and the amount of sugar in suspension being 25–50%. W. J. WRIGHT.

Manufacture of explosives. C. C. AHLUM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,751,064, 18.3.30. Appl., 20.4.28).—A liquid explosive of sufficiently low viscosity to permit of its being readily stabilised is produced by nitrating a solution of a polyhydric alcohol having 4–6 carbon atoms, such as sorbitol, in a liquid polyhydric alcohol, such as glycerin, ethylene glycol, or a mixture of these. Alternatively, a mixture of the solid and liquid polyhydric alcohols may be nitrated, or the alcohols may be introduced separately into the nitrator and then nitrated.

W. J. WRIGHT.

Treatment of nitrated bodies. J. V. SKOGLUND, Assr. to TROJAN POWDER Co. (U.S.P. 1,751,367, 18.3.30. Appl., 8.10.21).—To remove residual acid from nitrostarch, the material is placed on a filter plate in a displacement vessel and subjected to suction or pressure. Sulphuric acid of the same sp. gr. as that of the mixed acid is then caused to flow over the material to displace the mixed acid, under suction or pressure as before, the nitrostarch, saturated with sulphuric acid, being finally removed and "drowned" in water. Alternatively, the sulphuric acid may be removed from the nitrated material by displacement with water instead of by "drowning."

W. J. WRIGHT.

Manufacture of (A) blasting explosive, (B) gelatinised explosive, (C) nitrated sorbitol. F. H. BERGEM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,751,436–8, 18.3.30. Appl., [A] 3.5.28, [B] 27.6.28, [C] 6.10.28).—(A) A non-freezing dynamite contains erythritol tetranitrate, an inorganic nitrate, and a

carbohydrate, together with a solid explosive, such as nitrostarch, if desired. (B) A gelatinised explosive is prepared by mixing nitrated sorbitol, together with another polyhydric alcohol having 4–6 carbon atoms, if desired, with nitrocellulose and inorganic oxidising salts and combustibles. (C) An explosive of low m.p. and high power, suitable for use in bombs and high-explosive shells or as a component of dynamites, is obtained by nitrating sorbitol, dissolving the hexanitrate formed in alcohol, and precipitating with water. The product contains 17–18.48% N and has m.p. 44–54°. W. J. WRIGHT.

Lachrymators (U.S.P. 1,750,101).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Iodine content of waste water [from town sewage]. R. KÖHLER (Z. angew. Chem., 1930, 43, 503–507).—Analyses of effluent water before and after filtration at various German sewage farms show that it contains appreciable quantities of iodine, much of which remains in the slime. During the course of purification of the water the iodine decreases with decreasing content of organic matter; most of this loss of iodine escapes into the air, especially during filtration through the sand filters. The use of the slime as a fertiliser results in an appreciable increase in the iodine content of the crop, especially in the case of rye.

A. R. POWELL.

Fumigation with ethylene oxide. J. M. RUSS, JUN. (Ind. Eng. Chem., 1930, 22, 328–332).—The most efficient methods of fumigating grain, store rooms, and refrigerator cars with ethylene oxide are described. Tests show that a mixture of ethylene oxide and carbon dioxide is a more efficient fumigant for the destruction of insect pests than the former substance alone. For the fumigation of grain in enclosed bins a dosage of 2 lb. of ethylene oxide and 14 lb. of solid carbon dioxide per 1000 bushels of grain, introduced gradually into the grain as it enters the bin, is recommended. This fumigant has no deleterious effect on any of the materials on which it has been tested (grain, cotton, dried fruit, nuts, etc.); it does not leave an odour and is non-inflammable. A mixture of carbon dioxide and ethylene oxide ("Carbo-oxide") is supplied in cylinders and can be used for house fumigation. The dosages and times of contact for various types of fumigation are given. A. SHORE.

See also A., July, 884, **Distillation apparatus for water** (FRIEDRICHS).

Disintegration. CHWALA.—See I.

PATENTS.

Insecticides. I. G. FARBENIND. A.-G. (B.P. 308,661, 25.3.29. Ger., 24.3.28).—A mixture comprising ethylene oxide and carbon tetrachloride is claimed.

H. ROYAL-DAWSON.

Fumigating apparatus. H. A. SMITH, and LAWES' CHEM. MANURE Co., LTD. (B.P. 331,192, 23.3.29).

Alkylene-substituted phenols (B.P. 304,727).—See III.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 12 and 19, 1930.*

L—GENERAL; PLANT; MACHINERY.

Coefficient of heat transfer from the internal surface of tube walls. A. EAGLE and R. M. FERGUSON (Proc. Roy. Soc., 1930, A 127, 540—566).—Full details are given of the apparatus and technique used for measuring the coefficient of heat transfer k from a heated brass tube to water flowing through it. The principal innovations in the present method are (a) the direct heating of the tube by a low-tension alternating current, and (b) the discarding of the use of thermocouples to obtain the temperature of the water, this being calculated for any cross-section from the amount of heat put into the water up to that cross-section. No definite value of k is obtainable till the temperature distribution over the cross-section has reached its limiting form. The water temperature was varied from about 5° to 60°, the water velocity from 30 to 330 cm./sec., the inside diameter of the tube from 1.02 to 3.56 cm., and the heat flow rates from 0.30 to 1.50 g.-cal./sq. cm./sec. It is shown that the limiting coefficient is, apart from a simple factor, a function of Reynolds' number $\tau \equiv \rho v d / \mu$ and $\sigma \equiv \mu s / c$. The results for very small rates of heat flow, H , can be expressed in the form $\rho v s / k_0 = A + B(\sigma - 1) - C(\sigma - 1)^2$, where A , B , and C are positive functions of τ only and k_0 is the value of k when $H = 0$. This holds for all values of σ up to about 15, and for values of τ from about 5000 to ∞ . A theory, agreeing with experiment, is given showing the difference between k_0 and k_H when τ is large, and a simple empirical rule for the difference when τ is smaller. L. L. BIRCHMISHAW.

Rapid film evaporator for unstable liquors and extracts. O. ZAHN (Chem. Fabr., 1930, 285—286).—The evaporator is built up of a number of superimposed elements each consisting of a distributing plate surrounded by a circular gutter, from which the liquid overflows, passing down the sides of a funnel to fall upon the centre of the next plate. A central vertical shaft rotates the plates. Separate heating chambers are provided for each compartment, so that temperature can be controlled as required, and the vapours can be drawn off separately and fractionally condensed if necessary. The liquid passes through the evaporator in 2—5 min., only 20—30 litres being in the apparatus at one time, and an evaporation of 80—90 kg. of steam per hr. per sq. in. of heating surface is obtained without vacuum. C. IRWIN.

Life of filter cloths. M. WERNER (Chem. Fabr., 1930, 277—279, 286—287).—Twill filter cloths may be treated with sodium nitrate and mixed acids so as to contain 12—12.5% N. The nitrated cloth is much more resistant to acid than is untreated cloth, but as it is explosive when dry it must be kept under water. Nitration beyond the point mentioned results in a total

loss of mechanical strength. Tests were made of the strength of treated and untreated cloth after exposure to sulphuric and hydrochloric acid at various temperatures and concentrations. The initial strength is reduced by nitration, but the reduction in strength by time of exposure or by rise of temperature is very much less. The life of the cloth is in either case reduced by increase in concentration, and more rapidly by hydrochloric acid than by sulphuric acid. It is estimated that the life is increased 50 times by nitration when used in acid solutions. Nitrated cloth can be used in 20% sulphuric acid at 40°, 20% hydrochloric acid at 20°, or 10% hydrochloric acid at 40°. C. IRWIN.

Comparison between horizontal and vertical centrifugals. G. OEHLER (Chem. Fabr., 1930, 265—266).—A horizontal centrifugal machine yields an even layer of solid matter around the container, whilst a vertical machine deposits the solid layer parabolically. A method of calculating the capacity of a vertical centrifuge is worked out. Theory indicates that in consequence of this distribution a vertical machine should be less efficient than a horizontal one. Comparative tests on machines of the two types with common salt, Glauber's salt, sodium bicarbonate, and potassium carbonate are described. These showed that under similar conditions the residual moisture in material from the horizontal centrifugal was 70—90% of that from vertical machines. Measurements of liquid separated are in agreement with this result. C. IRWIN.

Centrifugal concentration. H. A. DOERNER (U.S. Bur. Mines Tech. Paper 457, 1929, 39 pp.).—Experiments in a laboratory tube-centrifuge proved that the separation of minerals effected was due to centrifugal concentration, and not merely to free or hindered settling. Using a machine of semi-commercial size, the possibility of such separation on a large scale was demonstrated and the various factors involved were ascertained.

C. A. KING.

Determination of particle size in pulverised substances. K. KASAI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1930, 13, 135—183).—An absolute value of the surface area of large fractionated glass particles is determined from the rate of dissolution in hydrogen fluoride. The surface of a smaller particle relative to that of the larger ones is determined by adsorption. These particles are conveniently conceived as ellipsoids, the axis of the ellipsoid being used to specify any given set of particles. A. B. D. CASSIE.

Method of measuring granular material by volume. H. WINKELMANN (Chem. Fabr., 1930, 269—270).—A description of the Lea apparatus, which is claimed to give an accuracy of $\pm 2\%$. C. IRWIN.

* The remainder of this set of Abstracts will appear in next week's issue.

Classification of plastics and definition of certain properties. E. KARRER (J. Rheology, 1930, 1, 290—297).—The meaning of the terms solidity, elasticity, viscosity, and plasticity is discussed, and these properties are made the basis of a comparison of the plastic state with the ideal liquid and solid state. Plastics are classified according to their method of preparation as mechano-, chemo-, solvo-, thermo-, and cheo-plastics.

T. H. MORTON.

Search for a general law of the flow of matter. M. REINER (J. Rheology, 1930, 1, 250—260).—Mathematical. The use of empirical formulae for the representation of viscous flow is criticised, and it is shown that by using a generalised expression connecting velocity gradient with shearing force it is possible to arrive at an expression which permits the derivation of a numerical law for capillary or rotating-cylinder viscosimeters which is independent of the instrument.

T. H. MORTON.

Kinetic energy correction in fluid flow. S. B. STONE (J. Rheology, 1930, 1, 240—249).—A mathematical investigation of the modification of the flow of fluids through a capillary tube by an energy term which is extended to include, not only the usual kinetic energy of efflux, but also the rotational energy of suspended particles and the energy absorbed in the change of colloidal structure. It is shown that a fluid may obey Poiseuille's law without obeying the fundamental (Newton's) viscosity law.

T. H. MORTON.

New viscosimeter. A. KÄMPF (Kolloid-Z., 1930, 51, 165—167).—The viscosimeter described earlier (cf. Kämpf and Schrenk, A., 1930, 1014, originally made for work on artificial silk, is suitable for the general investigation of highly viscous liquids. E. S. HEDGES.

Testing and calibration of the new Kämpf viscosimeter. O. SCHRENK (Kolloid-Z., 1930, 51, 167—171).—Tests on the Kämpf viscosimeter (cf. preceding abstract) show that it is comparable in accuracy with any existing viscosimeter.

E. S. HEDGES.

Influence of the proximity of a solid wall on the consistency of viscous and plastic materials. G. W. S. BLAIR (J. Physical Chem., 1930, 34, 1505—1508; cf. B., 1930, 351).—Theoretical. The case of material streaming through a tube and having a layer showing modified consistency constants near the wall of the tube is discussed.

L. S. THEOBALD.

Two new ultrafiltration funnels for rapid filtration. S. R. ZINZADZE (Kolloid-Z., 1930, 51, 164—165).—Two ultrafilters, one for quantitative and the other for qualitative work, and particularly rapid in action, are described. They are also applicable to electro-ultrafiltration.

E. S. HEDGES.

Mercury volumeter. J. F. McMAHON (J. Amer. Ceram. Soc., 1930, 13, 363—367).—The Goodner mercury volumeter (*ibid.*, 4, 288) has been improved by the addition of a counterbalance, a light signal, and a dipping device. The apparatus is fully described and illustrated. The advantages claimed are more speedy manipulation, no string marks, cleaner mercury, and better suspension of the briquettes in mercury.

R. J. CARTLIDGE.

Many-sided illumination in the dark field. C. SPIERER (Kolloid-Z., 1930, 51, 162—163).—A special immersion objective lens and dark field condenser are described.

E. S. HEDGES.

New consistometer and its application to greases and oils at low temperatures. R. BULKLEY and F. G. BITNER (Bur. Stand. J. Res., 1930, 5, 83—96).—See B., 1930, 674.

PATENTS.

Gas-fired furnace. W. A. MORTON (B.P. 331,315, 3.5.29).—In a reverberatory or similar furnace the flues for gas, air, and waste gases are all at one end of the furnace, and are separated by relatively thin walls, so that some preheating of both air and gas is effected therein; the air is further preheated in a recuperator.

B. M. VENABLES.

Tunnel kilns. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 331,109, 8.7.29).—In a tunnel kiln having a travelling platform vertical rollers or other squeezing means are provided at the point where the platform enters or re-enters the kiln to press back any loose material projecting beyond the normal width of the platform.

B. M. VENABLES.

Feeding finely-divided solid material into high-pressure vessels. F. B. GRANT, H. HARPER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,988, 22.3.29).—The material is conveyed in a pocket formed in the side of a ram which passes through a gland longer than the pocket. The ram may be rotated through 180° after entering the vessel so that the material drops out by gravity, but a blast of gas under a pressure greater than that in the vessel is also supplied through a borehole in the ram to clear out the pocket.

B. M. VENABLES.

[A] Method and apparatus for cooling air. [B] Air-treating devices. H. A. GILL. From COOLING & AIR CONDITIONING CORP. (B.P. 331,218—9, 25.3.29).—(A) Two sets of water sprays and a refrigerating system comprising condenser, evaporative cooler, and compressor are provided. The water from the first set of sprays passes through the condenser and thence to waste; the water of the second set of sprays passes in a closed circuit through them and the cooler. (B) Centrifugal spraying devices for atomising scrubbing liquid are described. [Stat. ref.]

B. M. VENABLES.

Heat-exchanging apparatus. M. PAYNE (B.P. 331,392, 4.7.29).—Discs of sheet metal slit radially or strip slit longitudinally are used in the construction of gills for attachment to heat-exchanging tubes.

B. M. VENABLES.

Heat-exchange apparatus. G. W. WATTS, Assr. to STANDARD OIL Co. (U.S.P. 1,741,043, 24.12.29. Appl., 27.7.25).—Gas is removed from a space in a heat exchanger, which should contain liquid only, by means of a Venturi throat inserted in the outlet pipe for the liquid. A side entry to the throat is connected by a small pipe to the place in the exchanger where the gas is most likely to collect.

B. M. VENABLES.

Temperature-regulating apparatus. L. D. WEILL (B.P. 299,714, 29.10.28. Belg., 28.10.27).—The apparatus comprises a heat-sensitive device (thermometer)

actuating a primary relay, a secondary relay with adjustable timing attachment, and a power switch for the heaters. When the secondary relay is operated (through the primary) by a rise of temperature it cuts off or reduces the heat supply, which remains reduced for a definite period of time, at the end of which the timer restores the heating current; if meanwhile the temperature has not fallen, the secondary relay again operates and reduces the current for another definite period, and so on until the temperature has fallen below a fixed limit, when all parts return to normal with heating current on. The device is stated to reduce the lag between the fall of temperature of the thermometer and that of the furnace.

B. M. VENABLES.

Roller and other grinding mills. BRINJES & GOODWIN, LTD., C. F. N. SEAMAN, and F. ATHERTON (B.P. 330,923, 18.3.29).—In a grinding mill for paint etc. the rollers or discs are adjusted to a definite clearance by the rotation of cams by hand levers which are provided with a quick release. The grinding elements are held together by springs which are strong enough to provide pressure when necessary to grind occasional hard lumps.

B. M. VENABLES.

Grinding mills. H. DRYSDALE (B.P. 330,989, 23.3.29).—A pair of rubbing convex and concave surfaces are given relative rotation about axes that form an angle with each other. One surface, preferably the convex and larger one, is rotated about a horizontal axis, and the axis of the other is so inclined that its periphery coincides (or nearly so) with the periphery of the first at one point. The inclined member may be stationary and the machine may be made in double form, having pairs of members both sides of a driving means.

B. M. VENABLES.

Grinding and pulverising machines. V. BOOTH (B.P. 331,384, 26.6.29).—A disc grinder with one fixed and one rotating disc is provided with a circumferential screen between which and the edges of the discs are paddles attached to the rotating disc which work the undersize through the screen and deflect the oversize through ports formed in an end wall, preferably back into the feed hopper which communicates with the centre of the discs.

B. M. VENABLES.

Evaporation of liquids. E.M.S. INDUSTRIAL PROCESSES, LTD., R. A. STOKES, and E. G. L. ROBERTS (B.P. 330,931, 20.12.28).—An evaporator or crystalliser is formed from a number of troughs abreast through which the material flows transversely. Paddles are provided for stirring and to assist the solid matter over the ridges.

B. M. VENABLES.

Desuperheating of steam. BRIT. ARCA REGULATORS, LTD., and T. LINDSAY (B.P. 331,361, 12.6.29).—Water is heated by indirect interchange with either the superheated steam before or the desuperheated steam after passing through the apparatus, and when thus raised nearly to its b.p. this water is sprayed into the steam.

B. M. VENABLES.

Cooling towers. FILM COOLING TOWERS (1925), LTD., and W. A. SCOTT (B.P. 331,011, 10.4.29).—The lower ends of wooden masts of cooling towers which are subject to rapid rotting are formed of material

impervious to water, such as concrete, and are spliced to the upper parts.

B. M. VENABLES.

Manipulation of liquids. P. CHARMAT (B.P. 309,541, 3.4.29. Fr., 12.4.23).—Liquids such as champagne, beers, etc. which are saturated with gas under pressure and have, e.g., to be filtered without reduction of pressure and without contact with air, are stored in a container with a flexible diaphragm or balloon to each side of which separate inlet-outlet pipes are connected. Except for a gas space to allow for temperature changes, the vessel is always kept full, the liquid being withdrawn from one side of the diaphragm, passed through the treatment plant, and returned to the other side of the diaphragm, which is sufficiently extensible for the total volume of the liquid to be stored on either side of it.

B. M. VENABLES.

Centrifugal separators. H. W. FAWCETT (B.P. 330,958, 3.1.29).—A centrifugal separator for treating, e.g., wool-washing liquor is provided with peripheral outlets for sludge which may be screened by cross-wires, and adjacent to them are interior jets of an auxiliary liquid, preferably valueless water, which are supplied through separate conduits from an axial inlet and small bowl. Provision is made for dividing the bowl for ultimate cleaning, packed joints being provided in the auxiliary conduits.

B. M. VENABLES.

Electrically-driven centrifugal apparatus. Soc. ANON. CONSTRUCTIONS ELECTRIQUES PATAY (B.P. [A] 270,723, 4.5.27. Fr., 7.5.26. [B] Addn. B.P. 330,836, 11.9.29. Fr., 4.5.29).—A motor drives a centrifuge through a spring coupling; in (B) means are provided for regulating the tension of the spring.

B. M. VENABLES.

Apparatus for catalytic transformation of gases. Soc. ANON. D'OUGRÉE-MARIHAYE (B.P. 309,169, 19.3.29. Belg., 7.4.28).—An apparatus for the oxidation of ammonia or other catalytic reactions comprises a heat interchanger, a catalyst chamber, and a dust collector for saving particles of catalyst; an adjustable proportion of the hot outgoing gases is finally passed through the heat interchanger.

B. M. VENABLES.

Air-purifying apparatus. MINE SAFETY APPLIANCES Co., Assees. of A. L. DOTTER (B.P. 307,428, 6.3.29. U.S., 7.3.28).—Untreated sawdust or other subdivided cellulosic material of large surface is used in a respirator.

B. M. VENABLES.

Removal of soot and other solid particles from smoke and fumes. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. R. WHITNEY (B.P. 307,805, 13.3.29. U.S., 13.3.28).—Acetone is added to the water that is used to remove soot either by direct sprays or from electrodes. Electrostatic apparatus is described.

B. M. VENABLES.

Instrument for indicating or recording sp. gr. of gases. W. WILSON (B.P. 331,112, 10.7.29).—The gas is admitted at a constant volume-rate and pressure to a floating bell, the outlet from which comprises a fixed orifice obstructed by a taper needle which rises and falls with the bell. The bell is counterpoised by a device that compensates for the varying depth of immersion of the bell so that the internal pressure is maintained

constant, and, since a heavier gas will need a larger orifice to discharge it, the bell rises with increase of density and operates a pointer moving over a scale.

B. M. VENABLES.

Lubrication of bearings and lubricants therefor.

W. B. D. PENNIMAN (B.P. 306,532. 25.1.29. U.S., 23.2.28).—Oil of low viscosity, about 50 Saybolt instead of the usual 250, is preferably used, and about 10% of lard, palm, or castor oil, or metallic soap, or oils treated with sulphur or oxygen, is added.

B. M. VENABLES.

Heat transferrer. R. UHDE, Assr. to HEISSDAMPF-GES.M.B.H. (U.S.P. 1,768,222, 24.6.30. Appl., 19.12.28. Ger., 29.12.27).—See B.P. 303,172; B., 1930, 124.

Drying of moist material. O. SÖDERLUND and T. GRAM, Assrs. to TECHNOCHEMICAL LABS., LTD. (U.S.P. 1,766,843, 24.6.30. Appl., 11.10.29. U.K., 7.10.26).—See B.P. 286,743; B., 1928, 352.

Mixing or stirring machine. E. CHRISTIANSEN (U.S.P. 1,767,346, 24.6.30. Appl., 24.9.29. U.K., 10.1.29).—See B.P. 327,465; B., 1930, 537.

Atomiser for liquids. R. ELSÄSSER, Assr. to SIEMENS-SCHUCKERTWERKE A.-G. (U.S.P. 1,764,437, 17.6.30. Appl., 8.3.28. Ger., 15.3.27).—See B.P. 287,105; B., 1928, 658.

Securing temporary intimate contact between immiscible liquids [without emulsification]. L. D. JONES, Assr. to SHARPLES SPECIALTY Co. (Re-issue 17,719, 1.7.30, of U.S.P. 1,575,116, 2.3.26).—See B., 1926, 397.

Filter for liquids under pressure. G. JAVITCH (U.S.P. 1,764,567, 17.6.30. Appl., 4.4.29. Fr., 5.4.28).—See B.P. 309,143; B., 1930, 126.

Cleaning of filters. E. FOLTA (U.S.P. 1,768,314, 24.6.30. Appl., 21.3.29. Czechoslov., 2.4.28).—See B.P. 308,974; B., 1930, 126.

Apparatus for continuous distillation of difficultly distillable liquids. H. A. VON STADEN, A. BERENBRUCH, and A. HÖHN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,699, 24.6.30. Appl., 28.9.27. Ger., 11.9.26).—See B.P. 288,775; B., 1928, 431.

Recovery and use of heat from furnace gases of variable temperature. SUPERHEATER Co., LTD. From COMP. DES SURCHAUFFEURS (B.P. 332,274, 16.4.29).

[Firebridge construction for] consuming smoke in boiler and like furnaces. J. NEWNES (B.P. 332,023, 14.6.29).

Heat-exchange apparatus. A.-G. BROWN, BOVERI & Co. (B.P. 310,327, 16.4.29. Ger., 23.4.28).

Heat-exchanging apparatus. [Air and gas heaters.] W. E. KOCHS & Co., LTD. From H. FÖGE (B.P. 332,280, 17.4.29).

[Gilled] heat-exchanging apparatus. H. JUNKERS (B.P. 332,455, 16.9.29. Ger., 26.10.28).

Heat-exchanging device [for radiators etc.]. J. M. HARRISON (B.P. 331,982, 13.5.29).

[Corrugated] refractory brick for heat-exchange apparatus. J. CHATWIN. From VEREIN. STAHLWERKE A.-G. (B.P. 332,092, 9.8.29).

Absorption cooling devices. SCHWARZWALDWERKE LANZ G.M.B.H. (B.P. 332,160, 2.11.29. Ger., 30.11.28).

Device for regulating amount of flow or rate of feed of powdered, granular, or lump material to crushing or pulverising mills etc. BRIT. "REMA" MANUF. Co., LTD., and P. HOWDEN (B.P. 332,382, 3.7.29).

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKT. (B.P. 311,273, 23.4.29. Swed., 8.5.28).

Refrigerators and refrigerating apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. STEENSTRUP (B.P. 318,932, 12.9.29. U.S., 12.9.28).

Refrigerating apparatus. KELVINATOR CORP., Assees. of F. R. ERBACH (B.P. 310,464, 6.3.29. U.S., 26.4.28).

[Reinforced concrete] construction of water-cooling towers. L. G. MOUCHEL & PARTNERS, LTD., and A. T. J. GUERITTE (B.P. 332,268, 18.3.29).

[Coupling for] centrifugal machines. S. K. D. M. VAN LIER (B.P. 331,323, 8.5.29. Holl., 12.3.29).

Gas washer (U.S.P. 1,752,045).—See II. Kilns (B.P. 331,224—5).—See VIII. Gas purification (B.P. 309,149 and U.S.P. 1,752,920). **Electrical precipitation plants** (B.P. 331,699).—See XI. **Drying of grass** (B.P. 327,409).—See XVI. **Boiler feed water** (B.P. 331,778).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Chemistry of coal. VI. Benzenoid constitution as shown by oxidation with alkaline permanganate. W. A. BONE, L. HORTON, and S. G. WARD (Proc. Roy. Soc., 1930, A, 127, 480—510; cf. Bone and Quarendon, B., 1926, 305).—A detailed examination has been made of the alkaline permanganate oxidation of the residues from the benzene-pressure extraction of five typical coals of widely different geological ages and maturities. It is found that, under suitable conditions of concentration and temperature, the entire coal substance in any case may be oxidised so that the whole of its carbon is transformed into a mixture of carbonic, acetic, oxalic, succinic, and benzenecarboxylic acids, the first and last always being the main products; and from the fact that the proportions of the benzenecarboxylic acids do not seem to vary materially from one coal to another, it would appear that the maturing process, whatever its chemical nature may have been, has not destroyed the essential chemical structure of the coal substance. A detailed investigation of the complex mixture of acids produced by the oxidation was carried out in the case of three of the coals. Under suitable conditions, 100 pts. of the original coal substance yield about 3—7 pts. of acetic acid, 15—35 (usually about 20) pts. of oxalic acid, and 33—50 pts. of benzenecarboxylic acids of mean composition closely approximating to that of a benzenetricarboxylic acid. From this mixture of benzenoid acids, all except two of the twelve possible benzenecarboxylic acids have been isolated, including the three phthalic, the three benzenetricarboxylic acids, two out of the three possible tetracarboxylic acids, and

the penta- and hexa-carboxylic acids. Moreover, in the cases of Morwell and Busty coals, complete "carbon balances" worked out for the oxidation under optimum conditions showed a very similar distribution of the original carbon in the coal substance among the various products, viz., 42% as carbon dioxide, 2% as acetic acid, 7% as oxalic acid, and 48% as benzenecarboxylic acids. In each case nearly one third of the original carbon appeared as C₆-rings in the oxidation products. A study of the "stepwise" alkaline permanganate oxidation of the coal substance led to the conclusion that complex colloidal "humic" acids are the initial oxidation products, the simpler crystalline benzenoid and oxalic acids arising subsequently from their further oxidation. This was confirmed by experiments showing that on separate oxidation, 100 pts. of these humic acids yield about 60 pts. of a mixture of benzenecarboxylic acids, 20 pts. of oxalic acid, 3 pts. of acetic acid, and 1 pt. of succinic acid. The transition from the humic to the benzenecarboxylic acids is probably not direct, but occurs through intermediate crystalline acids more complex than the latter. Since the benzene-pressure-extracted "residues" constitute 85–98.5% of the original coal substance, and about one third of the carbon in such residues appears as C₆-rings in the oxidation products, it is inferred that a considerable part of the original coal substance is of a benzenoid character. Also 57–67% of the benzene-pressure extracts from Morwell brown coal and the Estevan lignite is composed of phenols and phenolic esters, and the constituents which are mainly responsible for the "coking propensities" of bituminous coals are essentially benzenoid in character. Such results suggest that the coal substance may have arisen through condensations of phenolic and amino-compounds with aldehydic compounds. Preliminary experiments on the effects of progressive carbonisation of the coal substance on the relative yields of the oxidation products indicate that, with Estevan and Busty coal residues, the yields of benzenoid acids increase with carbonisation up to 500–600°, after which they diminish; considerable yields of benzenoid acids are, however, obtained even with material that has been fully carbonised at 1000°.

L. L. BIRCUMSHAW.

Chlorine content of coal and its distribution in the products of carbonisation. W. A. DAMON (66th Ann. Rep. Alkali etc. Works, 1929, 21–28).—A series of analyses show the water-soluble chlorine content of different coals to range from 0.0029% to 0.355%. It tends to increase with fineness of division of the coal. The greater part of this reappears in the tar and first liquor condensed, and at one coke-oven works ammonium chloride (3 lb. per ton of coal) is manufactured by a system of spraying the gas. In gas works the bulk of the ammonium chloride is also in the first liquor condensed in the hydraulic main; if such liquor is evaporated the ammonium chloride produced contains tar acids and darkens on exposure to light. These impurities can be removed by agitating the liquor with lead carbonate. The chlorine content of tar is found to be in proportion to that of liquor emulsified with it, but once it has been deposited in the tar in a solid state it is very difficult to remove by washing. The ammonium chloride content of tar may be deter-

mined by adding to 20 g. of tar 2–3 g. of sodium stearate; 2 g. of lime and 3 g. of precipitated chalk are then added and the whole is charred over a small flame, powdered, and extracted with water. The aqueous extract is neutralised with nitric acid, boiled with 10 c.c. of hydrogen peroxide, and the chlorine content determined by titration.

C. IRWIN.

Mineral constituents of coal and their influence on the determination of ash. F. SCHUSTER (Brennstoff-Chem., 1930, 11, 237–239).—Determinations of the carbonate-carbon dioxide and the ash from nine gas coals have been made. The alkalinity of the ash, determined by titration, was considerably less in each case than corresponded with the amount of calcium oxide calculated from the carbonate content of the coal. Since sulphur dioxide may be absorbed from the combustion of the coal itself and also from the heating gases of the muffle, the furnace should be so designed that contact of sulphur-containing gases with the ash is avoided, and should preferably be electrically heated.

A. B. MANNING.

Sulphate content of coal ash. F. SCHUSTER (Brennstoff-Chem., 1930, 11, 262–263; cf. preceding abstract).—By burning coals in an atmosphere free from sulphur compounds it has been shown that the sulphate content of the ash is derived principally from the sulphur present in the coal in other forms, e.g., as pyrites. The original sulphate content of the coals investigated was negligible. The following formula is suggested for calculating the true percentage of mineral constituents from the ash: mineral constituents = ash + CO₂ (as carbonate) + (0.335 × FeS₂) + SO₃ (in the ash), all quantities being expressed as percentage of the coal. It is assumed that the loss of water of hydration from the ash is compensated, within the limits of error, by the gain due to oxidation of ferrous to ferric oxide. When coke is burned the mineral constituents increase in weight due to oxidation of the iron and to the formation of sulphate.

A. B. MANNING.

Apparatus for determining tendency of coal and other materials to self-ignition. D. J. W. KREULEN (Brennstoff-Chem., 1930, 11, 261–262).—The apparatus is constructed of an aluminium block, 80 mm. high and 50 mm. in diam., having a central boring 20 mm. in diam. for the sample of coal, which rests therein on a copper gauze support. A current of oxygen is passed through a channel bored in the block, down through the coal, and thence to an outlet. The block is heated by a burner, and temperature measurements are made on two thermometers, one in the coal and the other in a special boring in the block. The coal is prepared by briquetting the finely-powdered average sample and carefully breaking the briquettes down to 10–20-mesh (per cm.) size.

A. B. MANNING.

Classification of Canadian coals. E. STANSFIELD and J. W. SUTHERLAND (Trans. Canad. Inst. Min. Met., 1929, 32, 360–388).—Various systems of coal classification are discussed.

C. W. GIBBY.

Constant factors for the calculation of the calorific value of Cape Breton coals from proximate analysis data. J. L. BOWLBY (Trans. Canad. Inst. Min. Met., 1929, 32, 469–490).—A comparison of

various formulæ. The expression preferred is $Vx + 145.5C + 40.5S = B.Th.U.$ per lb., where V is the volatile matter, C the fixed carbon and S the sulphur percentage, and x is an arbitrary constant depending on the colliery. C. W. GIBBY.

Steaming of horizontal chamber ovens. G. M. GILL (Gas World, 1930, 93, 99—101).—The possibility of adding steam to charges in horizontal chamber ovens increases the flexibility of this type of plant. Modifications to a setting for this purpose involved passing steam into each oven at eight points near the base, where special moulded bricks were built in to the recuperators to form a number of channels. Steam at the rate of 14 lb./min. was admitted during the first 20 hrs. and 220 lb./min. during the last 4 hrs. of carbonisation; the therm yield per ton of coal was considerably increased and further advantages are anticipated from improved design. R. H. GRIFFITH.

Origin and decomposition of carbon disulphide in gas making. III. Chemical and thermodynamic effects in formation of organic sulphur compounds. W. J. HUFF and J. C. HOLTZ (Ind. Eng. Chem., 1930, 22, 639—645; cf. B., 1928, 77).—The formation of organic sulphur compounds by the action of heated carbon (from sucrose) on hydrogen sulphide in low concentration in different gases has been investigated. The formation of carbon disulphide is always accompanied by that of important quantities of other sulphur compounds of carbon, possibly the monosulphide when the gas stream consists mainly of nitrogen and also the oxysulphide when it contains water vapour, the oxides of carbon, or oxygen. The formation of sulphur-carbon compounds increases with an increase in the concentration of hydrogen sulphide or with a rise in temperature, but is inhibited by hydrogen. The data support Lewis and Lacey's criticisms (A., 1915, ii, 767) of Koref's conclusions (A., 1910, ii, 289). The addition of copper, iron, or zinc in the form of oleates to a sulphur-bearing oil before cracking decreases the amounts of hydrogen sulphide and other gaseous sulphur compounds which are formed. L. S. THEOBALD.

Carbonising and briquetting Saskatchewan lignite. W. G. HEPTINSTALL (Trans. Canad. Inst. Min. Met., 1929, 32, 395—404).—A detailed description of the use of the Lurgi process for lignite containing 30% of moisture. C. W. GIBBY.

Dehydration of gas. W. S. DOLE (Gas J., 1930, 191, 258—260).—The performance of compression plant for dehydration of coal gas or of oil gas is described for two small American works. Resulting diminution in blockage complaints and, presumably, greater life of mains and fittings contribute to balance the additional running costs of about 1½d. per 1000 cub. ft. R. H. GRIFFITH.

Conversion of methane or coke-oven gas into acetylene by the electric discharge. K. PETERS and A. PRANSCHKE (Brennstoff-Chem., 1930, 11, 239—247; cf. B., 1929, 703).—Improved forms of discharge tube suitable for the continuous treatment of larger quantities of gas are described. With increased rates of passage the consumption of electrical energy falls to a minimum of about 12 kw.-hrs./m.³ of acetylene

produced, for rates of passage of 1000—1200 litres/hr. About 38% of the electrical energy expended is then utilised in the transformation of methane into acetylene. The percentage of acetylene in the product passes through a maximum as the rate of passage is increased; at the higher rates corresponding with the minimum energy consumption it varies from about 4% to 8% as the methane content of the original gas increases from 25% to 95%. The energy consumed by the pump maintaining the necessary low pressure amounts to 2—3 kw.-hrs./m.³ of acetylene produced. A. B. MANNING.

Adsorption of benzol vapour by mixed adsorbents. J. K. CHOWDHURY and H. N. PAL (J. Indian Chem. Soc., 1930, 7, 451—464).—Activated bauxite containing varying percentages of ferric oxide shows a slight increase in adsorptive capacity, whilst bauxite on which silica gel has been precipitated shows a large increase (from 8% to 38%). The adsorptive power is unaffected in presence of hydrogen sulphide, carbon monoxide, ethylene, or small amounts of water vapour; the presence of tarry matter does not interfere with regeneration of the adsorbent by roasting. It is suggested that "silicalised bauxite" should prove a suitable substance for recovery of benzol from coal gas. J. R. I. HEPBURN.

Viscosity-temperature diagram [of mineral oils]. C. WALTHER (Petroleum, 1930, 26, 755—757).—The diagram described is obtained from the equation $\log V_k = -m(\log T - 2.309) + \log \log V_{k50^\circ}$, where V_k denotes the kinematic viscosity, m is a characteristic temperature factor for each oil, and T is the absolute temperature. A diagram is constructed in which $\log V_k$, $\log T$, V_k , and $^\circ E$ are represented on the four sides of a rectangle. The value of M ($\log \log V_k$ between 0° and 100°) for the oil can be found from the diagram. It can also be applied to determine the viscosity of an oil at a third temperature when the viscosities at two temperatures are known. A similar equation is derived, by means of which the viscosity curves of mixtures of two or more oils may be found. W. S. E. CLARKE.

Cracking processes in the Russian petroleum industry. L. SINGER (Petroleum, 1929, 25, 893—906; 1930, 26, 482—491).—Three types of cracking processes are described which serve for the production of (i) fuel oil of a better quality as regards η and cold test, cracking being carried out at 425° and under 10—60 atm.; (ii) benzine and fuel oil, and (iii) benzine and coke, in which treatment is carried out at 450° and 20 atm. These processes were utilised in the cracking of Grosni and Surakhani mazouts, and of various heavy crude oils. The results obtained with Grosni mazout show that gentle cracking (first method) causes a decrease in the cold test of the fuel oil obtained. In the second method, with a 1% yield of coke, 35% of cracked benzine is obtained; the quantity of coke formed is independent of the temperature, but is constant with equal benzine yields. With 15% of coke in the third method 57% of cracked benzine is produced. The following conclusions have been reached: (a) Greater yields of light products cause an excessive formation of coke and deteriorate the quality of the residue. (b) The velocity of the

reaction is doubled for each rise in temperature of 10°. (c) The rate of coke formation depends on the chemical composition of the cracked petroleum products. Paraffinic products yield less coke than paraffin-free or asphaltic products. Kerosene yields practically no coke, solar and spindle oils very little, machine and cylinder oils large quantities, and tars greatly increase the formation of coke. (d) Increase of pressure causes a decrease in the content of unsaturated hydrocarbons, paraffin hydrocarbons yield no coke on cracking, whilst aromatic hydrocarbons tend to give significant quantities. (e) The extent of the production of cracked benzines and kerosenes from heavy crude oil and mazout is practically the same. (f) The details of a cracking process are determined not so much by the yields of cracked benzines (which are the same for a given temperature and cracking time with heavy crude oils and mazout) as by the yields of coke, which vary for different petroleum products. The cracking of Grosni oil tar and paraffin tar is not important, on account of excessive quantities of coke formed.

W. S. E. CLARKE.

Efficiency in the distillation of light oils from crude oil. A. A. ASHWORTH (J. Inst. Petroleum Tech., 1930, 16, 40—48).—The efficiency of a refinery is arrived at by means of a system of distillation (based on a number of specified temperatures) in which a series of definite cuts is obtained which are subsequently combined, chiefly arithmetically, so as to give the maximum value of the products obtained from the crude oil. The points finally selected at which cuts should be made are the following: (1) initial b.p. of the crude to 97°, (2) 97—136°, (3) 136—175°, (4) 175—200°, (5) 200—280°, or to the point necessary to give a limiting sp. gr. of 0.825 for the kerosene, (6) from the end of cut (5) to 300°. A special fractionating apparatus was used in which the reflux ratio was controlled by varying the insulation and thence the cooling of the column. Crude oil (100 g.) was added to the flask, distillation commenced without any insulation on the column and slowed down to about 2 drops/sec. within 5° of the first cut point until the temperature was reached; the receiver was then changed. The weight, volume, and sp. gr. of the cut were measured. When cut (2) had distilled, so much of it was blended with (1) as was necessary to make a light benzine which would distil 60% up to 100° in an Engler flask. Generally 52% of (1) and 48% of (2) were required. The distillation was continued, the remainder of cut (2) was blended adequately with cut (3), the remainder of (3) with (4), etc., so as to obtain the specified products. Insulation was necessary for the last two cuts. Specifications of standard products as denoted by the cuts are given, i.e., light and heavy benzines, white spirit No. 1, kerosene, and gas oil. Special products, e.g., aviation benzine, were produced from the light benzine by removing part of cut (2); medium heavy benzine was a mixture of light and heavy benzines, white spirit No. 2, and kerosene distillate. For refinery plant a pipe-still was used in which all light oils were vaporised prior to fractionation; such a still gave an efficiency of 97—98% in the light-oil distillation.

W. S. E. CLARKE.

Synthetic fuels and lubricating oils. A. W. NASH (J. Inst. Fuel, 1930, 3, 347—353).—The sources and methods available for the production of synthetic fuels and lubricating oils are discussed. The light-oil fractions of low-temperature carbonisation tar could be treated to give a motor fuel with valuable "anti-knock" properties. A lubricating oil has been prepared by low-temperature distillation of Shirebrook bastard canal coal. The oil possesses lubricating properties comparable with mineral oils. The lighter fractions of the oil from the Bergius hydrogenation process for coal can yield valuable motor spirits which will probably contain "anti-knock" substances. The lubricating oils from the Bergius process will need to be freed from phenol. By the use of catalysts in berginisation the I. G. Farbenindustrie claim to obtain greater flexibility. Recently it has been shown that oxides of zinc, nickel, cobalt, and copper may be used in place of iron oxide. Zinc oxide increases the oil yield. Alkaline-earth chlorides and aluminium chloride have been shown to extend the scope of high-pressure hydrogenation. The reduction of carbon monoxide by hydrogen at atmospheric and high pressures with or without catalyst is also a possible source of fuel. The cracking of natural gas and the use of gaseous fuel are also discussed.

H. E. BLAYDEN.

Bleaching and desulphurisation of mineral oils with silica gel and bleaching earths. E. BOSSHARD and W. WILDI (Helv. Chim. Acta, 1930, 13, 572—586; cf. A., 1929, 256).—The bleaching properties of various bleaching earths and silica gels have been studied with mineral lubricating oils, and the gels have been found inferior to the best earths. Conversely the gels are more effective than the natural earths in desulphurising petroleum solutions of amyl sulphide and its homologues. When wetted with alcohol the various earths produce widely differing amounts of heat, which are proportional to the capacity for bleaching and desulphurisation, and are due to the unequal amounts of active surface in the different specimens. The method of preparation of silica gels influences their activity. Those made from concentrated solutions show increased bleaching capacity (adsorption of colloids) but diminished capacity for desulphurisation (adsorption of crystalloids). Gels with fine pores are the best sulphide adsorbents. Adsorption of ethyl, propyl, butyl, and amyl sulphides on prepared silica gels shows that the substance with the lowest mol. wt. is most easily taken up; the heats of wetting decrease in the same order. The bleaching and desulphurisation of a crude petroleum with silica gels of different pore sizes bears out the above results.

F. L. USHER.

Physical properties and constitution of mineral lubricating oils. W. BIELENBERG (Z. physikal. Chem., 1930, 149, 42—50).—A comprehensive examination of refractivity data for certain homologous series of hydrocarbons has shown that the refractivity-mol. wt. graphs differ considerably in form from those given by Kyropoulos (B., 1929, 931). This author's deductions from these curves in connexion with the determination of the constitution of certain mineral oils are therefore invalidated.

R. CUTHILL.

Relationship between calorific value and other characteristics of residual fuel oils and cracked residuums. W. F. FARAGHER, J. C. MORRELL, and J. L. ESSEX (Ind. Eng. Chem., 1929, 21, 933—941).—Experimental results establish that the relationship between the calorific value in B.Th.U./lb. and the A.P.I. gravity of straight-run fuel oil and cracked residuum is linear. New formulæ for calculating the calorific value per lb. of dry oil were determined as follows: $17,010 + (90 \times \text{°A.P.I.})$ for straight-run fuel oils and $17,645 + (54 \times \text{°A.P.I.})$ for cracked residuums, whether of the normal or flashed type. The calculated values are accurate to within 30 B.Th.U. of the values obtained by precision calorimetric methods. Combustion analyses show an average C:H ratio of 8.66 for the normal residuum, 9.22 for flashed residuum, and 7.08 for straight-run fuel oils. The calorific value calculated from the Dulong formula gives higher results than those determined experimentally for straight-run fuel oils and residuum. The net calorific values calculated for several straight-run fuel oils and the two residuums showed about 1200 B.Th.U./lb. less than the observed or gross calorific values as the result of the correction for the latent heat of vaporisation of water. The source of the oil, the amount of coke formed by Engler distillation, the volatility, the benzene-soluble material, etc. do not appear to affect the calorific value so long as the A.P.I. gravity is not altered. The benzene-soluble material separated from a flashed or a normal residuum contains organic matter of which 40—45% is soluble in such organic solvents as chloroform, carbon tetrachloride, carbon disulphide, aniline, etc. The dissolved matter has the same C/H ratio as the residuum itself; in the insoluble matter the ratio is about 18 and the ash and sulphur contents are high. The benzene-soluble material has a composition corresponding to 1.3% of petroleues, 98.7% of asphaltenes (contaminated with inorganic matter), 10.0% of carbenes, 33.8% of asphaltenes insoluble in carbon tetrachloride, 42.6% of carbonaceous material insoluble in carbon disulphide and combustible in oxygen at 700°, and 12.3% of insoluble and non-combustible matter.

H. S. GARLICK.

Cracking process without coke formation. M. NAPHTALI (Brennstoff-Chem., 1930, 11, 247—249).—H. Wolf's "Carburol" process is briefly described. The oil is heated under pressure in the cracking coil for a definitely controlled time. The cracked oil passes thence through a specially constructed expansion valve, and is immediately mixed with fresh oil or cooled distillate. In this way the cracking process is stopped just before coke formation begins. A continuous turbulent flow of oil is maintained through the cracking coil, the heating of which is carefully controlled to avoid overheating of the oil at any point. The chamber into which the oils pass acts as a dephlegmator, wherein they are distilled, the lighter and intermediate fractions passing through a second dephlegmator to the condensers, while the heavy residual oil is discharged to a tank. This oil may be used as fuel oil or for the production of road material. The intermediate fractions may be returned for further treatment. Some results of tests on a cracking plant of this type are tabulated. No formation of coke

was observed in the cracking coil, valve, or the vessel wherein the residual oil is separated. A. B. MANNING.

Cracking of saturated gaseous hydrocarbons. W. von PIOTROWSKI and J. WINKLER (Petroleum, 1930, 26, 763—780).—An investigation of the pyrolysis of the propane-butane fractions of gas oil, of degasolined Boryslaw natural gas, and of the gas from a Cross high-pressure distillation plant in a specially constructed oven made of chrome-nickel steel shows that hydrocarbons with 2 to 5 carbon atoms can be decomposed under optimum temperature and time conditions so as to give the maximum yields of unsaturated hydrocarbons. The optimum temperature and heating periods for a pure propane-butane mixture (so-called gas oil) are 730° and 8 sec., for Cross cracked gas containing methane and ethane 740—760° and 10 sec., and for a Boryslaw natural gas containing only a few of the higher homologues 780° and 11 sec. The reacting gases show a tendency, on remaining in contact with the separated carbon, to extensive decomposition and further deposition of carbon. Cracking temperatures above 800° (e.g., 870°) tend to yield aromatic hydrocarbons (up to 22%, calc. on the gas oil). Butadiene is found amongst the products of decomposition from gas oil at about 700°, accompanied above 800° by a little acetylene.

W. S. E. CLARKE.

Separation of bitumen from Alberta bituminous sands. K. A. CLARK (Trans. Canad. Inst. Min. Met., 1929, 32, 344—359).—Separation may be effected by the use of hot water, after first treating the dried sand with calcium oleate and water. The efficiency is improved by preliminary mixing of the sand with dilute sodium silicate solution and heating to 85° before washing. All reagents which favour the formation of oil-in-water emulsions have a similar effect. The emulsion is unstable, and the bitumen rises to the surface as a froth. The silt can be coagulated by the addition of calcium chloride equivalent in amount to the sodium silicate. The separated bitumen may be dehydrated by breaking the emulsion with phenol and adding enough salt to increase the density of the aqueous layer so that the bitumen rises to the surface.

C. W. GIBBY.

Oleum deelinæ [dee oil]. W. KESSLER (Pharm. Ztg., 1930, 75, 808).—The preparation is merely a highly purified and very viscous yellow vaseline oil.

S. I. LEVY.

Steel mains and corrosion. JEAVONS and PINNOCK.—See X. **Brown coal and crop growth.** KISSEL.—See XVI.

PATENTS.

Fuel composition. A. MONTELS (B.P. 329,924, 30.8.29).—A solid fuel is formed by briquetting a mixture of sawdust or tannin dust, crude petroleum, tar residues, and a concentrated solution of glue.

A. B. MANNING.

Briquette. R. T. GOODWIN, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,749,378, 4.3.30. Appl., 18.2.26).—Coal is briquetted by means of the material precipitated by treating the heavy oil residues from cracking processes etc. with dilute acids (cf. U.S.P. 1,660,295; B., 1928, 326). These materials have

fusing pt. between 160° and 230°, and are 15–30% soluble in carbon disulphide. A. B. MANNING.

Production of carbon granules. STANDARD TELEPHONES & CABLES, LTD. From WESTERN ELECTRIC Co., INC. (B.P. 329,652, 21.2.29).—A mass of carbonisable material is broken up into particles which assume a spherical shape due to surface tension, and these are then hardened and carbonised. The material may consist of cellulose dissolved in zinc chloride solution, the hardening being effected by immersion in alcohol. Fusible phenolic condensation products or artificial resins form other suitable initial materials; they are granulated and melted, the molten particles allowed to assume the spherical shape, hardened by being slowly heated to about 270°, and then carbonised. The granular carbon produced is suitable for use in variable-resistance units for telephone transmitters etc.

A. B. MANNING.

Manufacture of absorbent carbonaceous material. O. L. BARNEBEY and M. B. CHENEY (U.S.P. 1,751,612, 25.3.30. Appl., 19.5.21; cf. U.S.P. 1,541,099; B., 1925, 581).—Carbonaceous material, *e.g.*, coconut shells, rice husks, wood, etc., is incompletely charred at 350–500° to form a preliminary product containing preferably 20–40% of tarry matter, and this is treated at 500–900° with an oxygenated gas, *e.g.*, carbon dioxide or steam, to complete the carbonisation and remove all tarry matter. A. B. MANNING.

Manufacture of decolorising or activated carbon. T. A. GOSKAR (B.P. 329,630, 19.2.29).—The raw carbonaceous material is “pugged” or macerated in a moist condition, and then formed by extrusion into pellets, which are charged into a vertical chamber wherein they are successively dried, carbonised, and activated. The first drying zone is formed by a section of the chamber which has louvred walls, and across which a current of hot combustion gases is passed. A second drying zone is heated externally by the gas and vapours rising from the carbonising zone. Carbonisation is effected by passing the hot gases from the activating chamber through the material. The activating zone is maintained at 900–1200°, and means are provided for supplying water to be vaporised within the zone to produce therein an activating atmosphere of superheated steam. Valves are provided for discharging the material evenly from the activating chamber. A. B. MANNING.

Manufacture of highly active absorption carbon. I. G. FARBENIND. A.-G. (B.P. 306,490, 20.2.29. Ger., 21.2.28).—Wood chips, shavings, etc. are boiled with a solution of the activating agent, *e.g.*, zinc chloride, the hot liquid is drained off, and the wood again soaked with the cold solution. Instead of boiling with the solution, an equivalent swelling of the wood may be produced either by prolonged immersion, with stirring, in the hot solution, or by first boiling with water and then soaking in a cold or hot solution of the activating agent. A. B. MANNING.

Manufacture of smokeless fuel. C. VIEU (ASSR. to COMP. DES MINES DE VICOIGNE, NOEUX, & DROCOURT) and L. MOURGEON (U.S.P. 1,752,044, 25.3.30. Appl., 22.6.26.

Fr., 27.6.25).—The apparatus comprises a number of heating or distillation chambers for the fuel, each having an inlet and outlet for the heating fluid, and a distributing valve consisting of a hollow outer cylinder having ports in the periphery and an inner rotatable cylinder closely fitting within the other and having channels therein. The ports in the outer cylinder are connected by conduits to the inlets and outlets of the heating chambers, and the channels in the inner cylinder are so arranged that a number of the heating chambers are traversed by the heating fluid in series, while the others, which are disconnected, are being charged or discharged. By turning the valve a freshly charged chamber is brought into the series, and that in which distillation is complete is disconnected. A. B. MANNING.

Treating solid hydrocarbon-containing material. H. M. ROBERTSON (B.P. 329,674, 23.1.29).—Oil shales, coal briquettes, etc. are subjected to heat treatment in a tunnel retort wherein they pass successively through an oxidising and vaporising chamber at about 290°, a carbonising chamber at about 540°, and a cooling chamber. The heat evolved in the cooling chamber is used to preheat the air supplied in the oxidising and vaporising chamber. This air is further heated by being conveyed through pipes passing through the carbonising chamber. The latter is heated by the hot combustion gases from suitably arranged furnaces, these gases being conveyed through flue pipes running horizontally along both sides of the chamber. Provision is made for supplying steam to the carbonising and the cooling chambers. The vapours evolved in the first chamber and the products of carbonisation from the second chamber are withdrawn separately. A. B. MANNING.

Distillation of coal for the production of hard and large coke. E. ROSER (B.P. 306,092, 13.2.29. Ger., 15.2.28).—Dry coal poor in bitumen, *e.g.*, brown coal, peat, or dust coal, or the coke obtained by the distillation of brown coal or peat, is mixed with wet fuel rich in bitumen, *e.g.*, coal, and the loose mixture is pressed or rammed into a retort and distilled at 600°. A. B. MANNING.

Heat-treatment of granular carbonaceous material. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,957, 26.1. and 17.5.29).—The material, *e.g.*, brown coal, is passed through an inclined bundle of tubes which is rotated with its upper end in a stationary bunker charged with the granular material, and so designed that the material covers the whole cross-sectional area of the bundle. The tubes are mounted in a heating space traversed by hot gases, and are fitted in a gastight manner to perforated end-plates. The collecting chamber for the semi-coke is provided with an offtake for the volatile products. By mounting the bundle of tubes in a rotatable high-pressure drum, through which a heating medium can be passed, and suitably modifying the charging and discharging means, the apparatus can be utilised for the heat-treatment of carbonaceous materials with gases under pressure. A. B. MANNING.

Low-temperature carbonisation retorts. C. T. DRIGENKO and L. SELIGMAN (B.P. 329,827, 14.5.29).—A vertical retort is provided with a central rotary

shaft having agitating and propelling blades at intervals, and an extractor at the lower end. The charge is heated by a current of a carrier gas which enters the retort at the top and leaves at the bottom. The shaft and blades are hollow and serve to distribute the carrier gas, which can enter the shaft at the top, throughout the charge. The carrier gas leaving the retort is stripped of oil, preheated, and recirculated. A. B. MANNING.

Arrangement for heating coke ovens. A. OTT (U.S.P. 1,748,187, 25.2.30. Appl., 6.6.25. Ger., 7.6.24).—A perforated hollow column of fireproof material extends up the centre of each vertical heating flue, and serves to distribute the air supply thereto in order to lengthen the flame and bring about uniform heating of the oven. Hollow cap-shaped gas-distributing means are provided around the lower ends of the hollow columns. The device may be used for heating a coke oven or other type of furnace. A. B. MANNING.

Coke oven. C. OTTO (U.S.P. 1,748,142, 25.2.30. Appl., 20.3.25).—The burner opening in the flue is raised to such a height that the air, which is supplied at the bottom of the flue, is flowing uniformly upward without eddies when it reaches the burner, the end of which is tapered in order to avoid the formation of further eddies. The burners may extend to different heights in adjacent flues. The arrangement permits uniform heating in a vertical direction when the oven is heated with rich gas, and avoids deposition of carbon in the flue. A. B. MANNING.

Tunnel nozzle for rectangular coke ovens. A. S. KNOWLES, Assr. to TAR & PETROLEUM PROCESS Co. (U.S.P. 1,745,996, 4.2.30. Appl., 27.5.27).—A rectangular, sole-fired coke oven is heated by means of separate, parallel, adjacent combustion flues. Below each flue are two hair-pin regenerators which alternately and periodically supply air to and receive the products of combustion from the corresponding flue. Gas is admitted at each end of the flue alternately through the usual burner pipes. In order to produce a uniform heating in long ovens tunnel nozzles are provided in each flue for distributing the air supplied from one regenerator and for discharging the waste gases to the other. Each nozzle communicates with the corresponding regenerator through a supplementary opening in the floor of the flue, and consists of an open-bottom tunnel, closed at the front end, open at the rear end, and provided with auxiliary openings in the top. A. B. MANNING.

Cooling of coke. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,751, 23.3.29).—The hot coke is charged into a relatively deep chamber at the bottom of which an enlarged space is provided in which the partly cooled coke is sprayed with a regulated amount of water. The steam thus produced rises up the column of hot coke, cools the intermediate layers, and reacts with the hot upper layers to form water-gas. The amount of water used is just sufficient to cool the coke to 200°. A. B. MANNING.

Devices for improving the working of pressure gas producers. K. KOLLER (B.P. 309,874, 16.4.29. Hung., 16.4.28).—The internal cross-sectional area of the producer is diminished step-wise at the top and a

central charging cylinder and a gas-discharge connexion open into the portion of diminished cross-section. The charging cylinder is of such length that the gas-discharge connexion opens into the producer above the cone of inclination of the coal leaving the charging cylinder, whereas the lower horizontal boundary of the cone of coal extends above the lower edge of the diminished upper portion of the producer. A. B. MANNING.

Grates for gas producers. GIBBONS BROTHERS, LTD. From O. PISTORIUS (B.P. 329,776, 9.4.29).—The grate is conical and consists of a number of superimposed concentric rings of continuously increasing diameter, each resting on a flange on the ring below. A number of comparatively small orifices and nozzles are provided in the grate inclined to the radii and widening inwardly and downwardly. A. B. MANNING.

Manufacture of water-gas. D. J. YOUNG, Assr. to YOUNG-WHITWELL GAS PROCESS Co. (U.S.P. 1,751,501—3, 25.3.30. Appl., [A] 25.11.24, [B] 6.7.25, [C] 26.3.26. Cf. U.S.P. 1,468,190; B., 1923, 1013 A).—(A) The plant consists of a water-gas generator, a carburettor, and a superheater. After the blasting run water is introduced into the superheater, and the steam produced is passed successively through the superheater, the carburettor, and the fuel bed, the water-gas being withdrawn from the bottom of the generator. The process permits the use of raw coal, lignite, etc. as fuel. (B) A similar process may be used for the production of uncarburetted water-gas, the carburettor being either omitted or used simply as a second superheater. (C) Various modifications in the method of running a plant comprising a generator, a primary heat interchanger (carburettor), a secondary heat interchanger (superheater), and a wash box are described. The air-blasting run may be continued until the fuel bed and heat interchangers are hotter than in the usual water-gas process; steam may then be introduced at the top of the secondary heat interchanger and passed in the reverse direction to that usually employed through both heat interchangers and finally through the incandescent fuel in the generator. Other gases may be used in place of steam, water or oil may be introduced into the heat interchangers and vaporised therein, etc. A. B. MANNING.

Water-gas manufacture. F. W. STEERE, Assr. to SEMET-SOLVAY ENGINEERING CORP. (U.S.P. 1,752,036, 25.3.30. Appl., 22.12.24).—The plant, which operates without any outside source of steam, comprises a generator, a superheater, and a wash box. The blow gases are used to heat the superheater. The steam for the "down run," generated by spraying water into the top of the superheater, passes down through the superheater and thence down through the fuel bed. An "up run" is effected at suitable intervals by spraying water into the bottom of the generator. A. B. MANNING.

Manufacture of combustible gas containing hydrogen and carbon monoxide. G. WIETZEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,751,117, 18.3.30. Appl., 17.9.25. Ger., 1.12.22).—A pulverulent solid fuel is gasified by a continuous exothermic process with the aid of a gas containing a higher percentage of free oxygen than atmospheric air. A. B. MANNING.

Gas washer. F. H. WAGNER, ASST. to BARTLETT HAYWARD Co. (U.S.P. 1,752,045, 25.3.30. Appl., 28.6.27).—The gas is passed up through a vertical cylindrical washer wherein it is compelled to take a tortuous path through various sprays, water-curtains, and wetted screens. A number of shallow pans are supported at intervals within the casing in such a way as to leave annular spaces for the passage of the gas. A conical pumping and spraying element, attached to a central rotating shaft, dips into the water in each pan and sprays it laterally into intimate contact with the gas. Extending outwardly from the spraying element and below the perforated portion thereof is a series of vanes which act as a fan and throw the gas outwardly into pockets formed by a series of perforated plates attached to the casing. A current of washing water passes down through the apparatus. It falls from overflow tubes in each pan on to rotating plates which overlie the spraying elements and distribute the water on to perforated plates over the above-mentioned pockets through which the gas is forced.

A. B. MANNING.

Removing or recovering sulphur from fluids [gases]. PREMIX GAS PLANTS, LTD., and A. DOCKING (B.P. 329,973, 25.1.29).—Producer gas leaving the scrubbers at about atmospheric temperature is heated to about 32° and purified with iron oxide. The gas is then passed through a condenser to remove moisture and cool it to normal.

A. B. MANNING.

Freeing gas liquor from tar. GUTERHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 318,173, 23.8.29. Ger., 29.8.28).—The gas liquor is passed up through a layer of tar and then through a layer of filling bodies, *e.g.*, flints, small coke, etc. Provision is made for drawing off any light tar oils not retained by the filling bodies from the surface of the purified liquor, which is itself withdrawn through a tube dipping below the surface.

A. B. MANNING.

Recovery of tar and other by-products from coal-distillation gases. BARRETT Co., Assees. of S. P. MILLER (B.P. 303,167, 27.12.28. U.S., 29.12.27. Cf. B.P. 289,378; B., 1929, 803).—The gases from the earlier and the later portions of the coking operation in coke ovens or other coal-distillation plants are collected and cooled separately to give low-carbon and high-carbon tars, respectively. The tars, which are separated in different hydraulic mains, may be recirculated through the respective mains to effect further cooling. After the gases have been separately cooled to condense the heavier tars they may be combined and further cooled to condense a composite light tar. Suitable arrangements of the by-product recovery system are described.

A. B. MANNING.

Working-up of tars containing paraffin waxes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,671, 18.1.29).—Tars containing paraffin wax, *e.g.*, brown-coal tar, are worked up by first removing the paraffin waxes without distillation, *e.g.*, by addition of acetone and subsequent cooling, subjecting the oils to a condensation process, *e.g.*, by heating with aluminium chloride (cf. B.P. 318,311; B., 1929, 883), and then converting the constituents which have not been changed

into lubricating oils into hydrocarbons of low b.p. by cracking or destructive hydrogenation.

A. B. MANNING.

Refining benzene. SOC. DU GAZ DE PARIS (B.P. 314,052, 2.4.29. Fr., 23.6.28. Addn. to B.P. 307,935; B., 1930, 704).—The process is made continuous by means of a single apparatus, which is charged with filling substances, *e.g.*, Raschig rings, over which the sulphuric acid flows countercurrent to the benzene vapours. The gums and resins formed are sufficiently fluid to be drawn off with the excess acid, and the apparatus can operate for long periods without cleaning.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 309,859, 27.2.29. Holl., 16.4.28).—In the process which consists in heating the initial materials in the presence of hydrogen under pressure, preferably in the presence also of one or more catalysts, in order to form chiefly high-boiling tar-like products, and avoid extensive splitting, and thereafter subjecting these products to further heat treatment with hydrogen under pressure, preferably in the presence of one or more catalysts, in order to form lower-boiling products, the gaseous products are removed from the reaction vessel at the end of the first stage and, if desired, at the end of the second stage, while the reaction mass is still hot. Molybdenum or its compounds, and iodine or its compounds, are suitable catalysts for the first and second stages of the process, respectively.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,688, 17.1.29).—The gases issuing from the reaction chamber are freed from condensable products and treated with agents which take up hydrogen sulphide, whereby the concentration of hydrogen sulphide is reduced to less than 0.3 vol.-%. Thus the gas may be scrubbed under the reaction pressure with the middle oil (b.p. 200–300°) obtained in the process or other organic solvent for hydrogen sulphide; the latter is subsequently removed from the oil by reducing the pressure. The purified gases may be recycled.

A. B. MANNING.

Extraction or recovery of useful products from non-fibrous materials [by means of suint liquor]. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (B.P. 330,649, 5.3.29. Fr., 5.3.28. Cf. B.P. 307,360; B., 1930, 858).—Shales, oil-bearing sands, filtering and decolorising material, fish meal, waste from treatment of tallow, beeswax, and paraffins, etc. are treated with suint liquor (*d* 1.04) at about 60°; the extracted oils, fats, waxes, resins, etc. are separated by centrifuging, and the purified suint liquor is used for further extractions.

F. R. ENNOS.

Dehydration of oil and water emulsions. J. M. CAGE, ASST. to DEHYDRATORS, INC. (U.S.P. 1,754,009, 8.4.30. Appl., 17.9.27).—Emulsion is caused to flow upwards between successive sets of electrodes, the spacing of the electrodes being reduced as the resistance of the emulsion increases. The *P.D.* between the electrodes is controlled by the amount of current flowing

and in the event of a "flash over" the current is automatically cut off.

T. A. SMITH.

Enhancing the flow of oils through pipe lines.

A. H. ACKERMAN, Assr. to CATALYTIC CHEM. CO. (U.S.P. 1,754,296, 15.4.30. Appl., 16.2.27).—Viscous oils are made more fluid by the addition to each 30 brls. of 1 lb. of a mixture consisting of naphthalene (133 lb.), anthracene (21 lb.), caustic soda (56 lb.), sodium phosphate (10 lb.), ammonium carbonate (20 lb.), sugar (20 lb.), nitrobenzene (1 lb.), sulphuric acid ($\frac{1}{2}$ lb.), and phosphate rock (3 lb.).

T. A. SMITH.

Treating oils with finely-divided solid material.

N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,753,171, 1.4.30. Appl., 12.6.28).—Crude oil to which clay or other solid treating material has been added is passed into the central portion of a bubble-cap tower through which still vapours are ascending. The oil and clay from the bottom of the tower are cooled by passing through a heat exchanger and the clay is then removed by filtration. The filtered oil is distilled in the still.

T. A. SMITH.

Removal of paraffins from fluid hydrocarbons.

AKTIEB. SEPARATOR-NOBEL, N. O. BACKLUND, and K. G. MALM (B.P. 331,961, 26.4.29. Addn. to B.P. 296,805; B., 1928, 843).—Chilled distillate containing wax is passed through a centrifuge fitted with a mechanical device for removing the crystalline paraffin, which is separated. The cold oil which still contains amorphous paraffin is then passed through another centrifuge in which a paraffin-free portion and a fluid portion rich in amorphous paraffin are separated. The wax-free stock is used for the preparation of lubricating oils, and the fraction containing paraffin is returned to the first centrifuge for further treatment.

T. A. SMITH.

Distillation of hydrocarbons.

H. H. HEWETSON, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,753,149, 1.4.30. Appl., 25.6.23).—Oil flows through a series of stills each fitted with a fractionating tower containing bubble-cap plates, condensate from the bottom of each tower being passed to the middle of the tower on the succeeding still. From the towers all fractions that can be obtained at ordinary pressures are taken. The condensate from the tower on the last still is taken to the centre of a final tower maintained under vacuum so that lubricating fractions may be distilled off. Oil from the last still may also be taken to the tower.

T. A. SMITH.

Method and apparatus for cracking oil.

L. DE FLOREZ (B.P. 331,940, 17.4.29).—Oil is topped in a still fitted with a fractionating column. The heavier fractions from the tower are passed through a heating coil and delivered into a cracking still into which the hot residue from the topping still is pumped. Heat for the cracking of the residual oil is supplied by means of the hot light fractions which can be heated to a high temperature without coke deposition. Direct heating of the residual oil to cracking temperature is avoided.

T. A. SMITH.

Cracking hydrocarbons.

E. W. ISOM, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,753,432, 8.4.30. Appl., 25.5.27).—The overheating of tubes and cracking apparatus is prevented by returning a portion of the

cooler flue gases for admixture with the hot furnace gases before these pass over the apparatus to be heated.

T. A. SMITH.

Refining used lubricating oil.

J. C. PATRICK (B.P. 331,876, 10.4.29).—The oil is heated with sulphuric acid (d 1.84) at 50° and light products are driven off under vacuum at 230°. While hot, the oil is treated with 15–18% of fuller's earth to which lime or strontium oxide has been added.

T. A. SMITH.

Cracking or destructive hydrogenation of oils, or suspensions of coal in oil.

T. G. HUNTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,025, 16.3.29).—The material to be treated is caused to pass upwardly through a heated vertical tube or tubes by the injector action of a gas, *e.g.*, hydrogen, and to impinge on a baffle which causes it to fall back in the form of a parabolic curtain through which the gases and vapours must pass in order to leave the apparatus. The process may be carried out in the presence of a catalyst, which may, *e.g.*, be suspended in the material, or may be in the massive metallic form coated on the tubes.

A. B. MANNING.

Manufacture of olefines.

J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 330,623, 11.3.29).—The yield of olefines by catalytic thermal decomposition of hydrocarbons above C_2 at 600–700° is improved by addition of 1–9 vols. of methane to the initial gases.

C. HOLLINS.

Production of non-knocking motor fuels.

I. G. FARBERIND. A.-G. (B.P. 330,219, 30.11.28. Addn. to B.P. 281,247; B., 1928, 739).—The process of the prior patent is modified by extracting the bitumen or its primary conversion products in stages, under conditions of successively increasing rigour. The residue is destructively hydrogenated under pressure in presence of catalysts. The first extracts are worked up into montan or paraffin wax, and the others, after destructive hydrogenation if desired, into lubricating oils, benzines, etc.

A. B. MANNING.

Fuel for internal-combustion engines.

R. JOHN, Assr. to ARJON CHEM. Co. (U.S.P. 1,753,294, 8.4.30. Appl., 27.7.26).—Antimony trichloride, preferably dissolved in benzol, is added to gasoline as an anti-knocking dope in the proportion of 18 grains to the gallon.

T. A. SMITH.

Purifying exhaust gases of internal-combustion engines. L. MARKELS (B.P. 329,267, 8.12.28).—The exhaust gases are passed through a chamber containing a loosely packed charge of activated charcoal, preferably that prepared from carbonised nut shells.

A. B. MANNING.

Gas producer and combined furnace.

T. R. WOLLASTON (U.S.P. 1,767,952, 24.6.30. Appl., 31.5.27. U.K., 3.8.26).—See B.P. 274,286; B., 1927, 739.

Water-gas apparatus. C. S. CHRISMAN, Assr. to U.G.I. CONTRACTING Co. (U.S.P. 1,767,579, 24.6.30. Appl., 18.4.25).—See B.P. 245,688; B., 1926, 262.

Porous mass for storage of explosive gases and its manufacture. G. DALÉN, Assr. to AMER. GAS-ACCUMULATOR Co. (U.S.P. 1,767,514, 24.6.30. Appl., 14.5.25. Swed., 27.5.24).—See B.P. 234,462; B., 1925, 622.

Treatment of mineral and naphtha oils. G. PETROFF (U.S.P. 1,766,305, 24.6.30. Appl., 23.4.27. U.S.S.R., 8.11.26).—See B.P. 291,823; B., 1928, 595.

Continuous treatment of hydrocarbons with sulphur dioxide. G. CATTANEO and P. JODECK, Assrs. to ALLGEM. GES. F. CHEM. IND. M.B.H. (U.S.P. 1,766,281, 24.6.30. Appl., 10.5.27. Ger., 1.11.26).—See B.P. 279,774; B., 1928, 779.

Conversion of heavy hydrocarbon oils into light hydrocarbon oils or spirits. F. LAMPLOUGH (U.S.P. 1,765,167, 17.6.30. Appl., 27.5.26. U.K., 23.6.25).—See B.P. 258,656; B., 1926, 972.

Safety and stopping arrangements for water-gas producers and other intermittently operating apparatus. C. MIEDBRODT (B.P. 332,097, 12.8.29).

Gas burners. H. FRISCHKORN (B.P. 332,063, 19.7.29).

Oil burners. S. J. M. AULD (B.P. 331,874, 9.4.29).

Oil-gas burners. A. A. LAMBERTI and A. E. J. HOOPER (B.P. 331,881, 11.3.29).

Device for the purification of lubricating oil in centrifugal separators. AKTIEB. SEPARATOR (B.P. 332,133, 14.1.30. Swed., 17.1.29).

[Apparatus for] de-aerating lubricants. DRYSDALE & Co., LTD., and J. W. W. DRYSDALE (B.P. 331,950, 22.4.29).

Bitumen emulsions (B.P. 330,374).—See IX.

III.—ORGANIC INTERMEDIATES.

Modern methods of manufacturing absolute alcohol. E. MERCK (Z. angew. Chem., 1930, 43, 371). K. R. DIETRICH (*Ibid.*, 370—371).—Polemical. A discussion of the economics of preparing absolute alcohol by the lime-pressure process and of the difficulties attending the separation of ethyl and methyl alcohols by fractional distillation. (Cf. Dietrich, B., 1930, 261.)

A. R. POWELL.

Reactions of α - and β -naphthol. L. EKKERT (Pharm. Zentr., 1930, 71, 433—434).—Some modifications of the colour reactions with chlorine, chloroamine, and sulphuric acid, and the use of ultra-violet radiation in connexion therewith, are described.

S. I. LEVY.

Naphthols and fast bases (I.G.). ROWE.—See IV. **Determination of alcohol.** MARTIN.—See XVIII. **Urea in water.** MCCRADY. **Coal-tar disinfectants.** PHILBRICK.—See XXIII.

PATENTS.

Sulphonation of alcohols of high mol. wt. DEUTS. HYDRIERWERKE A.-G. (B.P. 307,709, 11.3.29. Ger., 9.3.28).—Non-carboxylated alcohols above C_8 , e.g., cetyl alcohol, are sulphonated with sulphuric acid in presence of acetic or other anhydride, with or without a diluent and/or a catalyst. C. HOLLINS.

Manufacture of aliphatic carboxylic acids from aqueous solutions thereof. I. G. FARBENIND. A.-G. (B.P. 306,097, 14.2.29. Ger., 15.2.28. Addn. to B.P. 300,923; B., 1930, 452).—The use of halogenated hydrocarbons for the separation of acetic acid by the method of azeotropic mixtures is extended to higher

acids; e.g., propionic, butyric, and isobutyric acid are obtained anhydrous by means of di- or tri-chloroethylene or chloroform. C. HOLLINS.

Manufacture of acetic anhydride. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,537, 8.3.29).—Acetic acid vapour is passed over an alkali phosphate containing boron phosphate, especially a mixture of boron phosphate, lithium phosphate, and sodium metaphosphate, at 600—700°. C. HOLLINS.

Catalytic removal of hydrogen or oxygen-containing [carboxylic] groups from organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 309,024, 8.3.29. U.S., 3.4.28).—Polybasic acids, esters, or amides are decarboxylated by passing the vapours with hydrogen, with or without steam, water-gas, etc., over a decarboxylating catalyst which has been incorporated in or combined with a base-exchange substance. In presence of ammonia or an amine vapour an amide of the monocarboxylic acid may be produced. The conversion of phthalic anhydride into benzoic acid or benzaldehyde is described. C. HOLLINS.

Manufacture of organic acyl halides. G. B. ELLIS. From Soc. DES USINES CHIM. RHÔNE-POULENC (B.P. 330,511, 1.3.29).— α -Halogenoethyl esters of organic acids are passed as vapour through a tube at 250—400°; the tube may contain a contact mass, such as pumice, kieselguhr, etc., with or without acid catalysts. α -Chloroethyl acetate gives acetyl chloride and acetaldehyde; $\alpha\beta$ -dichloroethyl acetate gives acetyl chloride and chloroacetaldehyde. The esters are suitably prepared by addition of hydrogen halide or halogen to vinyl esters. C. HOLLINS.

Preparation of primary and secondary amines. COMP. DE PROD. CHIM. & ELECTROMÉTALL. ALAIS FROGES, & CAMARGUE (B.P. 317,079, 29.7.29. Fr., 10.8.28).—A primary or secondary alcohol, other than a cyclohexanol, is heated with ammonia or a primary amine in presence of nickel, e.g., in alcohol at 190°. Tertiary amines are absent from the product. C. HOLLINS.

Manufacture of hydroxycarboxylic acids of naphthacarbazoles. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,643, 16.3.29).—The Kolbe reaction is applied to the carboxylation of hydroxynaphthacarbazoles. 4-Hydroxy- α -naphthacarbazole, from Laurent acid and phenylhydrazine followed by alkaline fusion, gives the 1(?)-carboxylic acid; 4-hydroxy- β -naphthacarbazole the 1(?)-carboxylic acid, m.p. 220°; 3-hydroxy- α -naphthacarbazole the 2- or 4-carboxylic acid; and 2-hydroxy- α -naphthacarbazole the 1-carboxylic acid, m.p. above 300°, which has a strong effect on the nasomucous membrane. C. HOLLINS.

Separating and purifying sulphonic acids of high mol. wt. G. PETROFF (U.S.P. 1,766,304, 24.6.30. Appl., 23.4.27. U.S.S.R., 26.11.26).—See B.P. 284,859; B., 1928, 361.

Condensation products of hydrogenated naphthalenes with ethylene. R. MICHEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,344, 24.6.30. Appl. 7.4.28. Ger., 4.2.26).—See B.P. 265,601; B., 1928, 740

Manufacture of derivatives of 2:3-hydroxynaphthoic acid. B. HEYN, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,754,390, 15.4.30. Appl., 28.2.29. Ger., 27.2.28).—See B.P. 326,971; B., 1930, 604.

Manufacture of alkoxy- -ketodihydrothionaphthens. E. RUNNE, K. MOLDAENKE, and E. FISCHER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,703, 24.6.30. Appl., 18.9.28. Ger., 8.10.27).—See B.P. 298,493; B., 1930, 95.

Production of alkyl ketones of the anthracene series. A. LÜTTRINGHAUS and F. KAČER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,443, 24.6.30. Appl., 24.3.27. Ger., 15.7.26).—See B.P. 289,585; B., 1928, 516.

[Manufacture of] 1-hydroxylamino-8-sulphoanthraquinone. A. SHEPHERDSON and A. J. HAILWOOD, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,767,230, 24.6.30. Appl., 28.3.27. U.K., 26.4.26).—See B.P. 274,226; B., 1297, 697.

Carbon disulphide (B.P. 331,734).—See VII. **Acetic and lactic acids** (B.P. 316,287).—See XVIII. **Phenols** (B.P. 330,333).—See XX.

IV.—DYESTUFFS.

Constitution of some Naphthols and fast bases (I.G.) used for the production of insoluble azo colours. F. M. ROWE (J. Soc. Dyers and Col., 1930, 46, 227—230).—Recent additions (cf. B., 1924, 704, 708; 1925, 797; 1926, 6, 310) to the Naphthol AS range of dyes include Naphthol AS-E, β -hydroxynaphthoic *p*-chloroanilide, m.p. 259° (commercial product) and 264° (from acetic acid); Naphthol AS-OL, β -hydroxynaphthoic *o*-anisidine, m.p. 162° (commercial) and 167° (from alcohol); Naphthol AS-BG, β -hydroxynaphthoic, 2:5-dimethoxyanilide, m.p. 181° (commercial) and 185° (alcohol); Fast Red RBE base, 6-benzamido-*m*-4-xylydine hydrochloride (free base, m.p. 176° from alcohol); Variamine Blue B base, 4-amino-4'-ethoxydiphenylamine sulphate, m.p. 100° (base, m.p. 100° from dilute alcohol). When diazotised in the usual manner at about 12°, Variamine Blue B base combines with 2 mols. of nitrous acid forming a nitroso-diazo-chloride which couples with naphthols to yield dull claret shades; on soaping or treating with a reducing substance such as sodium sulphide or sulphite the nitroso-group is removed and the shade changes to a permanent blue. The true diazo compound (which is very stable) may be prepared by diazotising at 45° and using only 1 mol. of nitrous acid. The following couplings are described: Fast Scarlet Salt GG with Naphthol AS-E, red, m.p. 294°; Fast Red R base with Naphthol AS-OL, red, with yellowish green lustre, m.p. 283°; Fast Scarlet Salt GG with Naphthol AS-OL, scarlet, with golden lustre, m.p. 280°; Fast Orange GC base with Naphthol AS-BG, dark orange-brown needles with green lustre, m.p. 225°; Fast Scarlet Salt GG with Naphthol AS-BG, reddish-brown, m.p. 278°; Variamine Blue B base with Naphthol AS, dark purple, m.p. 258°; Variamine Blue B base with Naphthol AS-E, purplish-black, m.p. 264°; the two last-named products obtained dissolve

with a crimson colour in sulphuric acid and are thus distinguished from Dianisidine Blue dyes.

A. J. HALL.

PATENTS.

Manufacture of [green] azo dyes insoluble in water [ice and pigment colours]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 330,349, 12.4.29).—A 6-amino-, 6-arylamino-, or 6-arylalkylamino-2:3-hydroxynaphthoic arylamide is coupled in substance or on the fibre with any non-sulphonated, non-carboxylated diazo-, tetrazo-, or diazo-azo-compound to give shades which are usually green to olive, bronze, or brown. Acid coupling yields bluish-red shades. The 6-arylamino-compounds are especially useful. The arylamides are made from the 6-amino-2:3-hydroxynaphthoic acids of B.P. 326,971 (B., 1930, 604).

C. HOLLINS.

Lakes of triarylmethane dyes. IMPERIAL CHEM. INDUSTRIES, LTD., F. W. LINCHE, E. H. RODD, and H. K. FREW (B.P. 330,229, 31.1.29).—Complex acid lakes are made by oxidising the leuco-compounds of basic triarylmethane dyes, or of acid triarylmethane dyes containing basic groups, in presence of a complex inorganic acid or its components, preferably in presence also of a dispersing agent stable to the oxidising agent used. The lakes may be improved by a subsequent heating with water. Leuco-malachite-green, *e.g.*, is added in hydrochloric acid solution to a solution of potassium permanganate containing sodium phosphotungstomolybdate. The leuco-compound of Acronol Brilliant Blue is oxidised with dichromate at 90—95° in presence of sodium phosphotungstomolybdate. A suitable dispersing agent is formaldehyde-naphthalenesulphonic acid.

C. HOLLINS.

Manufacture of [green] sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,308, 6.3.29).—The indophenol (or its leuco-compound) from *p*-aminophenol and an alkyl- or aralkyl- α -naphthylamine-6(or 7)-sulphonic acid is sulphurised, with or without addition of copper or a copper salt. Ethyl- or benzyl-Cleve acid, *e.g.*, is oxidised together with *p*-aminophenol, and the product is boiled with sodium polysulphide and copper sulphate solution.

C. HOLLINS.

Rubber derivatives (B.P. 313,919).—See XIV.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Reducing properties of wool. R. HALLER (Helv. Chim. Acta, 1930, 13, 620—628).—The behaviour of wool fibres towards different oxidising agents has been investigated. The two powerful oxidising agents chromic acid and potassium dichromate in aqueous-alcoholic solution are partly adsorbed without undergoing reduction. Alkaline Fehling's solution is unaffected and no blue colour is developed with an alkaline solution of Indanthrene Yellow R. The reduction of a 0.01% solution of potassium nitrate to nitrite observed by Schellens is due to bacteria, not to the wool itself. Wool bleaches a solution of Methyl Green, but itself becomes slightly coloured, and this effect is not due to combined sulphur. Wool will absorb 50% of its weight of iodine from an alcoholic solution, giving a unique

solid compound, fast to water and extraordinarily fast to light. Part of the iodine is reduced to hydriodic acid, which remains in the bath. Curves showing the rate of adsorption of iodine by wool from solutions of different concentration are given. F. L. USHER.

Change of some properties of sericin particles on the surface of the [silk] cocoon on drying. H. KANEKO, T. HAYASHI, S. CHINO, and M. MIYASAKA (Bull. Sericult. Silk Ind., Japan, 1930, 2, 2—3).—During the drying of the cocoon the sericin particles on the surface undergo dehydration and their physical properties change in such a way that the amount of dyestuff absorbed is decreased and the surface tension, viscosity, refractive index, and turbidity and colloidal properties of the sericin solution in water are altered.

W. O. KERMACK.

Behaviour of cellulose with bisulphite as compared with sulphurous acid solution. T. NAKASHIMA, S. OHORA, and J. MURAKAMI (J. Soc. Chem. Ind., Japan, 1930, 33, 199—200 B).—In both cases after heating for 24 hrs. at 100° no change in iodine value or acid value, or separation of sulphur, was observed; hydrolysis of the cellulose was slight, but dissociation occurred especially with sulphurous acid. Bisulphite-cooked cellulose diminished in viscosity with time of heating, but the increase in the copper number and the proportion of β - and γ -cellulose was small. On the other hand the sulphurous acid cook diminished the viscosity enormously and caused a distinct increase in β - and γ -cellulose, but the increase in copper number was not great, scarcely any sugar being formed. F. R. ENNOS.

Preparation of viscose from wood pulp. B. RASSOW, T. VOERSTER, and L. WOLF (Papier-Fabr., 1930, Fest- u. Auslandsheft, 77—103).—A summary of current knowledge relating to the preparation of viscose, with particular reference to the formation of alkali-cellulose and to ripening, is given. Experiments are described in which the effect of varying conditions during mercerisation, ageing, sulphidation, dissolution, and ripening on the viscosity is investigated. The main cause of the lower viscosity of wood-pulp viscoses, compared with those prepared from cotton, is held to be different molecular complexity of the two materials.

T. T. POTTS.

Viscose. VII. Acid decomposition of viscose. K. TANEMURA and S. MIYOSHI (J. Soc. Chem. Ind., Japan, 1930, 33, 184—186 B; cf. B., 1930, 758).—Changes in total and xanthate-carbon disulphide during ripening and the evolution of gases during the acid decomposition of viscose have been investigated. Carbon disulphide was determined by iodometric titration of the potassium ethyl xanthate formed by absorption of this substance in alcoholic potash; carbon dioxide by conversion into barium carbonate, dissolution of the latter in excess of standard hydrochloric acid, and back-titration with sodium hydroxide solution; and hydrogen sulphide and sulphur dioxide by means of iodine and sodium thiosulphate solutions. The total carbon disulphide decreases fairly quickly at the beginning of ripening, but afterwards only slowly. Of this total 16% is converted into other sulphur compounds when the viscose coagulates. The xanthate-

carbon disulphide decreases alternately slowly and quickly with ripening, giving a stepped curve, so that changes in the secondary reactions occur. Acid decomposition of spinning-viscose yielded CS_2 1.75, H_2S 0.38, SO_2 0.069, and CO_2 0.26, expressed as a percentage of the viscose. B. P. RIDGE.

Viscose. VIII. Gases evolved in spinning, reeling, washing processes, and in cabinet of viscose factory. K. TANEMURA and S. MIYOSHI (J. Soc. Chem. Ind., Japan, 1930, 33, 217—219 B).—From analyses of the yarn at various stages in its manufacture, the relative proportions and rates of evolution of carbon disulphide, hydrogen sulphide, sulphur dioxide, and carbon dioxide in the spinning and reeling rooms, skein washer, and cabinet are calculated. F. R. ENNOS.

Sulphonamide derivatives as plasticisers for acetylcellulose. T. S. CARSWELL (Ind. Eng. Chem., 1929, 21, 1176—1178).—The effect of incorporating various arylsulphonamides as plasticisers on some commercially important properties of cellulose acetate lacquers is examined. No relationship is apparent between the chemical constitution of these compounds and their "retentivity" ($[\text{wt. of plasticiser} \times 100]/\text{wt. of cellulose acetate}$) in cellulose acetate, but *p*-toluenemethylenesulphonamide has a significantly high value. Replacement of NMe by NEt tends to increase the elongation and decrease the tensile strength of the films produced. With increasing numbers of methyl groups in the nucleus the tensile strength of the films increases without notable effect on elongation. With the phenyl group attached to nitrogen there is a marked decrease in tensile strength. *p*-Toluenemethylenesulphonamide produced the highest tensile strength among materials examined. Toluenesulphonamide derivatives produced films of greater fastness to light than the corresponding benzene or xylene compounds. Derivatives with an NEt group are faster to light than corresponding methyl compounds, but an *N*-phenyl group markedly decreases fastness. The best results in this connexion were obtained with *p*-toluenemethylenesulphonamide. Relationships between chemical constitution of the plasticiser and resistance of films to outdoor exposure were indefinite, except that the sulphonmethylenamide is superior to simple alkyl derivatives. A. G. POLLARD.

Impregnation of wood during digestion in the pulping process. A. S. KLEIN (Papier-Fabr., 1930, Fest- u. Auslandsheft, 105—111).—A review of the literature. T. T. POTTS.

Effects of bleaching variables on strength of easy-bleaching spruce sulphite-pulp. P. K. BAIRD and R. H. DOUGHTY (Paper Trade J., 1930, 8, Ann. Rev. No. 175, 177, 179, 181, 183).—Increasing the rate of agitation during bleaching results in an increase in bursting and folding strengths, and no change in tensile and tearing strengths. Increasing the consistency from 2% to 7% results in a higher burst and a lower tear. The effect of raising the temperature from 21° to 41° is slightly to lower the folding and tearing strengths and to increase the tensile strength slightly. When the bleach ratio is raised from 12.3% to 17.0% a slight decrease in all

the strength properties is noted, except the burst, which is unchanged. The effects are explained on the grounds of a combination of chemical and mechanical actions during bleaching, which influence the behaviour of the fibres on beating, and hence the character of the resulting sheet.

T. T. POTTS.

Preparation of fibre test sheets. M. B. SHAW, G. W. BICKING, and L. W. SNYDER (Bur. Stand. J. Res., 1930, 5, 105—114).—A laboratory method for making small sheets of fibres has been developed and a suction sheet machine designed for studying the paper-making quality of pulp. The finished sheet is large enough for ordinary tests and results are closely reproducible.

C. J. SMITHELLS.

Filter cloths. WERNER.—See I. **Formation of Calcium sulphate in the sulphite-pulp process.** LAUBER.—See VII.

PATENTS.

Obtaining fibres from fibrous vegetable material. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (B.P. 307,360, 5.3.29. Fr., 5.3.28).—Fibrous vegetable material (flax, china grass, nettles, bark fibres) is treated at about 98° with suint liquor of a suitable concentration, or with alkaline salts derived from suint, and the liquor is afterwards centrifuged to recover the suint liquor for re-use and the substances dispersed therein; if desired, the suint liquor may be decolorised and/or deodorised and/or sterilised, or concentrated to a paste to facilitate transport and subsequently diluted for use.

F. R. ENNOS.

Manufacture of [artificial silk] fabrics. BRIT. CELANESE, LTD. (B.P. 310,845, 27.4.29. U.S., 1.5.28).—Fabrics having lustre pattern effects are manufactured by weaving together two types of cellulose acetate silk yarns (or yarns composed of other cellulose esters or ethers), one type being easily delustrated by treatment with boiling water or soap solutions and the other being more resistant and then subjecting the fabric to a delustring process such as immersion in a boiling soap bath.

A. J. HALL.

Precipitating baths for making threads or the like from viscose. I. G. FARBERIND. A.-G. (B.P. 304,244, 17.1.29. Ger., 17.1.28. Addn. to B.P. 303,514; B., 1930, 760).—In modification of the prior process, the precipitating bath contains no mineral acid or an amount not exceeding 12% (calc. as sulphuric acid), its content of the organic substance with tanning properties being increased to maintain it at the saturation point.

F. R. ENNOS.

Dry spinning of artificial threads. ACETA GES. M.B.H. (B.P. 317,368, 3.7.29. Ger., 14.8.28).—The spinning chamber consists of a gastight cylindrical pipe closed at one or both ends by a transverse pipe having a fixed or movable circular window at one or both ends, whilst the upper transverse pipe is provided with a tubulure for introduction of the spinning nozzle and for passage of the drying air.

F. R. ENNOS.

Manufacture of ammoniacal copper cellulose solutions. K. HESS and C. TROGUS (B.P. 307,939, 15.3.29. Ger., 16.3.28. Addn. to B.P. 301,752; B., 1930, 655).—During dissolution of the copper

alkali cellulose obtained in the prior patent, addition is made of a sufficient quantity of a salt (copper sulphate, ammonium sulphate or chloride) or an acid (sulphuric, hydrochloric, or carbonic) to remove the excess of free alkali over the amount required to produce a spinning solution containing the equivalent of 0.6% by wt. of caustic soda.

F. R. ENNOS.

Manufacture of acetylated cellulose fibres. HEBERLEIN & Co., A.-G. (B.P. 312,280, 23.5.29. Ger., 23.5.28).—Cellulose fibres are rendered immune to substantive dyes by partly acetylating with acetic anhydride, glacial acetic acid, and a catalyst (sulphuric acid or zinc chloride), in the presence of formaldehyde, a polymeride thereof, or a derivative or condensation product yielding either substance during the reaction.

F. R. ENNOS.

Manufacture of organic esters of cellulose and coating compositions containing the same. BRIT. CELANESE, LTD. (B.P. 326,515, 5.11.28. U.S., 5.11.27).—Low-viscosity organic esters of cellulose which have all the desirable properties of the high-viscosity esters and are in addition compatible with natural or synthetic resins, are obtained by esterifying at not above 50—55° (depending on the proportion of sulphuric acid used) and ripening the product as described in B.P. 300,140 (B., 1930, 655) to a stage between solubility in hot chloroform and very slight plasticity therein, the ester at the same time being soluble in a mixture of hot alcohol and benzene without the addition of water. The products are suitable for lacquers and coating compositions.

D. J. NORMAN.

Manufacture of cellulose esters. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 329,704, 25.2.29).—A solution of cellulose tricrotonate prepared as described in B.P. 328,588 (B., 1930, 655) is stirred with an aqueous solvent (acetic acid, alcohol) and a catalyst (mineral acids, sulphonic acids, acid salts) at 45—50° for about 20 hrs.; the partially hydrolysed ester is precipitated with water or aqueous alcohol.

F. R. ENNOS.

[Plastic] compositions containing cellulose derivatives [esters or ethers]. BRIT. CELANESE, LTD. (B.P. 313,405, 10.6.29. U.S., 9.6.28).—Halogenated aryl phosphates, e.g., tetrabrominated tolyl phosphate, are used as plasticisers for cellulose esters or ethers.

C. HOLINS.

Recovering salts from the waste water of artificial silk factories. E. RODOLFO (B.P. 331,648, 21.5.29).—Calcium sulphate, magnesium hydroxide, and zinc hydroxide (if zinc is present) are precipitated from the waste water by addition of milk of lime. The precipitate is separated and treated in aqueous suspension with carbon dioxide, when magnesium sulphate alone passes into solution; the liquor is concentrated until sodium sulphate separates. The free sulphuric acid in the waste water may be recovered as magnesium sulphate by neutralising with dolomite or calcined dolomite prior to treating with lime.

S. K. TWEEDY.

Continuous sulphate process. W. D. MOUNT, Assr. to K. A. FORREST (U.S.P. 1,754,902, 15.4.30. Appl., 11.1.29).—Sulphate-pulp is produced by alkaline

digestion, and the spent liquors are evaporated, incinerated, causticised, purified, and re-used, the process being continuous.

T. T. PORTS.

Paper and paper making. E. LIONNE (U.S.P. 1,752,802, 1.4.30. Appl., 12.11.28).—Celluloid is precipitated from solution by addition to the furnish of a paper-making beater, or is precipitated before adding to the paper pulp. The resulting stock is made into paper in the usual way, and the celluloid content of the sheet or board may be softened by the action of a suitable solvent under pressure.

T. T. PORTS.

Sizing of paper pulp. J. A. DE CEW, Assr. to PROCESS ENGINEERS, INC. (U.S.P. 1,753,775, 8.4.30. Appl., 24.5.29).—The furnish is brought to an optimum pH , e.g., by the use of an aluminium sulphate solution of pH 5—6.8, followed by the addition of free rosin size.

T. T. PORTS.

[Manufacture of] mineral-coated paper. W. W. TRIGGS. From CHAMPION COATED PAPER CO. (B.P. 331,578, 5.4.29).—Paper having a top coating consisting of at least 70% of mineral filling (clay, satin white, etc.) and not more than 30% (dry basis) of adhesive (casein, glue, starch, with a small amount of soap) is finished by bringing the coated side, while the coating is in a plastic condition produced by wetting or heating, in contact with a solid non-adhering surface, e.g., a revolving metal drum or a belt of hard rubber, having a finish similar to that required on the paper; the coating is then rendered non-plastic by drying or cooling and the paper removed from the solid surface.

F. R. ENNOS.

Process of making waterproof paper. G. A. BROWN, Assr. to BENNETT, INC. (U.S.P. 1,753,690, 8.4.30. Appl., 23.10.26).—Waste wax paper is disintegrated, the wax dispersed by heating and/or the addition of sodium silicate or rosin size, followed by alum, and the furnish is then run off on a paper machine.

T. T. PORTS.

[Carrier for] manufacture of films and foils etc. G. FRENKEL (B.P. 332,254, 18.2.29).

Manufacture of [composite] films, foils, etc. G. FRENKEL (B.P. 332,255—6, 18.2.29).

Manufacture of ornamented paper. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,707, 22.7.29).

Pulp beating or refining machines. T. D. NUTTALL, and BENTLEY & JACKSON, LTD. (B.P. 331,992, 21.5.29).

Products from non-fibrous materials (B.P. 330,649). See II. **Recovering alkali** (U.S.P. 1,753,128).—See VII. **Wall-board** (U.S.P. 1,754,413 and 1,754,843).—See IX. **Stencil sheet** (U.S.P. 1,753,204—5). **Moulded articles** (B.P. 331,851).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Stoving and allied effects in [wool] hosiery. A. T. KING (J. Soc. Dyers and Col., 1930, 46, 225—227).—From analogy of the special conditions which govern the fastness of certain azo dyes towards sulphur dioxide (King, B., 1928, 636) it was concluded that wool

materials would be satisfactorily bleached by the bisulphite process provided that the bleach liquor had $SO_2/NaOH = 1.25—1.35$; this was shown to be true in large-scale trials. Bleaching with a solution of sodium bisulphite is superior to the stoving process since it is effected without the odour of sulphur dioxide and without the danger of this gas affecting adjacent coloured materials; further the resulting white is purer and more permanent. The bisulphite bleach is very sensitive to iron, and iron stains in the wool immediately become evident; in the case of stoving, such stains are masked during bleaching, but return during subsequent storage.

A. J. HALL.

Naphthols and fast bases (I.G.). ROWE.—See IV. **Reducing properties of wool.** HALLER.—See V.

PATENTS.

Preparing wool-containing fabrics for dyeing. DEUTS. WOLLENWAREN MANUF. A.-G. (B.P. 308,605 and Addn. B.P. 331,529, 21.3.29. Ger., 23.3.28).—Waste materials containing artificial silks, or fabrics made from these and containing wool, are freed from the latter and also oily impurities by extraction with suitable organic solvents (e.g., esters of fatty acids, ketones, chlorinated hydrocarbons), adhering solvent being removed by means of cold or warm water. (Cf. B.P. 241,314 and 266,436; B., 1926, 9; 1927, 295.)

A. J. HALL.

Manufacture of evenly dyed viscose fabrics. I. G. FARBENIND. A.-G. (B.P. 306,908, 27.2.29. Ger., 27.2.28).—Level shades on viscose silk are obtained by the use of chromable azo dyes containing a single heavy metal (copper, nickel, or cobalt). [Stat. ref.]

C. HOLLINS.

Improving dyeing properties of viscose silk. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,612, 22.4.29).—Viscose silk threads are treated while swollen, after spinning and before the first drying, for about 1 hr. at ordinary temperature, with an ammoniacal solution of copper oxide containing 1—3 g. of copper and 20—100 g. of ammonia per litre, so that the loss of weight of cellulose by dissolution does not exceed 6%. Improved lustre, a roughened surface, and an increased and more uniform affinity for dyes are thus imparted.

A. J. HALL.

Dyeing of cellulose acetate. J. S. WILSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. [A] 330,216 and [B] 330,253, 27.11.28).—(A) Cellulose acetate silk is impregnated with a soluble leuco-ester of a vat dye (Soledon or Indigosol colours) at 50—80° for 30—60 min., preferably in presence of salt, and the colour is developed in the usual way without steaming. (B) The material is dried before development.

C. HOLLINS.

Printing white or coloured matt effects on esters of cellulose or its transformation products. I. G. FARBENIND. A.-G. (B.P. 309,194, 6.4.29. Ger., 7.4.28).—The materials are printed with a solution of urea (about 20—30%) thickened with gum tragacanth etc., dried, steamed for 5—10 min., and washed; a suitable dye may be added to the printing paste.

A. J. HALL.

Machines for continuously washing and treating, by a wet process, textile fibres and threads.

M. H. CARPMAEL. From SNIA-VISCOA (B.P. 332,003, 27.5.29).

Apparatus for dyeing tubular textile webs or fabrics. H. LOESER (B.P. 332,180, 2.1.30. Latvia, 5.1.29).

Apparatus for treating hair, feathers, etc. [with ozonised air]. W. W. TRIGGS. From AMER. HAIR & FELT Co. (B.P. 331,845, 4.4.29).

Titanium solutions (B.P. 309,090).—See VII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Evaporation of electrolytic caustic soda. J. A. LEE (Chem. Met. Eng., 1930, 37, 404—408).—An account is given of the continuous evaporation and salt-recovery system at the South Charleston plant of the Westvaco Chlorine Products, Inc. The original solution contains about 8% NaOH and 17.5% NaCl, and the final products from the steam evaporation plant are: 53 short tons of NaOH per 24 hrs. in the form of caustic soda solution, d 1.53, containing also about 0.9% NaCl in solution and none in suspension, and sodium chloride brine for return to the electrolytic cells. The water evaporated is about 1 million lb. per day, and the steam used, at 10 lb./sq. in. gauge, about $\frac{3}{4}$ million lb. Evaporation is in three stages, a double effect followed by a single, then follows a continuous crystalliser and Laughlin centrifugal filter which delivers the final caustic liquor. Swenson evaporators and Dorr thickeners and rake classifiers are employed. The materials of construction used are nickel-cast iron, nickel tubes, and Monel metal. B. M. VENABLES.

Determination of ammonia in synthetic ammonia solution. E. OTT (Chem.-Ztg., 1930, 54, 412).—For weighing the ammonia solution in the method of Egeling (B., 1930, 371) a two-bulb pipette is recommended. The upper bulb has a capacity of 100 c.c. and is closed by stopcocks above and below the bulb; the lower bulb has a capacity of 25 c.c. The tube above the upper stopcock is opened out to form a small funnel which facilitates introduction of rinsing water into the bulb. A. R. POWELL.

Preparation of ammonium sulphate and nitrogen. D. VORLÄNDER and A. LAINAU (Z. angew. Chem., 1930, 43, 647—648).—Ammonium sulphite solutions are oxidised very rapidly by air in presence of soluble cobaltamine sulphite compounds, the best conditions being a temperature of 18—19°, pH 8.4—8.6, and a concentration of 80 g. of $(NH_4)_2SO_3 \cdot H_2O$ (with 10 mg. Co) per litre. Addition of other substances which normally accelerate or inhibit catalytic oxidations does not affect the influence of the cobalt compounds. On crystallisation of the ammonium sulphate, the cobalt compounds remain in the mother-liquor. S. I. LEVY.

Determination of active chlorine in hypochlorite liquors. J. D. BLAKELEY, J. M. PRESTON, and F. SCHOLEFIELD (J. Soc. Dyers and Col., 1930, 46, 230—

233).—Ordinary indicators are eliminated in the Penot and Ehrenfried methods for determining active chlorine, which use standard solutions of arsenious acid, by using an electrical method for ascertaining the end-point in the titration, it being found that in the region of the end-point there is a sudden drop of electric potential from 691 to 250 millivolts and an increase from 512 to 820 millivolts, respectively, with regard to a platinum electrode immersed in the hypochlorite solution being titrated. The Penot and Ehrenfried methods may be combined for determining both the hypochlorite and chlorate contents of a bleaching solution, by titrating first with sodium arsenite in bicarbonate solution, then adding a known excess of arsenite, acidifying with hydrochloric acid, and titrating with potassium bromate, the difference between the two results being equivalent to the chlorate present. A simplified method is described for works' routine analysis in which the use of a platinum electrode within a suitable buffer solution avoids the necessity for a potentiometer, standard cell, accumulator, etc. A. J. HALL.

Bauxite. Float-and-sink fractionations and flotation experiments. B. W. GANDRUD and F. D. DE VANEY (U.S. Bur. Mines, Bull. 312, 1929, 101 pp.).—Gravity separation showed that the separation of silica from low-grade bauxites was almost impossible owing to the non-detachment of particles even when pulverised to 100-mesh. It is believed that bauxites in the United States consist of hydrated gibbsite ($Al_2O_3 \cdot 3H_2O$) with which is associated kaolinite as the source of silica, and apart from the fine-grinding involved, the difference in density does not promise successful separation. Concentration of iron oxide was more promising, and it was indicated that many bauxites containing 10—20% Fe_2O_3 could be concentrated on tables to a content of less than 10% Fe_2O_3 , also from 10% Fe_2O_3 to less than 3% in some instances. C. A. KING.

Determination of "sugar-soluble" silica in burnt limestone. O. SPENGLER and A. TRÄGEL (Z. Ver. deut. Zucker-Ind., 1930, 80, 413—419).—In the second saturator, where the dilute sugar syrup is treated with lime, relatively large quantities of silica enter the solution and subsequently cause the formation of silica incrustations in the plant. Most of this silica is derived from the lime, and a close approximation of this "sugar-soluble" silica may be obtained as follows:—2 g. of lime are ground to an impalpable powder, 0.5 g. of which is slaked with water in a nickel dish. The pasty mass is washed into a flask with more water to give a volume of 100 c.c.; 5—6 drops of 1% phenolphthalein solution are added and the mixture is carefully treated with *N*-sulphuric acid until the colour fades, after vigorous shaking, to a pale rose, boiled for $\frac{1}{2}$ min., and filtered. The filtrate is cooled to 20° and treated with 1 g. of ammonium molybdate and 5 c.c. of 3*N*-hydrochloric acid, and the resulting yellow colour is compared with that of a standard potassium chromate solution (cf. Spengler and Brendel; B., 1930, 260). The results are reasonably close to those obtained for the silica content of the syrups after the lime treatment. A. R. POWELL.

Formation of calcium sulphate in the tower-liquors of the sulphite-pulp process. II. LAUBER (Papier-Fabr., 1930, Fest- u. Auslandsheft, 50—53).—Traces of calcium sulphate are formed during the preparation of calcium bisulphite from pure sulphur dioxide. It is shown that this is not due to catalytic action, control experiments using catalysts failing to increase the velocity of sulphate formation. The possibility of a molecular side-reaction is precluded by the fact that it is possible, in an atmosphere of carbon dioxide and pure sulphur dioxide, to prepare bisulphite solutions free from sulphate. It is concluded that the reaction is one of pure oxidation dependent simply on the partial pressure of oxygen. T. T. POTTS.

Chlorine in coal. DAMON. **Silica gel.** BOSSHARD and WILDI.—See II. **Determination of potassium.** CALVERT.—See XVI.

PATENTS.

Manufacture of phosphoric acid and products containing phosphoric acid. AKTIEB. KENISKA PATENTER, ASSEES. OF KONSTGODNINGSFABR. AKTIEB. I. LANDSKRONA (B.P. 314,976, 4.4.29. Ger., 7.7.28).—Phosphate rock is treated at a minimum temperature of 80° with sulphuric acid solution containing more than 10 mol.-% of sulphuric acid. Under these conditions calcium sulphate is deposited in the anhydrous or hemihydrated form (or in a mixture of these forms).

S. K. TWEEDY.

Manufacture of dicalcium phosphate. R. D. PIKE (U.S.P. 1,753,478, 8.4.30. Appl., 14.11.27).—Gaseous hydrogen chloride is brought into contact with an aqueous suspension of pulverised phosphate rock, and, after neutralising with calcium carbonate, the precipitate is separated. H. ROYAL-DAWSON.

Treating leucitic rocks with oxides of nitrogen. F. JOURDAN (B.P. 309,957, 18.4.29. Ital., 18.4.29).—The rock is treated with nitric acid in vapour form, alone or mixed with oxides of nitrogen and water vapour and air. The quantity of nitrous gases is in excess of that necessary for forming nitrates of the potassium, aluminium, and iron contained in the rock. The aluminium and iron nitrates in the mixture formed are decomposed by heat, and the oxides of nitrogen thus evolved are again utilised. A cyclic process is described.

S. K. TWEEDY.

Recovering alkali. R. H. MCKEE (U.S.P. 1,753,128, 1.4.30. Appl., 24.10.24).—The black ash residue from soda-pulp mills etc. is ground with calcium carbonate sludge, excess of water is removed, and the residue is heated and recovered as caustic soda.

H. ROYAL-DAWSON.

Production of sulphates containing little or no water of crystallisation. O. KASELITZ, and KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 331,674, 17.6.29).—The sulphate solutions, of maximum concentration, are heated in closed vessels at temperatures (e.g., 200°) at which the solubility of the salt becomes practically zero, or very slight. The mixture of salt and liquor is separated without any appreciable reduction in pressure, the heat of the liquor being recovered for further use. When the salts to be treated are normally highly hydrated they may be treated alone or with quantities of water or mother-liquor insufficient to dissolve the

salts. When the sulphates contain soluble impurities these are frequently retained by the mother-liquor and thus a purified dehydrated salt is produced.

S. K. TWEEDY.

Treatment of sulphur-containing mineral complexes such as alunite, jarosite, copiapite, [carphosiderite], and the like. (SIR) D. MAWSON (B.P. 331,552, 4.4.29).—The crushed mineral is treated with liquid, dissolved, or gaseous ammonia (e.g., steam containing ammonia), if desired at moderate heat (100°) and/or under pressure. The mineral breaks down to aluminium, iron, and other metal hydroxides with simultaneous formation of the sulphates of alkali metals and of ammonium. Methods of separating the products are given. The process is adapted for fixing ammonia.

S. K. TWEEDY.

Preparation of anhydrous perborates. G. SCHOENBERG (B.P. 312,664, 17.5.29. Ger., 30.5.28).—Crystallised perborates, e.g., sodium perborate, are dehydrated completely by heating, e.g., in a rotating drum, first at 40—100°, with or without reduced pressure, and then at a pressure of 40 mm. or less. During both stages dry inert gases may be passed over the material, and the temperature during the second stage may be increased above 100°, particularly where the inert gas enters the apparatus and where there is little evolution of water vapour. The partially-dried perborate may be formed into tablets etc., and the final dehydration effected in the containers, e.g., of glass, subsequently used for storage or transport. F. G. CLARKE.

Production of potassium nitrate. O. KASELITZ, and KALI-FORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 327,909, 13.6.29).—A solution of potassium nitrate, aluminium nitrate, and aluminium chloride, saturated at 0°, is evaporated, preferably in a vacuum, so that on cooling to about 20° hydrated aluminium chloride separates out. Potassium chloride and its equivalent of aluminium nitrate are added to the mother-liquor, together with sufficient water to restore the original concentration, and the solution is cooled to about 0°. Potassium nitrate separates out and the mother-liquor is returned to the process. The precipitated aluminium chloride may be converted into nitrate. The starting liquid may also be saturated with potassium chloride in addition to, or in place of, the aluminium nitrate. In a modified process potassium nitrate is precipitated from the saturated solution by addition of potassium chloride; the aluminium chloride formed in the reaction is precipitated from the mother-liquor by adding aluminium nitrate at the point at which the solution is saturated with aluminium chloride, aluminium nitrate, and potassium nitrate, and the solution is re-used by adding further potassium chloride. In these processes the aluminium nitrate may be produced *in situ* from the oxide and nitric acid or nitrous gases.

S. K. TWEEDY.

Production of potassium monophosphate. KALI-FORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 327,885, 6.5.29. Ger., 19.11.28).—Potassium chloride is heated (e.g., at 130° and under reduced pressure) with an excess of phosphoric acid of over 30% concentration (together with available mother-liquors) until the chlorine is expelled; the whole is then cooled and the acid salt thus obtained,

KH_2PO_4 , H_3PO_4 , is decomposed with a quantity of potassium monophosphate solution, or water, such that a saturated solution of this acid salt and monophosphate is produced with the precipitation of monophosphate. Alternatively, the hot solution of the acid salt may be treated directly without cooling.

S. K. TWEEDY.

Treating mixtures of alkali stannates, arsenates, and antimonates. A. E. HALL, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,747,709, 18.2.30. Appl., 5.3.27).—The alkaline slag obtained in the Harris process of lead refining is fused and poured in a thin stream into a saturated solution of sodium chloride containing sufficient sodium hydroxide to give d 1.4. The mixture is agitated at 90° and the hot, alkaline arsenate solution is separated by decantation from the insoluble residue of sodium stannate and antimonate. On cooling the solution trisodium arsenate separates and the mother-liquor may be used again for leaching or evaporated to obtain a mixture of sodium hydroxide and chloride for use as a flux in refining further quantities of lead. The insoluble sodium salts are digested with hot water, which dissolves sodium stannate, and the insoluble sodium antimonate is washed, dried, and smelted to antimony. The stannate solution is agitated with powdered tin or treated with stannous salts to remove lead, copper, and arsenic, and the purified solution is electrolysed to obtain tin. A. R. POWELL.

Production of quicklime and sulphur dioxide. A. FLECK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,128, 9.5.29).—Gypsum or anhydrite is heated in a rotary kiln by means of a reducing flame of coal gas and producer gas mixed with insufficient air for complete combustion, the temperature being maintained at 1050 – 1300° . No dead-burnt lime or calcium sulphide is formed. W. J. WRIGHT.

Production of nitrate of lime. APPAREILS ET EVAPORATEURS KESTNER (B.P. 329,145, 3.5.29. Fr., 3.4.29. Addn. to B.P. 279,037; B., 1928, 230).—In the modified process the coarse limestone is added to a mixture of nitric acid with some of the previously prepared calcium nitrate solution, or a suspension of calcium carbonate sludge in calcium nitrate solution is treated with nitric acid. L. A. COLES.

Continuous calcination of gypsum. A. W. TYLER (U.S.P. 1,746,294, 11.2.30. Appl., 2.6.26).—The gypsum is fed automatically to a pressure kettle in which it is heated to expel the greater part of its water content and the steam evolved is superheated under pressure to 190° and returned to the kettle to drive the gypsum into the calcining furnace in which the final dehydration is effected by means of superheated steam.

A. R. POWELL.

Manufacture of purified zinc sulphate solutions. SOC. MINIERE & METALLURG. DE PENNARROYA (B.P. 331,886, 11.4.29. Fr., 30.1.29).—Zinc sulphate solutions containing cobalt are treated with dimethylglyoxime or other organic oximes or phenols, which take up the metal, forming a compound which is fixed by means of adsorbent material, e.g., active carbon, talc, etc.

H. ROYAL-DAWSON,

Manufacture of concentrated titanyl and titanic sulphate solutions. I. G. FARBENIND. A.-G. (B.P. 309,090, 4.4.29. Ger., 4.4.28).—A moderately hot solution (e.g., not exceeding 60°) of metatitanic acid in sulphuric acid is saturated with metatitanic acid, and then orthotitanic acid is added and the mixture concentrated *in vacuo* (e.g., at 70 – 90°). The titanic acids may be added in solid form or in aqueous solution. The solutions obtained are substantially free from iron and are adapted for use as tanning agents and mordants.

S. K. TWEEDY.

Artificial snow. R. W. SIMPSON (U.S.P. 1,746,717, 11.2.30. Appl., 24.8.27).—A material to represent snow for decorating purposes consists of light, glistening crystals of gypsum prepared by dissolving commercial calcium sulphate in hot 10% sulphuric acid, filtering the solution, and allowing it to cool slowly. A. R. POWELL.

Production of carbon disulphide. IMPERIAL CHEM. INDUSTRIES, LTD., W. B. FLETCHER, T. S. WHEELER, and J. MCAULAY (B.P. 331,734, 21.8.29).—Hydrocarbons (coal gas, coke-oven gas, etc.), preferably preheated at 600 – 800° , are heated with sulphur vapour at temperatures above 1100° , preferably above 1300° . The mixed gas is preferably passed through a heated tube at a rate such that 100–300 vols. of gas (measured at the reaction temperature) pass through one vol. of the reaction space per min. The yield of carbon disulphide corresponds to about 50% of the sulphur used; no carbon oxysulphide is formed and any unreacted sulphur may be recovered as such, or as hydrogen sulphide.

S. K. TWEEDY.

Catalytic oxidation of ammonia. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,765,352, 17.6.30. Appl., 14.4.28).—See B.P. 309,583; B., 1930, 764.

Production of amphoteric hydrated oxides of metals by hydrolysis. R. H. MONK and J. IRWIN (U.S.P. 1,755,512, 22.4.30. Appl., 18.2.29).—See B.P. 329,041; B., 1930, 661.

Production of hypochlorite compositions. (A, B) R. B. McMULLIN and (A) M. C. TAYLOR, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,754,474–5, 15.4.30. Appl., [A] 29.12.28, [B] 27.9.28).—See B.P. 329,896 and 319,727; B., 1930, 711.

Catalytic transformation of gases (B.P. 309,169).—See I. Recovering salts (B.P. 331,648).—See V.

VIII.—GLASS; CERAMICS.

Rate of expansion of glass threads on heating. G. TAMMANN and E. JENCKEL (Z. anorg. Chem., 1930, 191, 122–127).—From experiments on the rate of elongation of threads of various glasses under tensile strain, it has been found that the temperature at which the relative stationary elongation (cf. English, B., 1923, 550A) for the tensile strain of 1 kg./mm.² has the value of 0.0002/min. is approximately the same as that temperature within the softening interval at which the physical property-temperature curves have a point of inflexion (cf. A., 1929, 1138). Values of the temperature coefficient of the fluidity have also been deduced from the results. R. CUTHILL.

Transmission changes in ultra-violet glasses during high-temperature exposure to light. C. C. NITCHE and F. C. SCHMUTZ (Science, 1930, **71**, 590).—When kept in contact with the hot tube of the mercury arc lamp (about 450°), certain glasses showed a marked increase in short-wave transmission. L. S. THEOBALD.

Effect of oil on devitrified glass surfaces, and the detection of this defect in glass. P. WOOG (Bull. Soc. chim., 1930, [iv], **47**, 450—453).—Very slight devitrification of the surface of glass is rendered evident by treating the surface with an oil, especially if this contains traces of water. The particles of silica resulting from the devitrification become detached and form a suspension in the oil. A. B. MANNING.

Volumetric determination of silica in glass. N. A. TANANAEV and A. K. BABKO (Ukraine Chem. J., 1930, **5**, 71—85).—0.25 G. of powdered glass is treated in a platinum basin with 4 g. of potassium fluoride, 10 c.c. of water, and 5 c.c. of hydrochloric acid. After 15 min. 10 c.c. of alcohol are added, and after 2 hrs. the precipitate of potassium fluosilicate is collected, washed with 50% alcohol, mixed with 20 c.c. of 4*N*-calcium chloride solution, and diluted to 250 c.c. The solution is titrated at 100° with 0.5*N*-sodium hydroxide solution until the supernatant solution becomes clear. The number of c.c. of sodium hydroxide solution used in a blank determination is subtracted from the result obtained. The above method gives results about 0.3% higher than the theoretical. R. TRUSZKOWSKI.

Method of treating clays to overcome drying defects. J. G. PHILLIPS (Trans. Canad. Inst. Min. Met., 1929, **32**, 270—282).—Clays found in Alberta, Manitoba, and Saskatchewan, otherwise suitable for brick-making, crack readily in drying. This tendency may be minimised by preheating at 450—550°, or by adding finely-ground grog together with ferric chloride or a mixture of ferric chloride and sodium chloride. C. W. GIBBY.

Saskatchewan clays of Dominion importance. W. G. WORCESTER (Trans. Canad. Inst. Min. Met., 1929, **32**, 255—269).—A classification of clays and their uses is given, and the age of the Saskatchewan deposits is discussed. Analyses and fusion data, linear drying and burning shrinkages, porosity, and modulus of rupture are tabulated. C. W. GIBBY.

Method of measuring strains between glazes and ceramic bodies. H. G. SCHURECHT and G. R. POLE (Bur. Stand. J. Res., 1930, **5**, 97—104).—A 2-in. diam. hollow cylindrical ring of the material to be glazed is glazed on the outside only. Two reference marks are made about $\frac{1}{4}$ in. apart on the edge of the ring and their distance is accurately measured. The ring is then slit between the marks and the distance remeasured. The contraction or expansion gives an approximate indication of the extent to which the glaze was in tension or compression. By means of this test glazes can be fitted to ceramic bodies so as to be in sufficient compression to avoid crazing without causing shivering. C. J. SMITHELLS.

Manufacture of refractory products in the U.S.A. G. V. EVERS (J. Soc. Glass Tech., 1930, **14**, 205—218 r).

Particle size. KASAI.—See I.

PATENTS.

[Continuous-type tunnel ceramic] kilns. H. M. ROBERTSON (B.P. 331,224—5, 23.1.29. U.S., 9.5.28. Cf. B.P. 311,316; B., 1930, 664).—In a kiln having preheating, firing, salt-glazing, and cooling zones in succession with exhaust means at the junction of the last two: in (A), refuges or expansion chambers are formed in the walls of the kiln between the salt-volatilising fire and the exhaust ports in which the gases are retarded by circulation and from which they are thoroughly distributed among the goods. In (B), air is heated in pipes on the interior walls of the cooling zone and despatched by a fan through passages in the roof of the firing zone to ports distributed over the walls of the preheating zone, whence they mix with the goods and effect water-smoking and preheating. B. M. VENABLES.

Feeding and forming glass. F. ECKERT (B.P. 310,422, 24.4.29. U.S., 25.4.28).

Manufacture of safety or unsplinterable glass. T. W. HOLT (B.P. 332,385, 4.7.29).

IX.—BUILDING MATERIALS.

Crushing and preliminary grinding in Portland cement manufacture. A. C. DAVIS (Cement, 1930, **3**, 813—822).—A description of various types of crushing and grinding machinery suitable for the preparation of the raw materials of Portland cement is given and their limitations are stated. In contradistinction to ore dressing, the production of fines by the crushers is an advantage. The power used for reduction of materials is about 80—85% of the total power used in a cement works, and for the raw materials before burning alone may be 8—10 kw.-hrs. per ton for chalk and clay or 35—40 kw.-hrs. per ton for harder materials. B. M. VENABLES.

Diminution of the water content of sludge in the manufacture of Portland cement by the wet process. Influence of diluting substances on the fineness of grinding materials for the manufacture of cement by the wet process. P. P. BUDNIKOV, G. V. KUKOLEV, and V. M. LESHOEV (Ukraine Chem. J., 1930, **5**, [Tech.], 27—39, 41—42).—The fluidity of Portland cement sludge containing 48% of distilled water is equal to that of a sludge made by adding 42% of a 0.01*N*-sodium silicate solution. This entails an economy of 8.3% of fuel required for the preparation of dry cement, and permits a greater throughput for a given plant, by diminishing the bulk of the products. The fluidity of cement sludges is adversely affected by the presence of alkaline-earth ions in the water; hence the addition of any reagents precipitating these ions, such as carbonates, silicates, and sucrose, will augment the fluidity. The proportion of grains of small diameter obtained by grinding cement in the presence of 0.04*N*-sodium carbonate solution is greater than when spring-water is used. R. TRUSZKOWSKI.

Constitution of cements. N. P. COSTA (Anal. Soc. Cient. Argentina, 1930, **109**, 73—96).—The variations of crystallographic structure of different types of cement with variations of the method of curing have been studied. In general, the cement is not merely a mixture

of the component aluminates and silicates, but contains solid solutions and isomorphous mixtures of calcium and iron silicates and calcium aluminates. "Ciment fondu" contains silicates and aluminosilicates, and aluminates crystallised upon the residue formed from iron silicates during the fusion; there is a preponderance of a metastable form of $5\text{CaO} \cdot 3\text{Al}_2\text{O}_3$ in cements of this class. "Ciment fondu" differs from Portland cement in that the chemical composition as well as the crystal arrangement varies with the mode of curing, and the double refraction diminishes with more intensive curing. Portland cement cured in water has the same structure as that obtained by dry-curing, except that the crystals of hallite and zeolite are more uniform and somewhat larger, and the zeolite crystals are oriented radially around crystals of hallite and are united by iron silicates; double refraction is also more marked in this case. Portland cement of a high initial strength, prepared by wet-curing, contains a high proportion of zeolite and relatively large and well-formed crystals of hallite and zeolite. The strength of such a cement is governed mainly by the orientation of the individual crystals, especially those of hallite.

H. F. GILLBE.

Testing of road-making materials, using small quantities. R. GRENGG (Mikrochem., 1930, 8, 281—292).—The technique of methods for the mineralogical and grain-size analysis of road-making materials, using only small quantities of substance, is outlined and small-scale apparatus for the preparation and mechanical testing of agglomerates is briefly described. A. R. POWELL.

Improving the swelling and shrinking of wood in conditioning processes. FALCK (Chem.-Ztg., 1930, 54, 569—571).—The degree to which partly or artificially seasoned timber swells and shrinks may be reduced to one half by (1) keeping in boiling water for several hours in open vessels; (2) heating to a definite seasoning temperature, which must be determined for each wood (for the beech used in the tests, this temperature was found to be 123°); or by (3) steaming at intervals over several days. Chemical treatments were either ineffective or unsuitable. S. I. LEVY.

PATENTS.

Manufacture of a material for building etc. from, e.g., Sorel cement or gypsum and bituminous substances. L. C. SCHILLING (B.P. 303,889, 2.1.29. Holland, 13.1.28).—An aqueous dispersion of bitumen is incorporated with a cement mixture composed of calcined magnesite, magnesium chloride solution, and filling materials. The water content of the dispersion is controlled so that the final water content of the mixture gives the optimum setting conditions of the oxychloride cement. C. A. KING.

Manufacture of laminated [building] materials. R. ARNOT (B.P. 331,561, 4.2. and 3.5.29).—Composite boards of vegetable fibrous materials, e.g., sugar-cane fibre, or laminated sheets of Balsa wood, are coated with fire- or water-proof compositions, e.g., asbestos cement etc. C. A. KING.

Manufacture of cement. BRIT. PORTLAND CEMENT MANUFACTURERS, LTD., S. G. S. PANISSET, and W. S. HANNAH (B.P. 331,584, 6.4.29).—Cement clinker is treated with

a reducing agent before cooling and is then cooled rapidly to prevent re-oxidation. Powdered coal may be blown upon the hot clinker or the clinker may be delivered on to a surface coated with a volatile mineral oil, and then cooled in water to such a point that the residual heat drives off adherent water. C. A. KING.

Cementitious composition. A. METHERELL, Assee. of G. E. BARNHART and H. E. PFAFF (B.P. 310,875, 17.4.29. U.S., 2.5.28).—A solution for gauging cement mixtures for waterproof and glazed artificial stone consists of aluminium sulphate 1 pt., magnesium sulphate 1 pt., potassium (sodium) sulphate 2 pts., and water 120 pts. (all by vol.). Substitutions in the preferred composition by iron or zinc sulphates or sodium silicate provide modified formulæ.

C. A. KING.

Fibrous wall-board. H. E. BROOKBY, Asst. to U.S. GYPSUM Co. (U.S.P. 1,754,413, 15.4.30. Appl., 5.2.27).—A mixture of comminuted maize stalks and peat fibres is treated with caustic soda and pulped to sufficient degree of fineness with water; it is then formed into sheets, dried, and pressed. H. ROYAL-DAWSON.

Fireproof wall-board. F. S. VIVAS, Asst. to INTERNATIONAL FIREPROOF PROD. CORP. (U.S.P. 1,754,843, 15.4.30. Appl., 8.10.27).—Cellulose pulp is saturated with calcium chloride solution, a resinated soap solution is added, and the material is treated with a solution of alum or aluminium sulphate, and compressed into sheets.

H. ROYAL-DAWSON.

Emulsions of bitumens. N. BENDIXEN and J. D. MORGAN (B.P. 330,374, 7.5.29).—Tar or other bituminous substance is emulsified with water and a small proportion of fossil diatomaceæ, e.g., kieselguhr, and sufficient alkali is added to dissolve a part of the silica.

A. B. MANNING.

Wood preservation. H. W. WALKER (U.S.P. 1,753,000, 1.4.30. Appl., 26.10.25).—The wood is impregnated with an ammoniacal solution of arsenic trioxide and heated, the ammonia driven off being recovered.

H. ROYAL-DAWSON.

Device for testing constructional material by means of oscillations of flexure of test pieces. O. FÖPPL (B.P. 332,299, 24.4.29.)

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Oxides in pig iron: their origin and action in the steel-making process. ANON. (U.S. Bur. Mines, Bull. 308, 1929, 56 pp.).—The oxide content of iron from blast furnaces varied from nil to more than 0.13%, the higher quantities corresponding with irregular furnace working. Elimination of these oxides depends on their presence as silica or silicates, the quantities of FeO and MnO in the metal, temperature, viscosity of slag, and time effects. Additions of ore, cinder, or pig iron cause an increase in silicate content unless the viscosity of the slag is sufficiently high to support the added material until dissolved. The silica introduced in pig iron is in a form most difficult to eliminate, often remaining to the end of the heat and causing defects in forging due to the non-plasticity of the particles.

C. A. KING.

Effect of manganese on distribution of carbon in steel. B. M. LARSEN (U.S. Bur. Mines, Tech. Paper 466, 1929, 31 pp.).—Manganese increases the temperature range of γ -iron or austenite, forming a carbide more stable than cementite. Little diffusion of manganese through the iron space lattice occurs below 1100° , which is much above the heat-treatment temperature at which carbon diffuses freely. The dendritic patterns in cast steel containing more than 1% Mn are therefore not affected by the usual thermal treatment, but diffuse uniformly on heating the steel at 1300° for 1 hr. When a casting (1–3% Mn) is cooled at a rate slower than about 3° per min. carbon tends to segregate in the high-manganese fillings between the dendrites of lower manganese concentration. A marked banded structure occurs when the carbon content falls below 0.3%, aided also by the presence of phosphorus and arsenic. With increasing manganese a "manganese-cementite" tends to form, and with more than 2% Mn a fine-grained structure of this character may be distributed and linked together through the manganese atoms scattered through the iron space lattice.

C. A. KING.

Apparatus for demonstrating the "arrest points" of 0.9% carbon steel. R. C. GALE (J. Sci. Instr., 1930, 7, 165–166).—The extension of an electrically heated length of steel wire is magnified by a pointer moving over a graduated circle. C. W. GIBBY.

Effect of rate of cooling on the structure and constitution of steel. J. M. ROBERTSON (Safety in Mines Res. Brd. Paper No. 59, 1930, 57 pp.).—The effect of variation in the rate of cooling steel cannot be accounted for by the supposition that an increased cooling rate produces in turn sorbite, troostite, and martensite, as between these states many other different forms of structure are evident. It is suggested that the constitution of steel should always be considered in terms of the quantity of α -solid solution, γ -solid solution, and cementite present, and their relation with one another. Four series of structures may be obtained by different methods of cooling. The first includes all structures consisting of ferrite and pearlite or the latter alone. Again, continuous rapid cooling which suppresses the Ar1 and lowers the Ar2 point produces the series of quenched steels the structure of which may be varied considerably. When cooled so that the Ar1 point is suppressed and the rate then retarded so that the Ar2 change takes place at constant temperature, a third series results, and a fourth is similar except that the steel is withdrawn before the change is complete, the final change being effected by atmospheric cooling. The general arrangement of all ferrite-pearlite structures is determined by the manner in which ferrite develops from austenite, but the structure of the grains of pearlite is determined by simultaneously formed ferrite and cementite. The only structures which temper in a different manner are those which contain considerable quantity of α -solid solution, since, during reheating, decomposition of this α -phase occurs by the gradual separation of carbon as the temperature is raised and not by a sudden precipitation of fine carbon with subsequent coalescence to form larger globules.

C. A. KING.

Effect of low temperatures on the impact-resistance of steel castings. R. W. MOFFATT (Canad. J. Res., 1930, 2, 327–340).—The impact-resistance of cast iron, cast carbon steel, and alloy steels decreased considerably for temperatures below f.p., e.g., for normally cast steels the values obtained at -35° were only $\frac{1}{3}$ – $\frac{1}{2}$ those for atmospheric temperature. Heat treatment improved the resistance values both at ordinary and low temperatures. Vanadium steels have a much higher resistance than plain carbon steels, and at low temperatures compare favourably with heat-treated carbon steels tested at ordinary temperature. Nickel steel (2–3% Ni) showed similar values to 0.18–0.22% vanadium steels, and the highest impact-resistance was given by a steel (1.56% Ni, 0.1% V) which showed also only a slight decrease at the low temperatures.

C. A. KING.

Flow characteristics of special iron-nickel-chromium alloys and some steels at elevated temperatures. H. J. FRENCH, W. KAHLBAUM, and A. A. PETERSON (Bur. Stand. J. Res., 1930, 5, 125–183).—The results of creep tests at different temperatures are given for three groups of alloys. The first group included commercial iron-nickel-chromium alloys with or without tungsten, and low-chromium steels. The second comprised low-carbon steels and nickel steels, and was tested only at 370° . The alloys of the third group were castings of nickel-chromium-iron melted in the induction furnace, and were tested at temperatures above 540° . The addition of nickel to mild steel produced little change in the stress required to produce 1% elongation in 1000 hrs. Chromium additions were advantageous only when over 50%. With 14% Cr, corresponding to stainless steel, the creep stress was the same as for mild steel. The addition of small quantities of chromium to steels containing 40% Ni produced marked improvement. The highest creep stress was found in castings of about 50% Ni, 50% Cr. Wrought alloys containing 11–20% Cr and 8–40% Ni showed intercrystalline weakness at about 630° . No marked difference between coarse- and fine-grained samples of an 18% Cr, 8% Ni alloy was found below 538° . The best commercial alloy contained 20% Cr, 8% Ni, 4.5% W, 0.3% C, and 1% Si.

C. J. SMITHELLS.

Steel [gas] mains and corrosion. E. E. JEAUVONS and H. T. PINNOCK (Gas J., 1930, 191, 203–204, 255–256).—Extensive experiments on a large high-pressure gas-distribution system have shown how steel mains can be advantageously used and preserved. Although originally coated carefully with bituminous material, the mains showed corrosion after 5 years, and this grew rapidly in intensity. Electric currents were detected, particularly near tramways and similar installations, and corrosion always began at an anodic spot unless the covering were perfect. Also rapid changes in the direction and intensity of current were noted as the jointing material gave had contact between individual pipe sections. The corroded spot was almost invariably anodic and generally accompanied by a neighbouring intensely cathodic area. Preventive measures, adopted 10 years ago, have now been fully

justified by almost complete cessation of troubles, and recommendations for dealing with similar cases are made. Earth bars should be fitted at frequent intervals to the main, and the sections should be linked into one circuit by bonding with copper; alternatively, in laying fresh mains with welded joints the same result would be achieved.

R. H. GRIFFITH.

Rate of grain-boundary displacement in the primary and secondary recrystallisation of aluminium. G. TAMMANN and K. L. DREYER (Z. anorg. Chem., 1930, 191, 69—73).—In the primary recrystallisation of aluminium the isothermal growth of the grains follows the same law as with other metals (cf. A., 1930, 530). In the secondary recrystallisation, however, Karnop and Sachs' observation of a stationary rate of grain-boundary displacement (*ibid.*, 530) has been confirmed. It seems probable that the gradual decrease in the rate of primary recrystallisation is due to the separation from the crystallites of impurities which form resistant films and ultimately bring the grain-boundary displacement to a standstill. In the secondary recrystallisation, however, no further separation of impurities can occur, and therefore grain growth may be expected to proceed more readily, until, in the ideal case, a single crystal results.

R. CUTHILL.

Intercrystalline substance in lead and its effect on grain size. G. TAMMANN and K. L. DREYER (Z. anorg. Chem., 1930, 191, 65—68).—An investigation of the impurities present in various samples of lead has shown that the presence of even very small amounts of copper causes a considerable reduction in grain size. The relatively fine-grained character of the Pattinson lead examined by Brenthel (B., 1930, 286) is due to the presence of copper.

R. CUTHILL.

Bradford sulphur dioxide process [for lead-zinc slimes]. A. LOWRY (Proc. Austral. Inst. Min. Met., 1929, 207—232).—The application of the Bradford sulphur dioxide process for the selective flotation of lead and zinc concentrates depends chiefly on the regular addition of sulphur dioxide to slime pulp of definite density, the time factor for the absorption of the gas, and the control of sulphur dioxide subsequent to absorption. Under the conditions operating at the Broken Hill North mine the pulp contains approx. 0.0032% SO_2 as it enters the flotation machine; the limiting range for efficient work is 0.0021—0.0064% SO_2 . Excess of sulphur dioxide causes a partial loss of lead concentrate and difficulty in flotation of zinc blende, though the effects of an excess may be counteracted by adding potassium xanthate. The flotation agent is a mixture of equal parts of unrefined coal tar and eucalyptus oil.

C. A. KING.

Corrosion of rabbles in zinc concentrate roasting furnaces. J. N. GREENWOOD and A. J. ROENNFELDT (Proc. Austral. Inst. Min. Met., 1929, 99—120).—In the Barrier type of furnace working at 950—1000°, in which the rabble teeth are embedded in zinc concentrates containing about 6% of lead sulphide, considerable corrosion of the steel occurs particularly at the "wash" line, due to the attack of lead sulphide which is volatilised and condenses on the metal in the form of either bright columnar crystals or a light grey porous

scale which latter appears to exert a very corrosive action. The most resistant alloy contained 1.5—3% C, 26—27% Cr, and 0.5—1% Si, a low content of carbon being preferred. It is considered that in the primary attack of lead sulphide the iron chromium carbide is replaced by sulphides, and possibly by metallic lead, and the solid solution of iron, chromium, and carbon is unattacked. From a practical point of view any improvement in ore dressing which lowers the lead content will be reflected in a longer life of the rabbles.

C. A. KING.

Roasting of zinc ores. Y. OGAWA (Tech. Rep. Tôhoku, 1930, 9, 175—191).—The progress of roasting a clean zinc blende was examined by means of the Honda thermo-balance, the specimens heated consisting in some instances of one single crystal. At 550° the smaller size of grains began to lose weight, but those greater than 40-mesh only began to oxidise above 600°, the velocity rapidly increasing at 700—800°. From the roasting of a single crystal cut and ground to a true sphere it was deduced that the linear velocity of roasting is inversely proportional to the radius of the specimen, and that the time requisite for complete oxidation is proportional to the square of the radius. Since the oxide shell once formed has no fissures it seems to offer a uniform resistance to diffusion and to be amenable to Fick's law.

C. A. KING.

Zinc chloride flux used in the manufacture of tinplates. J. C. JONES (Trans. Faraday Soc., 1930, 26, 249—254).—The extents to which iron and tin are attacked by hydrochloric acid produced by the hydrolysis of zinc chloride solutions after 2 hrs. at 300° were measured from the amounts of metal in the flux. The amounts dissolved increase with the water content of the flux, and in the case of iron there is also evidence of oxidation to the tetroxide by water. Ferric chloride in the flux is reduced by metallic tin to ferrous chloride and/or iron, whilst ferrous sulphate is reduced to sulphide which is immediately converted into chloride. The cleaning of the steel surface in tinning with zinc chloride fluxes is due to the acid, whilst the accumulation of iron salts is prevented by the formation of a tin-iron alloy ("dross" or "hard tin") of high m.p. Spent flux contains stannic oxide and stannous chloride formed by replacement of iron from its compounds.

J. GRANT.

Properties of strontium-tin alloys. K. W. RAY (Ind. Eng. Chem., 1930, 22, 519—522).—A series of alloys was prepared by the electrolysis of a mixture of fused sodium and strontium chlorides over molten tin in a chromium-plated iron crucible. Alloys containing more than 30% Sr could not be prepared by this method as the m.p. was high, the fused salts volatilised, and the crucible oxidised seriously. Alloys containing 20—30% Sr were of the eutectic type, one component of which was Sn_3Sr , the other probably being SnSr . Intermetallic compounds represented by Sn_2Sr and Sn_3Sr were also found, the former being unstable above 338° and also very slow in formation, so that only a small quantity of Sn_2Sr was present in chilled castings. Cast alloys low in strontium are composed of long needle-shaped crystals of Sn_3Sr in a matrix of tin, which spheroidise more or less completely when annealed at above 338°. The hard-

ness of the alloys increases with increasing content of strontium, and cast alloys containing more than 18% Sr were brittle.

C. A. KING.

Analysis of white metals and solder. H. L. MATTHIJSSEN (Chem. Weekblad, 1930, 27, 284—287).—Slight modifications in standard methods, and particularly in simultaneous electrolytic determination of lead and copper, are described.

S. I. LEVY.

Treatment of Lake View and Star low-grade sulphide ore. W. G. CLARKE and B. H. MOORE (Chem. Eng. Min. Rev., 1930, 22, 324—326).—The ore contains 7.6 dwt. Au per ton and 4.9% of pyrites. By grinding through 150-mesh in a tube mill and passing the material over amalgamating plates, strakes, or Wilfley tables, 34, 64, and 58% of the gold was recovered respectively. Flotation of the tailings with 0.25 lb. of "Euco" and 0.25 lb. of xanthate per ton yielded a further 61.4, 30, and 36.7% of the gold, respectively, using a pulp ratio of 1:3 and either fresh or salt water. Thus the total recovery in all cases was about 95%. After roasting the flotation concentrates 90—95% of their gold content was readily extracted by cyaniding. Washing the roasted material with water prior to the cyanide treatment did not improve the recovery, but materially reduced the lime and cyanide consumption.

A. R. POWELL.

Ore concentration at the North Mount Farrell Mine, West Tasmania. J. E. COLDHAM (Chem. Eng. Min. Rev., 1930, 22, 320—322).—The ore contains 10—12% Pb as galena, 8—10% Zn as blende, 14 oz. Ag per ton, and 10% of iron pyrite in gangue consisting of slate, quartz, and carbonates. The ore is crushed through a $\frac{3}{8}$ -in. screen and then separated on $\frac{1}{4}$ -, $\frac{3}{8}$ -, and $\frac{1}{2}$ -in. screens. The oversize on each screen is concentrated separately in a series of Luhrig jigs to give a galena concentrate containing 60% Pb and 62 oz. Ag per ton, and the undersize and jig tailings are crushed to 60% through 200-mesh and passed to the flotation plant. Lead is floated with 0.2 lb. of "Aero-float," 0.25 lb. of cyanide, and 4.5 lb. of sodium carbonate per ton and the tailings then pass to the zinc cells, where they are floated with 0.2 lb. of xanthate and 1 lb. of copper sulphate per ton. The lead concentrate assays 60% Pb, 5% Zn, and 80 oz. Ag, per ton and the zinc concentrate 54% Zn, 6% Pb, and 13 oz. Ag per ton.

A. R. POWELL.

Flotation with xanthates. I. K. KELLERMANN and E. BENDER (Kolloid-Z., 1930, 52, 240—243).—The flotation effect of xanthates is not a property of the xanthate radical, but is due to the simultaneous effect of two products of hydrolysis. When carbon disulphide and alcohol were led simultaneously by means of a stream of air bubbles into a suspension of finely divided galena in water normal flotation occurred; a separation from zinc blende was also effected. For every alcohol a maximum flotation effect occurs at a certain dilution and the position of this maximum lies at a greater dilution with increasing number of carbon atoms in the alcohol. The best conditions were realised with butyl alcohol. Alcohols containing two or more hydroxyl groups are much less effective.

E. S. HEDGES.

Determination of the porosity of electro-deposits.

D. J. MACNAUGHTAN (Trans. Faraday Soc., 1930, 26, 465—481).—The influence on the corrosion at a discontinuity in a metal coating of such factors as the relative potentials of the two metals, the amount and composition of the electrolyte that covers them, the character of the cathodic and anodic products, and the ease of access of oxygen is discussed. A review of the methods suggested for testing porosity indicates that the ferricyanide test is the most suitable. This test is best made by means of paper dipped in a solution containing sodium chloride, potassium ferrocyanide, and ferricyanide: a rapid and permanent record of porosity tests may be obtained. The method has been found applicable to coatings of nickel, tin, chromium, copper, lead, silver, and gold on iron, steel, copper, and copper-base alloys. The test is unsuitable for coatings on a nickel base. The porosity of cathodic coatings on zinc, aluminium, and alloys of these metals is best determined by immersion of the specimen in a hot solution of sodium hydroxide, when attack of the basis metal occurs at the site of pores or discontinuities with the evolution of gas bubbles at these points.

F. G. TRYHORN.

Soldering tungsten. R. D'E. ATKINSON (Nature, 1930, 126, 97).—Gold, palladium, and zinc (as brass) can be used for soldering tungsten, using borax as a flux.

L. S. THEOBALD.

Use of micro-analysis in the streak test [for precious metal alloys]. R. STREBINGER and H. HOLZER (Mikrochem., 1930, 8, 264—270).—The streak test is made on a roughened depression in a microscope slide. When the alloy is chiefly silver the streak is dissolved in nitric acid on the slide, the silver precipitated as chloride and removed by centrifuging. The filtrate is evaporated to dryness, the residue dissolved in water, and 1 drop of potassium mercurithiocyanate solution added; green crystals indicate the presence of copper and brown crystals the presence of cadmium. For the examination of a gold streak it is dissolved in aqua regia, the metals are precipitated as sulphides by passing hydrogen sulphide through the solution from a capillary tube, the precipitate is collected in a micro-centrifuge, washed, digested with ammonium sulphide to remove the gold, and dissolved in nitric acid. Minute drops of the solution are tested for lead by addition of potassium acetate and nitrite (triple potassium lead copper nitrite), for bismuth with potassium sulphate (double sulphate), for palladium with dimethylglyoxime in acetic acid, nickel with the same reagent and ammonia, cobalt, zinc, and iron with ammonium mercurithiocyanate, and aluminium with alizarinsulphonic acid. Platinum streaks may be similarly tested for the presence of copper, palladium, and silver; gold is detected by the purple of Cassius test.

A. R. POWELL.

Process of failure of metals under stress. K. YUASA (J. Fac. Eng., Tokyo, 1930, 18, 271—345).—In a tensile-testing apparatus the load applied to the test piece is measured by means of a specially designed mirror extensometer having twice the sensitivity of the Martens instrument. At certain temperatures many irregular jumps occur in the curves after the so-called yield point is passed, and after the sudden decrease the

resistance increases uniformly until another discontinuity is reached. These breaks in resistance, reaching a maximum at about 200° and ending at about 340°, are termed "sudden yieldings" and are considered to indicate the instantaneous occurrence of slip, crack, rotation, or breaking of crystal grain in the test-piece or a combination of these effects, which may be regarded as a dangerous condition of failure not recorded by the usual tensile tests. No "sudden yieldings" occur in eutectoid steel, but the number increases with increase of carbon, and the observation of the number of sudden yieldings is suggested for the determination of the percentage of carbon and also for the effect of heat-treatment of metals. The relation between sudden yieldings and Fry's strain figures is also discussed.

C. A. KING.

Relationships between Rockwell and Brinell numbers. S. N. PETRENKO (Bur. Stand. J. Res., 1930, 5, 19—50).—The tensile strengths and the Brinell and Rockwell indentation numbers have been obtained for various ferrous and non-ferrous metals. Empirical formulæ relating Rockwell and Brinell numbers were found which give values differing by not more than 10% from the experimental results. The tensile strength of steels can be calculated from their Rockwell numbers, using empirical formulæ, with an error not exceeding 15%. No relation was found between the tensile strengths and indentation numbers of non-ferrous metals.

C. J. SMITHELLS.

Centrifugal concentration. DOERNER.—See I.

PATENTS.

Annealing dishes. W. SHAW, and W. SHAW & Co., LTD. (B.P. 328,506, 8.7.29).—Two plates are connected together by a series of ribs, thus forming longitudinal channels through which the heating medium is conducted. The external faces may be grooved in line with the ribs, and the dish is reversible if buckling occurs during use.

C. A. KING.

Annealing [of car wheels]. G. S. EVANS, Assr. to GRIFFIN WHEEL Co. (U.S.P. 1,745,362, 4.2.30. Appl., 6.2.22).—The annealing furnace is in the form of a tunnel kiln divided longitudinally into three parts. The central passageway is heated along a portion of its length and the carriers are pushed through the heating zone, then through a slow-cooling zone, and finally travel through one of the side tunnels which serves as a more rapid cooling zone.

C. A. KING.

Pickling of iron and steel. I. H. DERBY, Assr. to P. C. REILLY (U.S.P. 1,729,097, 24.9.29. Appl., 30.8.28).—An inhibitor for use in pickling baths comprises the reaction product of phosphorus pentasulphide with ammonia or its organic substitution derivatives, or with an aliphatic or aromatic alcohol, or with a hydrocarbon of the naphthalene or anthracene series.

A. R. POWELL.

Metal-coating [galvanising] metal [iron or steel] sheets. E. R. WEHR and C. C. MAHLIE, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,741,388, 31.12.29. Appl., 13.9.26).—Iron or steel sheets are coated with an alloy of zinc with 3—20% Al by passing them through a zinc bath floating at one end of a bath of molten lead, thence

through the lead bath upwards into a bath of molten zinc-aluminium alloy, and finally through hot rolls.

A. R. POWELL.

Protection of iron, copper, and their alloys. G. NOBILLEAU and J. GUIPET (B.P. 308,778, 1.2.29. Fr., 28.3.28).—The articles are packed tightly in a box filled with a mixture of 40% of powdered zinc, 52% of powdered silica, 3% of potassium ferrocyanide, and 5% of barium carbonate. The box is heated at 400° for 1½ hrs., allowed to cool, and emptied. The articles are then similarly packed in a mixture of 50% of powdered zinc, 45% of powdered silica, and 5% of barium carbonate, and heated at 550° for 2 hrs. After cooling, the zinc-coated articles are cleaned and polished with a mixture of fine sand and petrol.

A. R. POWELL.

[Beryllium-nickel-iron] alloy. SIEMENS & HALSKE A.-G., and W. KROLL (B.P. 306,035, 9.2.29. Ger., 14.2.28).—Alloys of iron with 0.1—12% Be, up to 0.2% C, and nickel in excess of the beryllium are claimed. Up to 25% of the iron may be replaced by one or more of the elements copper, chromium, tungsten, molybdenum, vanadium, manganese, silicon, or phosphorus. The alloys are quenched from 800—1200° and aged at 300—700°. An alloy of 71.3% Fe, 7.1% Ni, 20.1% Cr, 1.2% Be, and 0.3% Mn after quenching has a Brinell hardness of 279, which increases to 524 after ageing at 500° for 1 hr. [Stat. ref.] A. R. POWELL.

Manufacture of [cutting steel] alloys. DEUTS. EDELSTAHLWERKE A.-G. (B.P. 311,267, 8.2.29. Ger., 8.5.28).—The alloy comprises iron with 1—1.5% C, up to 2% Cr, 2—18% Co, and 10—30% W. Molybdenum or vanadium may replace part or all of the chromium, and with 25—30% W the alloy may contain up to 2.5% C. [Stat. ref.] A. R. POWELL.

Production of dark oxide coatings on magnesium and its alloys. B. JIROTKA, Assr. to DR. O. SPRENGER PATENTVERWERTUNG JIROTKA M.B.H. (U.S.P. 1,747,776, 18.2.30. Appl., 27.5.29. Ger., 19.4.29).—The metal articles are immersed in a hot bath containing 100 g. of manganese sulphate and 100 g. of sodium dichromate per litre, whereby they become coated with a dark brown to black adherent coating of manganese dioxide.

A. R. POWELL.

Manufacture of bodies for tools and the like from materials such as carbides, the alloys thereof, and the like. K. SCHRÖTER and H. WOLFF, Assrs. to F. KRUPP A.-G. (U.S.P. 1,757,846, 6.5.30. Appl., 10.6.29. Ger., 15.6.28).—See B.P. 313,619; B., 1930, 616.

Material [flux] for low-temperature soldering of metals. J. A. ROGNON (U.S.P. 1,759,279, 20.5.30. Appl., 20.7.29. Fr., 24.7.28).—See B.P. 316,196; B., 1930, 773.

Electric furnaces (B.P. 332,015—6). **Coating organic articles** (B.P. 331,863).—See XI.

XI.—ELECTROTECHNICS.

Conversion of methane etc. into acetylene. PETERS and FRANSCHKE.—See II. **Steel mains and corrosion.** JEAVONS and PINNOCK. **Porosity of electro-deposits.** MACNAUGHTAN.—See X.

PATENTS.

Electric furnace. R. M. CHERRY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,775,051, 15.4.30. Appl., 1.3.28).—A heating resistor secured to hook members is pivotally fastened to refractory members secured together.

J. S. G. THOMAS.

[Tubular heat-treatment] electric furnaces. R. M. CATTERSON-SMITH (B.P. 332,015—6, 7.3.30).—(A) A heating tube rests in a trough-like recess formed in the bottom refractory lining of a box-like casing having a hinged or removable lid lined with refractory material. An inwardly-projecting flange is formed on or attached to one side wall of the bottom of the casing for the terminals of the heating tube, and connexions from the tube to these terminals are arranged in recesses in the lining, so that on opening the lid the tube can be lifted out after removing the connexions. (B) Billets are fed to a heating tube by a push rod reciprocated by a chain preferably moved forward against the action of a weight or spring by a solenoid or electromagnet.

J. S. G. THOMAS.

Electric annealing furnace. HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 331,793, 19.11.29. Ger., 15.12.28).—In electric annealing furnaces having a cantilever-arm charging-machine, heating resistances, arranged at right angles to the direction of the spaces for the introduction of the arms, are placed in recesses in the furnace hearth below the lowest working position of the arms of the machine.

J. S. G. THOMAS.

Manufacture of electrically insulating material. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 309,505, 11.4.29. Ger., 11.4.28).—A film of lacquer removed from the support, upon which it has been dried at ordinary temperatures, is dried at above 150°, *e.g.*, 200—270°, and further thin films of lacquer are applied thereto and similarly treated.

J. S. G. THOMAS.

Plastic [insulating] composition. A. R. KEMP, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,753,746, 8.4.30. Appl., 3.12.23).—Finely-divided fused silica is heated at 900—1000° for not less than 1 hr., whereby adsorbed and occluded gases are removed and a thin film of crystalline silica (tridymite) is formed on the surface of the individual particles, thus preventing readsorption of gases. This material when used as filler for rubber insulating materials etc. confers high water-resistance and stable electrical characteristics.

S. S. WOOLF.

Electrolysis of molten substances. I. G. FARBENIND. A.-G. (B.P. 309,605, 12.4.29. Ger., 14.4.28).—Electrolysis is carried out between electrodes of heaped carbonaceous material which are maintained separate and can be continuously renewed. Thus one (or more) electrodes is guided and maintained in place by a shaft or shafts widened towards the bottom and extending almost to the surface of the molten bath.

J. S. G. THOMAS.

Preparation of organic articles for electrolytically covering them with a metallic layer. I. AINSTEIN (W. I. EINSTEIN) (B.P. 331,863, 9.4.29. Addn. to B.P. 300,060; B., 1929, 25).—A thin preliminary metal-

lisation with easily fusible alloy is applied from a "gun," followed, if necessary, by pulverisation with copper or its alloys, and finally by electroplating in a bath at 10—30°, before the main plating operation.

J. S. G. THOMAS.

Manufacture of electrodes for secondary cells, in particular those with alkaline electrolytes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,540, 4.1. and 17.6.29).—The active mass, *e.g.*, nickel, is introduced into the pores of a coherent, porous material composed of iron, nickel, or cobalt, produced from the corresponding metal carbonyl, and/or ferrosferic oxide similarly produced. Alternatively, metallic hydroxides or oxides may be precipitated within the pores, and salts washed out by boiling. The hydroxides or oxides are then heated in a reducing atmosphere.

J. S. G. THOMAS.

Fixing the depolarising mass to salt-containing carbon elements for electric batteries. A. KUNZE (B.P. 332,155, 24.10.29).—A small percentage of resin, *e.g.*, less than 5% of coumarone resin, is added to the depolarising mass, which is heated for a short time above the m.p. of the resin.

J. S. G. THOMAS.

Reclaiming battery-plate material. W. C. SMITH, Assr. to PRIMOS LEAD Co. (U.S.P. 1,752,356, 1.4.30. Appl., 14.1.27).—Lead compounds are separated from the material by crushing and screening, and dissolved in a solution of caustic soda. Impurities are precipitated by treatment with sponge lead, and the solution is electrolysed to precipitate spongy lead.

J. S. G. THOMAS.

Manufacture of [cathodes for] photoelectric cells. GEN. ELECTRIC Co., LTD., and N. R. CAMPBELL (B.P. 332,002, 27.5.29).—A cathode consisting of a thin film of caesium or rubidium deposited upon an oxidised metal, *e.g.*, silver, is sensitised by an electric glow-discharge through a rare gas, *e.g.*, helium, at a pressure of 1 mm.

J. S. G. THOMAS.

Prevention of electrical leakage [in electrical discharge devices containing an alkali metal]. E. A. LEDERER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,752,748, 1.4.30. Appl., 14.9.26).—Glass near the leading-in wires is coated with a non-conducting, non-hygroscopic compound which, under the conditions existing in the discharge device, converts any deposited alkali metal into a stable, non-conducting compound which does not adversely affect the operation or life of the device. Thus a mixture of potassium and sodium nitrates may be placed near the wall of a discharge tube containing caesium vapour.

J. S. G. THOMAS.

Electrical precipitation plants. LODGE-COTTRELL, LTD. From H. V. WELSH (B.P. 331,699, 11.7.29).—In electrical precipitation plants comprising a number of units in series, flues controlled by valves are provided so that on closing down any units gases to be treated can be by-passed from the inoperative units either to the next operative unit or to the delivery outlet from the series.

J. S. G. THOMAS.

Electrical purification of gases. F. BARTLING (B.P. 309,149, 5.4.29).—Gas is passed in a thin layer between smooth, substantially parallel, stationary or

moving electrodes, preferably not more than 4 in. apart, the surfaces upon which deposition occurs being constantly cleaned by the stream. J. S. G. THOMAS.

Electrical precipitator [for cleaning gases]. G. E. McCLOSKEY, Assr. to BARRETT CO. (U.S.P. 1,752,920, 1.4.30. Appl., 10.2.27).—Means are provided whereby any liquid condensed in the chambers surrounding the insulators supporting the electrodes drains away immediately into the body of the precipitator, whereby short-circuiting is prevented.

J. S. G. THOMAS.

Forming copper-copper oxide rectifier plates. W. T. ANDERSON, JUN., and L. F. BIRD, Assrs. to HANOVIA CHEM. & MANUFG. CO. (U.S.P. 1,757,284, 6.5.30. Appl., 30.8.28).—See B.P. 328,701; B., 1930, 672.

Electric [baking] ovens. MOORWOODS, LTD., and H. WHEALE (B.P. 331,593, 9.10.29).

Manufacture of dollies for dry batteries. H. A. BUMKE G.M.B.H. (B.P. 332,103, 17.8.29. Ger., 26.7.29).

Production of carbon dollies for galvanic cells. H. A. BUMKE G.M.B.H., and AUTOM. SPEZIAL-MASCHINEN G.M.B.H. (B.P. 332,101, 14.8.29. Ger., 26.7.29).

[Cut-out for] electric incandescence lamps. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 332,164, 11.11.29. Ger., 13.11.28).

Electron-discharge tubes, and insulating means therefor. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. B. WANTZ (B.P. 314,341, 21.6.29. U.S., 25.6.28).

Anodes for electric-discharge devices. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. S. THELEN (B.P. 316,561, 30.7.29. U.S., 30.7.28).

Removing soot etc. from smoke (B.P. 307,805).—See I. **Carbon granules** (B.P. 329,652). **Dehydration of emulsions** (U.S.P. 1,754,009).—See II. **Lacquer composition** (U.S.P. 1,754,243).—See XIII. **Rubber compositions** (U.S.P. 1,752,531).—See XIV.

XII.—FATS; OILS; WAXES.

Examination of oils by the extended capillary diagram and the analytical quartz lamp. C. A. ROTHENHEIM and L. LETTENMAYER (Pharm. Ztg., 1930, 75, 862; cf. Rapp, B., 1929, 110).—A number of fatty oils in the presence of various solvents have been examined by these means, the methods of colour comparison having been improved. Cod-liver oils alone gave a characteristic yellow fluorescence. A solution of irradiated ergosterol in sesame oil ("Vigantöl") shows no typical colour. E. LEWKOWITSCH.

Decomposition of oils and fatty acids by heating with active nickel on a carrier. H. I. WATERMAN and M. J. VAN TUSSENBROEK (Chem. Weekblad, 1930, 27, 146—147; cf. B., 1929, 861; 1930, 66).—The hexabromide value of soya-bean oil, which is unaltered after the usual treatment for deodorising, is reduced by heating in a vacuum to 225°; the effect is greater in presence of kieselguhr, and in presence of nickel mounted on kieselguhr the treatment reduces the value to zero. The acid value is increased in the same order. Oleic and elaidic acids are stable to this

treatment, whilst linoleic acid and its glyceride are attacked. S. I. LEVY.

Twitchell reagent. Effect of addition of (II) acids, and (III) salts and variation of conditions, on the fat-splitting power of the reagents and on the darkening of fatty acid. K. NISHIZAWA and Y. MATUKI (J. Soc. Chem. Ind., Japan, 1930, 33, 179—182 B, 182—184 B; cf. B., 1929, 946).—II. The addition of the weak formic or acetic acids, although they did not much influence the emulsifying power (cf. *loc. cit.*), very markedly reduced the degree of hydrolysis obtained with Idrapid, Pfeilring, or Kontakt reagents. The fat-splitting power of all these reagents was greatly increased by the addition of strong acids (oxalic, sulphuric, and hydrochloric). Increased concentration of the added mineral acid increased the hydrolysis in the case of the Idrapid solution, but Kontakt and Pfeilring, which were salted out, gave extraordinarily irregular curves. The intensity of colour of the acids produced was calculated by a formula (involving the acid value) from the observed colorimeter readings: the depth of colour was nearly in direct proportion to the amount of oil saponified, and was practically the same whether sulphuric or hydrochloric acid was added; oxalic acid produced about half the intensity of colour. Under the conditions employed Idrapid was the most satisfactory reagent for fat-splitting power, colour of the fatty acids, and separation of the glycerol water.

III. The addition of normal salts such as sodium chloride or sulphate, or the sodium salts of the reagents themselves, greatly reduced the hydrolysing action, which could only be restored by the addition of large amounts of strong acids. The addition of glycerol or of free fatty acids similarly reduced hydrolysis. The increased hydrolysis effected by strong acids is explained by (1) the saponifying action of the acid itself, (2) the increased emulsifying power (cf. *loc. cit.*), and (3) the prevention of the formation of salts of the reagents. The influence of temperature and agitation on hydrolysis was considerable: hydrolysis at 100 r.p.m. by Idrapid, Kontakt, and Pfeilring was only 29.8, 43.7, and 8.7%, respectively, of that at 500 r.p.m., although variations were only slight if the stirring was kept at the latter rate.

E. LEWKOWITSCH.

Japanese fish oils. II. M. HIROSE (J. Soc. Chem. Ind., Japan, 1930, 33, 172—173 B).—The principal physical and chemical characteristics of the following oils: sardine, herring (iodine values 96—110), plaice, salmon, sand-eel, cod-liver, Alaska pollack-liver, and common ray-liver, from various districts are given. Sardine oil from Japan proper had higher iodine value, sp. gr., and n_D than those from Chosen (cf. B., 1930, 247). Alaska pollack-liver oil closely resembles cod-liver oil, but the iodine value, sp. gr., and n_D of the former are usually lower than the corresponding values for the latter.

E. LEWKOWITSCH.

Higher unsaturated alcohols from certain drying oils. S. KAWAI (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1930, 13, 254—259).—Reduction of tung oil with sodium and amyl alcohol in presence of toluene gave cetyl alcohol, m.p. 49.5—50°, together with an unsaturated alcohol, $C_{18}H_{34}O$, b.p. 200—205°/9.5 mm.,

containing two ethylenic linkings. Sardine oil similarly yielded cetyl alcohol, and a fraction, b.p. 220–230°/7 mm., which on catalytic hydrogenation yielded "dihydrolaganol," $C_{20}H_{41}OH$, m.p. 66–67·5°, oxidised by sodium dichromate in acetic acid solution to dihydrolaganic acid, m.p. 68·5–70° (Kotake, A., 1927, 1215), whilst linseed oil gave cetyl alcohol, and an unsaturated alcohol, b.p. 196–201°/6 mm., converted by hydrogen and platinum-black into stearyl alcohol, m.p. 59°.

A. I. VOGEL.

Testing for rancidity of fats. J. DAVIDSOHN (Chem. Umschau, 1930, 37, 193–196).—The Kreis rancidity test and its various modifications are discussed: the author's original experiments confirm the conclusions of the German Fat Analysis Commission and of the A.O.C.A. Kreis Test Committee, that none of these reactions can be considered entirely reliable.

E. LEWKOWITSCH.

Neutralisation and refining of Spanish olive oils. M. MASRIERA and A. PAR (Anal. Fís. Quím., 1930, 28, 771–783).—The methods available for the removal of free fatty acids from olive oil in order to fit it for food are reviewed and discussed. The usual method of treatment with alkali is commercially impracticable when the amount of acids exceeds 25% and impossible when the amount exceeds 50%. Processes of distillation, particularly with superheated steam or other vapours, are likely to be applicable to low-grade olive oils. The possibility of esterifying the fatty acids with glycerol is discussed, and preliminary experiments are described in which the amount of free acid in low-grade oil (from rind) is reduced from 70% to 10% by esterification at 120° in presence of sulphonated fatty acids ("divulson") as catalyst. The formation of emulsions, however, causes trouble in the subsequent separation.

R. K. CALLOW.

Volumetric determination of fatty acids in soaps etc. J. E. HEESTERMAN (Chem. Weekblad, 1930, 27, 434–436).—The soap or soap powder is dissolved and decomposed by means of hydrochloric acid in the butyrometer, a few drops of saponin are added, the whole is centrifuged, and the volume of separated fatty acids read. For accurate results it is necessary to determine the density of the acid layer.

S. I. LEVY.

Milk-in-fat emulsions. EICHSTÄDT; MOHR and EICHSTÄDT. **Butter fat.** LANA. **Olive oil in sardines.** WEWERS.—See XIX.

PATENTS.

Preventing development of rancidity in vegetable oils. IMPERIAL CHEMICAL INDUSTRIES, LTD., and H. J. TATTERSALL (B.P. 331,814, 7.3.29).—The castor oil etc. used in leather-cloth manufacture is treated with sufficient peracetic acid to convert the more unsaturated constituents (e.g., trilinolein) into polyhydroxy-poly-acetoxy-derivatives.

E. LEWKOWITSCH.

Preparing oil, fat, lard, tallow, etc. for storage or transit. M. M. BURMAN (B.P. 309,502, 11.4.29).—A homogeneous mixture prepared by melting the oil etc. with a proportion (e.g., 10–15%) of the same fat which has been hydrogenated is suddenly cooled to

below the m.p., yielding a solid compact product which does not melt in hot weather. E. LEWKOWITSCH.

Production of fats. T. P. HILDITCH (B.P. 331,880, 11.3.29).—Edible synthetic fats resembling lard or tallow are prepared by esterifying mixtures of saturated (40–60%) and unsaturated acids, such as are obtained by the saponification of vegetable oils or fats, or their mixtures, with a deficiency of glycerin; the excess (10–20%) of fatty acids is removed by the usual methods.

E. LEWKOWITSCH.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Plasticisers for acetylcellulose. CARSWELL.—See V.

PATENTS.

Production of painting oils of improved drying capacity. J. SCHEIBER (B.P. 306,453, 9.2.29. Ger., 20.2.28).—Fatty acids of good drying oils, e.g., tung or perilla oil, or isomeric linoleic acids with conjugated double linkings produced from ricinoleic acid (cf. B.P. 306,452; B., 1930, 726) are mixed with fatty acids from semi- or non-drying oils and the mixture is esterified with glycerin by heating at about 200° for 5–6 hrs., or a mixture of oils and fatty acids (in excess) may be transesterified by heat, and the excess acids esterified subsequently by glycerin: excess of glycerin is removed by distillation or by esterification with resin etc.

E. LEWKOWITSCH.

Non-tarnishing metallic ink. W. F. SMITH, ASSR-TO KINGSFORD PRESS, INC. (U.S.P. 1,752,462, 1.4.30. Appl., 16.11.27).—Powdered metal (2 pts.) is stirred slowly into a solution of nitrocellulose (pyroxylin 1 pt.) until the metallic particles are completely coated, and the solvent is then driven off in such a way as to leave the coated metal in powdered form, e.g., by spraying. The powder is then mixed with rosin and terpeneol, or equivalent vehicle.

E. W. LEWIS.

Stencil sheet. S. HORII (U.S.P. 1,753,204–5, 8.4.30. Appl., [A] 4.8.27, [B] 31.12.27).—(A) To prevent absorption of the coating composition, the paper is made from material having maximum felting and grease-proofing qualities. This is achieved, e.g., (1) by hammering mulberry (Kozo) bast tissue for about 5 hrs. until the degree of hydration, as shown by the Schopper-Riegler beating tester, reaches 40–45 units, or (2) by beating "Mitsumata" or "Gampi" bast tissue for 20 min. in a beating engine, followed by wet beating for about 5 hrs. until the hydration reaches 50–60 units. The beaten stuffs may be blended, and the colloidal mucilage "Neri" is added to them. The paper is coated with an ink-resisting composition including waxes or a gelatinous organic colloid, or cellulose esters, combined with a suitable tempering agent. (B) The fibrous base, such as Japanese "yoshino" paper, is coated or impregnated with a solution of esters of polysaccharides (other than cellulose and starch acetates), such as starch stearate, mannan acetate, cellulose stearate or nitrate, in a suitable solvent (e.g., acetone, alcohols), mixed with a suspension of an insoluble colloid such as aluminium tannate or hydroxide, titanium white, or kaolin in a tempering or softening

agent (hydrocarbon or fatty oils, fats, fatty acids, esters, etc.). Dyeing the insoluble colloids with a colloidal colouring matter exerts a protective action.

E. W. LEWIS.

Production of plastic materials for use in manufacture of lacquers, covering materials, binding agents, etc. FRANKFURTER GASGES, and H. W. HOELZER (B.P. 331,846, 4.4.29).—Coal tar, lignite tar, coumarone resin, etc. are heated under pressure in the presence of a catalyst, *e.g.*, iron chlorides, manganese sulphate, caustic soda, sulphuric acid, paraformaldehyde, with a sulphurous extract formed by extracting spent gas-purification materials with, *e.g.*, the tar oil or tar-oil vapour from the tar employed in the process, or carbon disulphide. Such mixtures as 15–25% of bitumen and asphalt and 85–75% of tar may also be vulcanised as above.

S. S. WOOLF.

Coating materials and their preparation. IMPERIAL CHEM. INDUSTRIES, LTD., and B. W. FOSTER (B.P. 331,837, 3.1.29).—A water-wet cellulose ester or ether is kneaded with a medium which includes gelatinisers, *e.g.*, "cellosolve," ethyl lactate, butyl acetate, chloroform, softeners, *e.g.*, raw or blown castor, linseed, or tung oil, and diluents, *e.g.*, industrial spirit, toluol, kerosene, and the water set free is removed, practically without evaporation. Pigments (dry or water-wet), resins, etc. may also be incorporated. Improved gloss of final product is claimed.

S. S. WOOLF.

Coating compositions containing *m*-styrene combined with softeners. E. I. DU PONT DE NEMOURS & Co. (B.P. 311,700, 3.4.29. U.S., 14.5.28).—Mixtures of *m*-styrene, softeners, *e.g.*, butyl phthalate, solvents, etc. are claimed.

S. S. WOOLF.

Coating compositions. E. I. DU PONT DE NEMOURS & Co. (B.P. 312,049, 3.4.29. U.S., 18.5.28).—Compositions containing polymerised vinyl compounds, *e.g.*, a vinyl chloride or acetate polymeride, and tetrahydrofurfuryl alcohol are claimed. Pigments etc. may be incorporated.

S. S. WOOLF.

[Urushiol] lacquer composition. T. SATOW (U.S.P. 1,754,243, 15.4.30. Appl., 23.1.26).—The milky liquid product from the "lacquer plant" (*Rhus vernicifera*) containing urushiol is heated with sulphur at 120–160° for 1–3 hrs., a thick mass semi-liquid at 100° being formed. This is heated with aldehydes and fillers are incorporated for use as impregnating insulating material. For coating purposes, solvents, *e.g.*, benzene, benzol, acetone are added.

S. S. WOOLF.

Lacquer and varnish thinners. CARBIDE & CARBON CHEM. CORP. (B.P. 304,759, 21.12.28. U.S., 27.1.28).—In liquid coating compositions containing natural and/or synthetic resins, and, if desired, a cellulose ester and a solvent therefor other than amyl or butyl esters, *e.g.*, "cellosolve," the use of ethylbenzene or its higher homologues is claimed. [Stat. ref.]

S. S. WOOLF.

Manufacture of pressed or moulded articles [from cellulosic materials]. H. P. BAYON. From MAGNASCO ROGGERO & Co. (B.P. 331,851, 8.4.29).—"Formed" cellulosic materials, *e.g.*, wood shavings, tree barks, woven fabrics, are treated with a dilute

caustic alkali at elevated temperature and pressure, washed and dried, treated with an aqueous oxidising acid solution, *e.g.*, a mixture of sulphuric and nitric acids, impregnated with a solution containing a synthetic resin, *e.g.*, phenol-formaldehyde, treated with *p*-dichlorobenzene with or without a hardening agent, *e.g.*, hexamethylenetetramine, and then pressed or moulded and heated sufficiently to "cure" the resinous material. The final product shows the original texture of the cellulosic material employed.

S. S. WOOLF.

Production of moulding compound. H. S. SNELL, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,752,580, 1.4.30. Appl., 8.11.24).—A water-insoluble proteinate, *e.g.*, aluminium caseinate, and an insoluble salt capable of waterproofing the compound, *e.g.*, aluminium oleate, are precipitated simultaneously and jointly by a common precipitant, *e.g.*, aluminium acetate, the precipitate is washed and dried, and furfuraldehyde is added.

S. S. WOOLF.

Improving aldehyde resins. W. W. GROVES. From CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 314,399, 26.6.29).—Aldehyde resins are combined with polymerised vinyl compounds and mixed with filling materials in the manufacture of articles, *e.g.*, gramophone records, by pressure or fusion. Improvement in toughness, freedom from bubbles in the molten state, and higher capacity for taking up fillers are claimed.

S. S. WOOLF.

Plates of artificial resin [with intermediate veneer]. F. OEHNING (B.P. 331,870, 5.4.29).

Processes and apparatus for surfacing sheet material with pigments, varnishes, lacquers, etc. G. M. BROWN (B.P. 332,190, 15.2. and 21.5.29).

Grinding mills (B.P. 330,923).—See I. **Coating compositions** (B.P. 326,515).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spanish rubbers. J. G. VIANA and M. TOMEO (Anal. Fis. Quím., 1930, 28, 709–717).—Analytical and physical data are given of samples of rubber (i) from the Canary Islands, and (ii) from Guinea. Sample (i), probably derived from *Euphorbia balsamifera*, Hors. Kew, contained 27% of caoutchouc and 56% of resins; it could not be vulcanised and resembled chicle gum in properties. Sample (ii), from *Landolphia* sp., contained 12% of caoutchouc and 58% of resins; it is used under the name "Almedina" as an addition to certain rubber mixings. Neither product is commercially important.

R. K. CALLOW.

Isoprene and caoutchouc. XXI. **Molecular size of caoutchouc and the nature of its colloidal solutions.** H. STAUDINGER (Kautschuk, 1930, 6, 153–158).—Dilute solutions of rubber or balata, like polystyrene solutions, contain macro-molecules and not micelles. The macro-molecules of rubber contain about 1000 isoprene nuclei in a long chain, the molecular length being responsible for the characteristic physical features. The high viscosity and other properties of the "gel solutions" of rubber are the result of insufficiency of space for free movement of the long molecules.

D. F. TWISS.

Caoutchouc molecule or caoutchouc micelle? H. KROEPELIN (Kautschuk, 1930, 6, 158—159).—The smallness of the influence of temperature on the viscosity of rubber renders unlikely the presence of any considerable proportion of micelles or molecular aggregates in equilibrium with simple molecules in the solutions. There are undoubtedly micelles present, although possibly not strictly of the Nägeli type. Only in stretched rubber do they become crystalline, and their number increases with increasing extension. The molecules in a micelle remain together on relaxation of the tension and also when rubber is dissolved in a solvent.

D. F. TWISS.

PATENTS.

Treatment of guayule etc. Extraction of rubber from guayule etc. D. SPENCE, Assr. to INTERCONTINENTAL RUBBER Co. (U.S.P. 1,753,184—5, 1.4.30. Appl., (A) 8.5.26, (B) 12.5.26).—(A) Deterioration of the rubber contained in plants such as guayule is prevented by treating the plants after harvesting and before extraction of the rubber, and either before or after crushing, with the vapour or dilute solution of an organic nitrogenous stabilising agent, *e.g.*, dimethyl-*p*-phenylenediamine, *p*-phenylenediamine, piperidine, or condensation products of amines with aldehydes etc. The product may be stored without deterioration. (B) Deterioration of the rubber from guayule etc. during extraction, whether mechanically or by means of solvents, is prevented by carrying out the process in presence of an organic stabiliser (see above). The rubber "worms" produced are devoid of tackiness, and can be baled without sheeting. The product is readily deresinised by acetone, alcohol, or caustic alkali.

E. W. LEWIS.

Manufacture of rubber derivatives. I. G. FARBENIND. A.-G. (B.P. 313,919, 13.6.29. Ger., 19.6.28).—Rubber derivatives (intended to find application in the manufacture of dyestuffs and pharmaceutical products) are obtained by the interaction of an alkyl- or aryl-sulphur halide, or a substitution product thereof, with natural or artificial rubber, preferably in presence of an indifferent solvent. For example, a solution of *p*-nitrophenylsulphur chloride (3 pts.) in carbon tetrachloride (50 pts.) gives, when mixed with a solution of rubber (1 pt.) in carbon tetrachloride (400 pts.), a quantitative yield of an additive product which, when dry, forms a bright yellow amorphous powder sintering at about 135°.

E. W. LEWIS.

Manufacture of artificial rubber. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,272, 29.12.28).—A diolefine is polymerised in water emulsion by the action of an emulsifying inorganic salt of a high-molecular non-amphoteric organic base. Examples of such bases are: oleic β -diethylaminoethylamide, oleic *p*- β -diethylaminoethoxyanilide, 2-*n*-pentadecylglyoxalidine (from ethylenediamine and the imino-ether of palmitic acid), and menthyl β -diethylaminoethyliminodicarboxylate, $\text{NEt}_2 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{N}(\text{CO}_2 \cdot \text{C}_{10}\text{H}_{19})_2$. These are used as hydrochlorides, and produce a synthetic rubber latex.

C. HOLLINS.

Manufacture of rubber-like masses. I. G. FARBENIND. A.-G. (B.P. 307,308, 4.3.29. Ger., 3.3.28).—

A butadiene, *e.g.*, $\alpha\gamma$ -butadiene, isoprene, $\beta\gamma$ -dimethylbutadiene, etc., is polymerised in two stages, a further quantity of the hydrocarbon preferably being added after the first stage, and polymerisation being completed by means of a different agent. Thus isoprene is partly polymerised with sodium at 35°, then dissolved in more isoprene, emulsified in sodium oleate solution, and polymerised at 60° to a rubber latex, which may be coagulated in the usual manner.

C. HOLLINS.

Manufacture of factice and the like. J. BAER (B.P. 313,917, 5.6.29. Switz., 19.6.28).—Sulphuretted oils, especially factice soluble in hydrocarbons, are prepared by vulcanising the oils with the caoutchouc-like mass obtained by the action of alkali or alkaline-earth polysulphides on saturated halogenated hydrocarbons (*e.g.*, methylene dichloride) (cf. B.P. 279,406, 302,399, and 298,889; B., 1929, 220, 652).

E. LEWKOWITSCH.

[Rubber] composition [for submarine insulation]. F. S. MALM, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,752,531, 1.4.30. Appl., 20.12.28).—Raw rubber or latex is heated with a 1% solution of sodium chloride for some hours at above 100° (about 150°), washed, and dried. This treatment is said to hydrolyse and render soluble some of the protein and to coagulate and render less water-absorbent other portions. The product is preferably used in admixture with, *e.g.*, commercial deresinated balata and montan wax (35 : 35 : 30). The heat-treated rubber has a dielectric constant of 2.61 as compared with 3.0 for good commercial gutta-percha, and is stated to be electrically stable when immersed in water.

E. W. LEWIS.

Manufacture of rubber articles. B. W. ROWLAND, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,757,928, 6.5.30. Appl., 10.11.28).—See B.P. 329,897; B., 1930, 729.

XV.—LEATHER; GLUE.

Mode of action [on pelt] of pickles of different compositions. A. KÜNTZEL (Collegium, 1930, 218—233).—Pickling solutions exercise two functions, viz., the completion of the deliming and neutralisation of the pelt which is still slightly alkaline from the bating liquor, and the characteristic pickling effect whereby the pelts absorb a definite amount of acid. When collagen is placed in hydrochloric acid-salt pickles, some of the acid is combined with the collagen and some taken up by capillary absorption; the maximum amount of combined hydrochloric acid was 0.7 mol. per kg. of dry collagen. The amount of combined acid was only slightly affected by the salt content of the pickle. The acid swelling was greatly reduced by the salt content of the pickle, and in highly concentrated salt solutions the swelling due to the salt was reduced by even small amounts of acid. Acid swelling was diminished by small amounts of salt, but only completely repressed by large amounts of salt. The collagen was swollen as much in pickles of high salt concentration as in water. It is shown that the repression of the acid swelling depended not on the ratio of acid to salt, but on the actual salt concentration. No acid swelling was observed if the salt concentration was greater than 5 mols. of sodium chloride per kg. of dry collagen, independent

of the presence of much or little acid. The swelling in collagen, which was either acid- or alkali-swollen before pickling, was only partly repressed by treatment with highly concentrated pickles. D. WOODROFFE.

Preparation and properties of raw collagen. A. KÜNTZEL and K. BUCHHEIMER (Collegium, 1930, 205—217).—The flesh and hair sides of a fresh cowhide were removed by splitting and the remaining pelt was split into an upper layer 0.35 mm. thick and two other layers each 0.95 mm. thick, all of which were allowed to dry in a ventilated warm room, when they became hard, horny, yellowish, and transparent. All epidermal structures, hair muscles, flesh muscles, and most of the blood vessels were removed with the flesh and hair side splits. Certain non-collagenous components, e.g., interfibrillary tissue cells, blood vessels, fat, and ash, are present in the above three layers. The amount of water absorbed by dry collagen is less than that present in the original collagen, and the thicker dry collagen absorbed less water on soaking than the thin. The loss in weight at 105° was thick split 20.75%, thin split 19.62%, dry collagen ground to powder 14.08%. Analysis showed C 50.24, H 6.58, N 17.75, fat 0.33, ash 0.41%. Washing the dry collagen for 88 hours removed 0.13% of mineral matter (ash). By treating the collagen first with 10% salt solution and then with saturated lime water for different periods of time and extrapolating the results it was shown that the dry splits contained 1% of non-collagenous proteins. 1 G. of the air-dry collagen combined with 7×10^{-4} mol. of hydrochloric acid, which gives an equivalent of 1142, the same as for gelatin. D. WOODROFFE.

Determination of chromium, iron, and aluminium in chrome[tanned] calf leathers. H. B. MERRILL and R. C. HENRICH (J. Amer. Leather Chem. Assoc., 1930, 25, 270—275).—Low chromium figures and high iron and alumina results were obtained by precipitating the latter with ammonia from the product obtained by fusion of the leather ash with borax, potassium carbonate, and sodium carbonate. The errors are reduced to 0.03% on a 3-g. leather sample by redissolving the iron and aluminium precipitate in acid and reprecipitating with ammonia. The iron and aluminium may be separated by the sodium peroxide method; an accuracy of 0.02% is obtained by the procedure detailed. The cupferron method was much less accurate. D. WOODROFFE.

Influence of splitting on the strength and stretch of commercial leathers. R. C. BOWKER and E. S. OLSON (J. Amer. Leather Chem. Assoc., 1930, 25, 275—290).—The breaking strength of grain splits was considerably less than was anticipated from the thickness, whilst that of the flesh splits was in the same proportion to that of the unsplit leather as were their relative thicknesses. The tensile strength of the grain splits in lb./in.² was only about 25% of that of the unsplit leathers, whereas that of the flesh splits was 80—106%. The stretch (%) of the grain splits at a definite stress was much greater than that of either the flesh splits or the unsplit leather. The stretch (%) of both flesh and grain splits at breaking point was less than that of the unsplit leathers. The stitch-tear resistance of the

grain splits was 10—20% and of the flesh splits was 40—80% of that of the unsplit portions.

D. WOODROFFE.

Birch bark [*Betula alba* and *B. pubescens*] as a tanning material. P. JAKIMOV and P. TOLSKI (Collegium, 1930, 233—239).—The following reactions are given by birch-bark tannin: bromine water, yellow precipitate; ferric chloride, black precipitate; gelatin, copious precipitate; sulphuric acid, red ring changing to reddish-brown on dilution with water; lead salts, rose-coloured precipitate; formaldehyde, rose-coloured precipitate and colourless filtrate. The outer and inner barks contained tans 2.4, 8.52% and non-tans 3.3, 8.8%, respectively. The percentage of tannin in the inner bark increased with the age of the tree from 5.4% (20 years) to 10.6% (80 years). The proportion of tans to non-tans diminished with increase in height from which the bark was derived. The optimum temperature of extraction was found to be 90°. The material was improved for subsequent leaching by preliminary leachings at 10° or 20°. A liquid extract of p_H 4.57 was prepared and used to tan hides into leather, which had the properties of an upper leather with elastic grain, no spring, and a light brown, rose-tinted colour. About 450,000 tons of birch bark containing 30,000—35,000 tons of tannin are available annually in European Russia. D. WOODROFFE.

PATENTS.

Unhairing hides and skins. H. M. ULLMANN and E. R. THEIS (U.S.P. 1,753,539, 8.4.30. Appl., 26.12.28).—Hides or skins are soaked for about 20 hrs. or less in water containing pancreatin (0.02 to 0.1%), at 60—80°, and are then limed until the hair is loosened (about 20 hrs.). The hair is removed in the usual way. E. W. LEWIS.

Leather and its production. A. EHRENREICH and R. TANDLER (B.P. 331,241, 26.3.29).—Soaked skins of marine and terrestrial animals, e.g., shark, are treated to fix the scales or shagreen, e.g., with 2% soda and $\frac{1}{2}$ % formaldehyde solution, then treated with solutions of ammonium chloride or hydrochloric acid to dissolve the mineral constituents in the scales or shagreen, after which they may be given a quick preliminary tanning with astringent materials, and finally subjected to an intensive tannage of long duration.

D. WOODROFFE.

Leather product. C. G. SHAW (B.P. 331,263, 19.2.29).—Leather is impregnated with rubber cement, an ultra-accelerator (e.g., piperidine pentamethylenedithiocarbamate, zinc ethylxanthate), and sulphur, the solvent removed by evaporation under vacuum at a temperature sufficiently low to retard vulcanisation, and the impregnated leather subsequently vulcanised under heat and pressure. D. WOODROFFE.

Titanium solutions (B.P. 309,090).—See VII.

XVI.—AGRICULTURE.

Comparison of elutriators for the separation of sands, silts, and slime in easily worked soils. E. BRIQUET (Arch. Sci. phys. nat., 1930, 12, 161—178).—The advantages and disadvantages of the Kopecky and

Crook elutriators are discussed. A modification, having some advantages over the latter form, is described. C. W. GIBBY.

Fluctuations of bacterial numbers and nitrate content of field soils. H. G. THORNTON and P. H. H. GRAY (Proc. Roy. Soc., 1930, B, 106, 399—417).—Bacterial counts have been made and the nitrate contents determined in samples taken every 2 hrs. from a field soil. Significant diurnal variations were found and there was some evidence that different groups of bacteria show different fluctuations. The fluctuations appear sometimes to be correlated with rainfall, but not with the moisture content of the soil nor with the nitrate content, although the latter also appears to fluctuate during the day. W. O. KERMACK.

Actinomycetes in Danish soils. H. L. JENSEN (Soil Sci., 1930, 30, 59—77).—A systematic survey of the types and chemical activities of actinomycetes is recorded. The highest numbers of the organisms occurred in soils of pH value 6.8—8.0. A. G. POLLARD.

"Single value" soil properties. Significance of certain soil constants. III. Technique of the Keen-Raczowski box experiment. J. R. H. COUTTS. IV. Technique of the "box" experiment. B. A. KEEN (J. Agric. Sci., 1930, 20, 407—413; 414—416, cf. B., 1929, 486).—III. A simplified technique for the box experiment is described. By its use values for pore space are about the same as those obtained by the older method; the old and new values for water retention and swelling are not the same, but are related by a simple linear expression.

IV. A note on the effect of compression on "single value" soil properties as determined by the "box" method. Such inert material as precipitated silica swells when saturated with moisture. E. HOLMES.

Occurrence of elementary carbon in soils. G. W. ROBINSON and W. MCLEAN (J. Agric. Sci., 1930, 20, 345—347).—The presence of free carbon from coal or coke in soils introduces an appreciable error in the determination of organic matter by organic carbon analyses. A mechanical sedimentation method of removing at least 75% of the elementary carbon is proposed. E. HOLMES.

Carbon-nitrogen ratio of soil organic matter. W. MCLEAN (J. Agric. Sci., 1930, 20, 348—354).—The C:N ratio of 50 British soils varied from 6.5 to 13.5:1 (average about 10:1), and of 16 foreign samples 2.0 to 23.0:1. The ratios for arable soils do not differ appreciably from those of grassland soils, both elements being in higher amount in the grassland soils. Although fertile soils usually contain relatively high amounts of carbon and nitrogen, there is no marked correlation between C:N ratios and fertility. It is suggested that in the same areas total organic carbon or total nitrogen might be used as an index of fertility. The C:N ratio decreases in descending a soil profile. E. HOLMES.

Significance of the carbon:nitrogen ratio, and other chemical properties of organic materials in the action [in soils]. O. LEMMERMAN, W. JESSEN, and H. ENGEL [with H. BORTELS, VON SCHMELLING, and

W. LESCH] (Z. Pflanz. Düng., 1930, 17A, 321—355).—The course of the decomposition and humification of organic materials (straw, long straw manure, etc.) is accompanied by definite variations in chemical properties, notably the C/N ratio, nitrogen and pentosan contents, and the "humus" content as determined by means of hydrogen peroxide or ammonia. The decreasing rate of carbon dioxide production as decomposition proceeds is not sufficiently characteristic for assessing the extent of humification of these materials. During decomposition the ash and nitrogen contents of the material increase and the C/N ratio decreases. The attainment of a C/N ratio of 20:1 during decomposition corresponds with a 50% loss of dry matter, and a nitrogen content in the product of 2% or more. The degree of humification of such material (soluble in 2% ammonia solution) is more than 30%, and its pentosan content is less than 12%. When this stage of decomposition is reached the material no longer causes plant injury when incorporated with soil, as a result of the temporary withdrawal of available nitrogen by micro-organisms. A. G. POLLARD.

Carbon and nitrogen cycles in the soil. I. H. J. PAGE. II. Extraction of organic matter of soil with alkali. C. W. B. ARNOLD and H. J. PAGE. III. Formation of natural humic matter. M. M. S. DU TOIT and H. J. PAGE (J. Agric. Sci., 1930, 20, 455—459, 460—477, 478—488).—I. The term "humic matter" is defined as the dark-coloured, high-molecular, colloidal organic matter which is a characteristic constituent of the soil, and "non-humic matter" includes colourless, chiefly soluble organic substances and undecomposed plant residues.

II. Although the total organic carbon in the soils of various plots of the classical permanent experiments at Rothamsted receiving, respectively, organic, artificial, and no manures, varied between 0.81 and 2.91%, and in the subsoils between 0.54 and 1.04% of the oven-dry samples, there was a marked similarity between the properties of their organic matter, especially in its behaviour on extraction with cold and hot dilute caustic soda. Colorimetric examinations of the extracts indicate that the organic carbon of the surface soils is more deeply coloured than that of the corresponding subsoils, that the organic carbon is most deeply coloured in extracts from surface soils receiving annual dressings of dung, and that that from subsoils of plots receiving no manure is least coloured.

III. Decomposition experiments in which soil extracts and nutrient salts were added to plant materials such as wheat straw, clover hay, maize cobs, and pine sawdust, and to purified preparations of plant constituents, including lignin, cellulose, xylan, xylose, potato starch, dextrose, and protein in the form of commercial blood fibrin, indicate that the formation of humic matter is more closely related to the change in lignin content of the original material than to the change in content of any other groups of plant constituents estimated. E. HOLMES.

Phosphorus-fixing compound in the soil. A. H. MEYER (Science, 1930, 71, 461).—An iron compound

existing as concretions in southern soils and responsible for the fixation of phosphorus has been discovered. The phosphorus is present as a basic ferrous phosphate of very low solubility. L. S. THEOBALD.

Separation of two products of solubilisation of sulphur in a soil rich in organic matter. G. GUITTONNEAU and J. KEILLING (Compt. rend., 1930, 191, 277—279).—The formation of thiosulphuric and pentathionic acids during the oxidation of sulphur in a soil rich in organic matter (peptone) (cf. B., 1927, 150, 587) is proved by their isolation as the corresponding bismuth potassium and potassium salts from an aqueous extract. H. BURTON.

Determination of exchangeable sodium in soils. R. WILLIAMS (J. Agric. Sci., 1930, 20, 355—358).—The amounts of exchangeable sodium extracted from soils by 0.5*N*-acetic acid and by *N*-ammonium chloride, and determined by the zinc uranyl acetate method (Barber and Kolthoff, A, 1928, 859), show good agreement. The experimental procedure for determining the sodium in acetic acid extracts is detailed.

E. HOLMES.

Determination of potassium in soil samples by the application of an X-ray method. J. T. CALVERT (Trans. Faraday Soc., 1930, 26, 509—514).—Potassium in soil samples and in minerals may be determined by mixing with the sample a known quantity of manganese oxide and comparing the intensities of the potassium $K\alpha$ - and the manganese $K\beta$ -lines excited by X-rays of shorter wave-length. An accuracy within 5% may be obtained by short exposures; increased accuracy follows more prolonged exposure. For minerals it is advisable to replace manganese oxide by cadmium oxide and to compare the potassium $K\alpha$ - and cadmium $L\beta$ -lines. F. G. TRYHORN.

Measurement of carbon dioxide evolution from soil. H. HUMFELD (Soil Sci., 1930, 30, 1—11).—The Lundegårdh "respiration bell" for determining respired carbon dioxide is modified by replacing the glass bell with a rectangular metal box having inlet and outlet tubes at opposite sides. The carbon dioxide produced by the soil is constantly removed by a measured current of air and trapped in standard caustic soda solution.

A. G. POLLARD.

Determination of available phosphoric acid in soils. S. DAS (Soil Sci., 1930, 30, 33—48).—The author's potassium carbonate method of extracting available phosphate from soils (B., 1926, 456) is successfully applied to a wide range of soil types, including alkali and calcareous soils with which Dyer's citric acid method fails. Cropping and fertiliser trials confirm the validity of the results obtained. A. G. POLLARD.

Variations in soil acidity, especially in Heinrichauer soils. E. PAASCH (Z. Pflanz. Düng., 1930, 9B, 309—326).—The effects of lime and various fertilisers on soil reaction are examined. Green manures, but not farmyard manure, increased soil acidity, and this effect was additive when physiologically acid fertilisers were used conjointly. A. G. POLLARD.

Carbon dioxide-bicarbonate and water extracts as bases for the determination of the phosphate requirement of soils. B. DIRKS and F. SCHEFFER

(Landw. Jahrb., 1930, 71, 74—99).—Solvents commonly used for extracting the "available" phosphate from soils have much lower p_H values than has the root sap of plants. For this purpose the authors utilise a calcium bicarbonate solution saturated with carbon dioxide for neutral and alkaline soils, and carbon dioxide-free water for acid soils. The phosphate content of a single extract serves as a measure only of the temporary condition. The total assimilable phosphate is determined by means of 4 or 5 successive extracts. The amount of phosphate removed by a single extract shows a definite relationship to Neubauer values. A. G. POLLARD.

Effect of a "soil mulch" on the quantity of water lost from a given soil by evaporation. E. S. WEST (J. Council Sci. Ind. Res., Australia, 1930, 3, 97—105).—The mulching of soil produced by digging to a depth of 10 cm. increased the quantity of water retained by the undisturbed soil layers.

A. G. POLLARD.

Relations between potash fertilisation and the effect of light. O. LEMMERMANN and H. LIESEGANG (Z. Pflanz. Düng., 1930, 9B, 256—268).—Comparison is made of the growth of plants variously fertilised and shaded to different extents. Potash, more than other nutrients, increases the ability of plants to utilise the diminished light under shaded conditions. Although the effect of potash fertilisers in increasing crop yields reaches a maximum in full daylight, it cannot be proved that the crop increases are the outcome of an increased utilisation of light by the fertilised plants.

A. G. POLLARD.

Crop variation. VII. Influence of rainfall on yield of barley at Rothamsted. J. WISHART and (Miss) W. A. MACKENZIE (TYRRELL). **VIII. Application of the resistance formula to potato data.** R. J. KALAMKAR (J. Agric. Sci., 1930, 20, 417—439, 440—454).—VII. A statistical examination of the effects of each inch of rainfall over the average at various parts of the year on the yields of barley on plots under different manurial treatments from 1852 to 1921.

VIII. A further examination of the validity of the resistance formula as applied to crop yield statistics (cf. B., 1928, 906).

E. HOLMES.

Effect of different fertilisers on the composition of the crop and the structure of the leaves of barley and wheat. K. BOEKHOLT (Z. Pflanz. Düng., 1930, 9B, 289—300).—The leaf area of wheat and also the density of the crop are largely controlled by the potash supply. Potash tends to reduce leaf size and nitrogen to increase it, whilst phosphate has but slight effect. The effects of the principal nutrients on the anatomical structure of the plants are systematic. Increased leaf area corresponds with larger stomata and larger vascular bundles, but smaller numbers of stomata per unit leaf area. A. G. POLLARD.

Nitrogen fixation in field soil under different conditions of cropping and soil treatment. L. A. BRADLEY and J. E. FULLER (Soil Sci., 1930, 30, 49—57).—The distribution and activity of a species of *Azotobacter* in soil treated with various fertilisers and carrying various crops is examined. The nitrogen fixation and distribution of the organism were practically unin-

fluenced by the nature of the crop or changes in soil reaction. The organism tolerated lower p_H ranges than those usually accepted as limiting values for *Azotobacter* growth.

A. G. POLLARD.

Influence of sodium nitrate and ammonium sulphate on the intake of potash and phosphate [by plants] from the soil. M. GRAČANIN (Z. Pflanz. Düng., 1930, 9B, 300—309).—The variable effects of ammonium sulphate on the resorption of phosphate and potash by plants in Neubauer tests is largely attributable to differences in soil reaction. In soils with $p_H < 6.0$ ammonium sulphate decreases the intake of phosphate, and to a small extent that of potassium. With $p_H > 6$ the phosphate intake increases. In soils having no reserve of calcium carbonate ammonium sulphate reduces the extent of root development. In all cases sodium nitrate reduces root size, the decrease being the greater in soils which, initially, had the higher reserves of available nitrogen. These results do not detract from the value of Neubauer tests provided the soil reaction is examined and due consideration given to its effect on the resorption of nutrients. There is no positive correlation between the available nitrogen content of soil and the nitrogen intake by plants.

A. G. POLLARD.

Sodium salts used in conjunction with potassium salts as plant nutrient. VI. Summer and winter rape, kohl-rabi, and horse beans. H. JACOB (Z. Pflanz. Düng., 1930, 17A, 355—392).—An extension of earlier investigations (Heinrich, B., 1928, 343) on the effect of sodium sulphate in increasing the potash absorption of various crops. The translocation of potash from straw to grain in cereals and from haulm to tuber in potatoes caused by sodium sulphate occurs in the later stages of plant growth and closely resembles the transference of nutrients from the dying parts of plants. The substitution of sodium for potassium in active plant organs cannot definitely be proved.

A. G. POLLARD.

Phosphate studies in culture solutions. J. W. TIDMORE (Soil Sci., 1930, 30, 13—31).—Water cultures of maize, sorghum, and tomatoes with varying concentrations of phosphate in the nutrient are described. The rate of absorption of phosphate by the plants was not directly proportional to the phosphate concentration of the nutrient. The rate of growth increased with the phosphate concentration throughout the growing period. Concentrations of 0.5 p.p.m. PO_4 produced maximum growth with all plants examined. With 0.2 p.p.m. PO_4 maize and sorghum produced 71% and tomatoes 42% of maximum growth. Plants grew better in displaced soil solutions with 0.02—0.03 p.p.m. inorganic PO_4 than in culture solutions with 0.1 p.p.m. PO_4 . The phosphate content of plants and plant sap increased with that of the nutrient. The tomato plants had a higher percentage of phosphorus than maize or sorghum, but a low PO_4 content in the leaf sap. Phosphates play a minor rôle as buffer agents in the plant sap. Maize and wheat plants absorbed phosphate more rapidly from acid nutrient solutions than from alkaline ones, but there was little difference in the rate of absorption over the p_H range 4—6.

A. G. POLLARD.

Causes of the beneficial effect of brown coal on the growth of crops. A. KISSEL (Brennstoff-Chem., 1930, 11, 257—260).—The effect of brown coal in promoting the growth of corn etc. is due, not to its inorganic constituents, but to (a) its mechanical action on the soil, (b) the carbon dioxide evolved by its slow decomposition, (c) the production of organic acids which render available the otherwise insoluble salts containing phosphorus, and (d) the decreased thermal conductivity of the soil due to which the optimum growing temperature is maintained therein for longer periods. The amount required to produce these favourable results, however, makes it uneconomical to use the raw material for this purpose.

A. B. MANNING.

Method of increasing the manurial value of bone phosphate. N. D. VYAS (Bull. Imp. Inst. Agric. Res., Pusa, No. 204, 1930, 21 pp.).—Composts were prepared with bone dust 4 pts., sulphur 1 pt., and sand 8 pts., together with added moisture and either with or without cultures of sulphur-oxidising organisms or soil containing the same. It was found that in 23 weeks the citrate-soluble phosphate, originally 5% of the whole, had risen to from 75% with uninoculated compost to 92% with compost inoculated with the culture. In absence of sulphur little change occurred. Losses of nitrogen in the same period in presence of sulphur did not exceed 10%, and ammoniacal nitrogen nitrified more rapidly than when sulphur was absent. Charcoal was added to the composts to remove the offensive odour produced, but was found also to increase the rate of solubilisation of phosphate. A series of trials on wheat, potatoes, and *marua* are described in which the value of this compost was found in some cases to be equal to superphosphate, in others better, and in most cases very considerably better than untreated bone meal. Bone meal could be used in preparing the compost in the same way as bone dust.

C. IRWIN.

Decomposition of keratin by soil micro-organisms. H. L. JENSEN (J. Agric. Sci., 1930, 20, 390—398).—Keratin from horn meal, when added to moist field and garden soils, slowly decomposed, 35—40% of its nitrogen being transformed into nitrate after 120 days. The addition of keratin produced little or no increase in the numbers of bacteria, but markedly increased the numbers of actinomycetes, especially in garden soil. Two strains of actinomycetes were isolated and found capable of thriving on keratin in pure culture to give ammonia.

E. HOLMES.

Modification of the citrate method [for the determination of soluble phosphate in superphosphate]. A. SUCHIER (Z. angew. Chem., 1930, 43, 672—673).—The arrangement of an apparatus for collecting and automatically washing with the correct amount of wash water the phosphate precipitate obtained by the method previously described (B., 1929, 992) is illustrated and briefly described.

A. R. POWELL.

Fungicidal properties of spray fluids. VII. W. GOODWIN, H. MARTIN, and E. S. SALMON (J. Agric. Sci., 1930, 20, 489—497).—Continuing earlier investigations

(cf. B., 1930, 632) on the fungicidal properties of sodium, potassium, calcium, and barium polysulphides against the conidial stage of hop powdery mildew, it is shown that the nature of the spreader may influence the concentration at which polysulphide sulphur attains fungicidal strength. Samples of potassium and sodium polysulphides had the same fungicidal power when applied at the same concentration of spreader and polysulphide sulphur. Solutions of calcium polysulphides which differed widely in the relative amounts of total and thiosulphate sulphur behaved as fungicides in accordance with the polysulphide sulphur content only, and, in general, it is concluded that the efficiency of polysulphide sulphur is independent of the base with which it is combined.

E. HOLMES.

Standardisation of Schweinfurth green for plant treatment. G. HILGENDORFF (Z. angew. Chem., 1930, 43, 648—650).—Differences in ease of suspension of various samples are completely masked when the preparation is mixed with preparations of chalk and copper sulphate, as is usual in making up suspensions for spraying. The fineness of the sample is therefore of much less importance than the hydrolysis value as measured by the amount of arsenic taken into solution under standard conditions. The German official specification is given.

S. I. LEVY.

"Dry pickling" or "dusting" seed wheat to prevent bunt. G. H. PETHYBRIDGE and W. C. MOORE (J. Min. Agric., 1930, 37, 429—439).—Co-operative trials by advisory mycologists in England and Wales on seed wheat contaminated with bunt spores indicate that 2.5% copper sulphate solution has a slightly higher fungicidal efficiency than formalin or copper carbonate, but the yields of grain favoured the copper carbonate treatment.

E. HOLMES.

***Stibaropus tabulatus*, Schiö (Hem., Pent.), a new pest of tobacco in S. India.** P. N. K. AYYAR (Bull. Entom. Res., 1930, 21, 29—31).—Crude oil emulsions (1:6) effectively controlled *S. tabulatus* without injury to the plants.

A. G. POLLARD.

The onion maggot (*Hylemyia antiqua*) in Ohio, 1929. M. P. JONES (J. Econ. Entom., 1930, 23, 394—398).—Onion crops were increased by 45% after spraying with Bordeaux-oil emulsion and with proprietary oil emulsions against the onion maggot.

A. G. POLLARD.

Plant juice clarification for nitrate nitrogen determinations. H. H. HILL (Science, 1930, 71, 540).—Details are given for the clarification of maize juice by means of silver sulphate and carbon black.

L. S. THEOBALD.

Method of estimating the yield of a missing plot in field experimental work. F. E. ALLAN and J. WISHART (J. Agric. Sci., 1930, 20, 399—406).

PATENT.

Drying processes [for grass], apparatus therefor, and products thereof. H. A. N. DELLOW, C. M. WRIGHT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 327,409, 29.9.28).—In drying and compressing grass crops into cakes that will not swell on keeping, the

grass is dried within 6—12 hrs. after cutting, and should not be more than 6 in. long, the maximum air temperature is 200°, and the air when hottest is brought in contact with the grass when wettest. The velocity of the air through the grass should not exceed about 15 ft./sec., and the moisture should not exceed 15%, preferably 12—14%, to prevent swelling after compression. A pressure of 5—10 tons per in.² should be maintained for 15—20 sec., or longer time lower pressure. The cake thus produced usually has sp. gr. about 0.8. Suitable apparatus is described. B. M. VENABLES.

XVII.—SUGARS; STARCHES; GUMS.

Sugar losses in beet-sugar factories. II. Recovery of sugar from factory waste products. H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1930, 80, 239—263; cf. B., 1930, 478).—The usual loss of 0.25% of sugar and 0.25% of non-sugar (calc. on beets) in the waste liquors from the diffusion battery and the pulp presses can be entirely avoided by returning the liquors to the diffusion battery after they have been freed from pulp. Only a few German factories follow this practice, but in the Dormagen factory the author has successfully applied it, in one form or another, for 25 years. The essentials are efficient de-pulping of the water, its rapid return to the battery, preferably within 15 min., and avoidance of occasions for frothing. At Dormagen the water from the pulp presses is passed through a pulp-remover and then mixed with the waste waters from the battery, the mixture being raised by a pump and passed through a second pulp-remover, after which the necessary amount of fresh water is added and the whole pumped back into use. A closed settling vessel of 5 cub. m. capacity is installed on the pressure side of the pump. The whole of the diffusion waste waters must be returned, for they become richer in sugar than ordinary waste waters, and much of the economy of the process would be lost by running part of the water to waste. The greater part of the non-sugar recovered by this process is retained by the pulp. The recovered soluble matter which passes into the juice has, after carbonation, a purity quotient corresponding to that of a normal after-product massecuite, viz., 70—80%. The necessary plant is not costly, its operation is not difficult, and with normal care the water should not be subject to injurious decomposition. A method of recovering most of the sugar usually lost in carbonation cakes is also described (cf. B., 1929, 490). This loss is seldom less than 0.12% of the beets, and can be reduced to 0.04—0.05% without extra dilution of the juice. The process has been worked for 2 years in the Dormagen factory, where the scum cakes retain an abnormal amount of sugar. Cakes from the first and second carbonation filters are mixed with hot water in worm conveyors below the filters and carried to mixers where a mash having \bar{d} 1.08—1.10 is produced. This is pumped to settling tanks 1 m. high in which it is kept for 1½—2 hrs. at 85°. The upper liquor is then drawn off and used for sweetening-off subsequent filter-press charges. The sludge is run into second clarifiers in which it is again mashed with hot water and allowed to subside. The sugar recovered has a purity of 84—85%, and therefore

lowers the purity of the thin juice less than the usual sweetening-off waters. The first cost and running costs are small. If the final scums are required as pressed cakes, filter presses can be used in place of settling tanks.

J. H. LANE.

Fall in purity and polarisation of beet-sugar juices during evaporation. G. CAPELLE (Bull. Assoc. Chim. Sucr., 1930, 47, 155—184).—The purity quotient of beet juices diminishes by 0.4—1.2% in the course of evaporation, whether this is carried out under pressure or in evaporators of the older type with temperatures below 108° in the first body. Of this fall in purity, not more than 0.31 can be ascribed to increase of density due to evolution of ammonia and carbon dioxide, and not more than 0.2 to the effect of heat on the optical rotation of the non-sugars. Assuming a total fall of 1%, at least 0.49 of this must accordingly be due to decomposition of sucrose, representing a loss of 0.08% of sugar, calculated on beets.

J. H. LANE.

Diffusion losses [in beet-sugar manufacture], and the Naudet "plus sugar." J. BOTHY (Bull. Assoc. Chim. Sucr., 1930, 47, 110—119).—Extraction of 200-g. lots of fresh cossettes with successive portions of hot water at 85—95° in a closed metal container immersed in a water-bath indicated, on polarisation of the extracts and the exhausted slices, total sugar contents 0.1—0.8% (on beets) in excess of those found by analysis of the pulped beets by the Pellet and Sachs-Le Docte methods. The excess amounts were larger with freshly harvested roots than with those which had been stored. It is suggested that they may represent sugar actually formed from reserve substances during extraction, by living cells in the cossettes, under the influence of heat and pressure, as claimed by Naudet for his modified diffusion process with high initial temperatures. Pulped beets similarly extracted gave no higher results than by the usual methods of analysis.

J. H. LANE.

Determination of the adhesive power of starch pastes. SPROCKHOFF (Chem.-Ztg., 1930, 54, 411—412).—Ekhard's method (B., 1929, 298) is adversely criticised owing to the time taken in making the test and the great effect of temperature variations on the results. Much more accurate figures are obtained by viscosity measurements.

A. R. POWELL.

Sugar-soluble silica. SPENGLER and TRÄGEL.—See VII.

PATENTS.

Defecation of sugar juice. A. W. BULL, Assr. to DORR Co. (U.S.P. 1,752,781, 1.4.30. Appl., 26.4.27).—Sugar juice is treated with defecating agents and the sedimentation process controlled by regulating the rate of agitation so that complete flocculation and the maximum size of the individual flocks are obtained.

C. RANKEN.

Stabilisation and preservation of inulin in vegetable material. SCHERING-KAHLBAUM A.-G. (B.P. 309,195, 6.4.29. Ger., 7.4.28).—The vegetable material which contains the inulin is made into a paste with an anti-fermentation agent such as carbon monoxide, hydrogen cyanide, potassium cyanide, or a fluorine compound.

C. RANKEN.

XVIII.—FERMENTATION INDUSTRIES.

Influence of shaking and temperature on acid production and nitrogen assimilation during fermentation. W. WINDISCH, P. KOIBACH, and E. SCHILD (Woch. Brau., 1930, 47, 327—331, 337—340).—Laboratory fermentations each of 500 c.c. of wort, with exclusion of air, were compared under various conditions and against brewery fermentations. The best, though not perfect, agreement with the latter was obtained by fermenting at 9°, with a thorough shaking every 24 hrs. The shaking hastens all the changes studied. At temperatures above 9° the products of yeast autolysis obscure the course of nitrogen assimilation after a very few days. Ammoniacal nitrogen is less readily used than formol nitrogen, but is assimilated more rapidly from worts poor in formol nitrogen than from more nitrogenous worts. There is a marked relationship between the assimilation of ammonia and acid formation, and the production of ammonia in the later stages of fermentation at higher temperatures is accompanied by decrease of titratable acidity and rise of *pH*. Volatile acidity increases most rapidly during the first few days; its production is favoured by low temperatures, and its amount is not decreased, as is that of fixed acidity, by the ammonia resulting from yeast autolysis. The production of fixed acidity is most marked at relatively high temperatures.

F. E. DAY.

Brewing value of barley and malt. F. KUTTER (Woch. Brau., 1930, 47, 309—314, 321—324).—Determinations are made of the extract, moisture, and protein in the barley, and values are allotted to these factors according to their closeness to certain fixed standards. The sum of these values is designated as the "chemical brewing value." Similarly, the "mechanical brewing value" is calculated from the values awarded for the 1000-corn weight, the size of the corns, and the amount of refuse. Marks given for the germinative energy are included in the physiological brewing value, whilst the valuations of colour, quality of husk, etc. are classified under the heading of "general brewing value." The total sum of these values is given as the brewing value of the barley. In the evaluation of malts, the determinations of the 1000-corn weight and the germination are replaced by those of the length of acrospire and the modification. The standards of valuation can be modified as desired, and any of the separate factors can be given a weighted value.

C. RANKEN.

Presence and determination of acetaldehyde in wines. E. CHARLES (Ann. Falsif., 1930, 23, 153—154).—Determinations in various samples of wine by the hydroxylamine hydrochloride and the Schiff's reagent methods gave concordant results, the amount of aldehyde varying from 0.11 g. to 0.75 g. per litre. Wines containing more than 0.5 g. per litre were judged unfit for consumption, and it is suggested that this substance should be tested for in all doubtful wines. The aldehyde is formed by oxidation of the alcohol of the wine.

A. SHORE.

Determination of alcohol [in wines etc.]. E. MARTIN (Ann. Falsif., 1930, 23, 154—156).—A reply to Semichon and Flanzky (B., 1929, 449) as to priority. The simplest method of carrying out this oxidation is

to collect the distillate directly in the warm chromic acid solution, when the alcohol is oxidised completely to acetic acid. The method is applicable to 1 c.c. of wine. A. SHORE.

Desiccation of potatoes and manufacture of alcohol therefrom. E. LÜHDER, B. LAMPE, and W. KILP (Z. Spiritusind., 1930, 53, 191—193, 199—200).—The washed potatoes are macerated and pressed so that 2 pts. of potatoes yield 1 pt. of potato juice and 1 pt. of potato pulp containing 60% of moisture. To prevent the potato starch forming a paste during drying, the temperature of the drying plant is not allowed to exceed 50° during the first 3 hrs., and thereafter is gradually raised to 100°. Desiccation is completed in 6 hrs., and yields potato flakes containing 14% of moisture. Both time and heat are saved by the prior separation of the juice, which contains in solution salts, protein, and a small amount of starch. The juice is used afterwards as the mashing liquor, and any surplus is run into the distillery slop. The replacement of the mashing water by juice permits a low mashing temperature, and as the mash does not require to be boiled, the slop has a relatively higher nutritive value. In addition, the yield of alcohol is greater. Approximately one fourth of the green malt is mixed with the potato juice at 55° and the dried potato pulp gradually added in the proportion of 50 g. of pulp to 120 c.c. of juice. The acidity of the mash is adjusted to 0.05—0.1° by the addition of sulphuric acid and the temperature raised to 75—80° for 15 min. After cooling to 55°, the remainder of the malt is added and the whole allowed to saccharify for 30—45 min. After finally heating the mash to 60—61°, it is cooled and fermented with yeast. C. RANKEN.

Fermentation-physiological properties of *Saccharomyces Saké*. Y. NISHIWAKI (Zentr. Bakt., 1929, II, 79, 194—204; Chem. Zentr., 1930, i, 1866).—The optimal temperature for yield of alcohol, carbon dioxide, and yeast is 23.5—24°; the maximum for daily production of carbon dioxide and fermented extract is 33—34°. The acidity is scarcely influenced by the temperature. Experiments on the optimal conditions for the fermentation of koji extract are described; multiplication and fermentation are not so rapid or complete as with maize. A. A. ELDRIDGE.

PATENTS.

Manufacture of a pure, constant yeast. E. I. LEVIN (B.P. 311,315, 21.1.29. Swed., 9.5.28).—A pure culture of yeast suitable for bread-making is mixed with sucrose and phosphates and incubated at 20—40° for 1—5 hrs. The yeast is then separated and dried. C. RANKEN.

Manufacture of acetic acid and lactic acid by fermentation. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 316,287, 5.4.29. Belg., 27.7.28).—Worts containing a high percentage of sugars are fermented without evolution of gas for 2 or 3 days by a new species of bacteria termed *Lactobacillus acidophilus*, B, which is obtained from milk. Its optimum temperature is 38°, and it differs essentially from *B. bulgaricus* in that it strongly attacks sucrose. C. RANKEN.

Manipulation of liquids (B.P. 309,541).—See I.

XIX.—FOODS.

Emulsions. I. Conditions for formation of various types of emulsion from fat and skim milk and the effect of emulsifying media on milk-in-fat emulsions. A. EICHSTÄDT. II. **Stability of emulsions in relation to viscosity of fat, surface tension, and the formation of adsorption films.** W. MOHR and A. EICHSTÄDT (Milchwirt. Forsch., 1930, 9, 388—395, 396—408; Chem. Zentr., 1930, i, 1869).—I. The process "first fat, then milk" gives an emulsion of milk in fat; the converse process usually gives a creamy emulsion. Rapid addition of fat, or simultaneous mechanical admixture, gives a double emulsion. The first type is that required for margarine emulsions, the stability of which is the greater when coconut or hardened arachis oil is used, and when small quantities of emulsifiers are added.

II. The reasons for the greater stability of certain milk-in-fat emulsions are discussed. The formation of an absorption film at the interface fat-skim milk is concerned. A. A. ELDRIDGE.

Nutritive value of condensed milk and milk powder. W. F. DONATH (Med. Dienst Volksgesondb. Ned.-Indie, 1929, 1—57; Chem. Zentr., 1930, i, 1638).—With one exception, considerable amounts of vitamin-A were present. With sterilised, evaporated, and condensed milk the loss of vitamin-B was 50% of that present in fresh milk. Only small quantities of vitamin-C were present. A. A. ELDRIDGE.

Relation between constants of butter fat. O. LAXA (Ann. Falsif., 1930, 23, 159—160).—The relation $(RM + WP + i)/n$ stated (B., 1928, 374) to be a constant is now shown to rise from 1.4 to 1.59 during the months June to September owing to the fact that during this period the iodine value rises more than the refraction. It is also shown that the relation $(RM + WP + i)/s$ (where s = solidif. pt.) rises from 2.8 to 3.6 during the period February to September. It is suggested that this number is below 3 for butters produced while the cows are on dry food, and above 3 for butters produced while the cows are having green food. A. SHORE.

Testing of casein for technical purposes. H. ULEX (Chem.-Ztg., 1930, 54, 421—422).—The following method is recommended for the determination of the acidity, calculated as lactic acid, of commercial preparations of casein: 1 g. of the powder is dissolved in 25 c.c. of 0.1N-sodium hydroxide at 20° and 175 c.c. of a 3% solution of calcium chloride previously neutralised to phenolphthalein are added, followed by 25 c.c. of 0.1N-hydrochloric acid and 8 c.c. of potassium mercuri-iodide solution (1.35 g. of mercuric chloride and 5 g. of potassium iodide in 100 c.c. of water); the mixture is diluted to 250 c.c. with freshly boiled water and 100 c.c. are filtered through a dry paper and titrated with 0.1N-sodium hydroxide using phenolphthalein as indicator. The total acidity of a good casein should not exceed the equivalent of 1.75% of lactic acid. A. R. POWELL.

Determination of acids in silage by Wiegner's method. K. GREIST (Tierernähr., 1929, 1, 65—69; Chem. Zentr., 1930, i, 1873).—A diagram permitting

direct reading of the amounts of acetic and butyric acids has been constructed. A. A. ELDRIDGE.

Baking value of flour, and possibility of determining it in the laboratory. A. TASMAN (Chem. Weekblad, 1930, 27, 138—143).—An account is given of the attempts to correlate chemical and physical constants, particularly viscosity and swelling power, with baking properties. Valuable information may be obtained by determinations of viscosity in presence of regularly increasing quantities of acid, the differences in viscosity being plotted to form a curve. From the form of the curve for a given flour, rough estimates may be made of the ash content, gluten content, and swelling power. S. I. LEVY.

"Adulterated" olive oil in sardines. H. WEWERS (Z. angew. Chem. 1930, 43, 656).—High iodine values of the oil used in preparing sardines, thought to be due to adulteration of the olive oil, were found to be associated with the extraction from the sardines of a fat of very high iodine value by the pure oil.

S. I. LEVY.

Gelation of fruit juices. W. ZIEGELMAYER (Kolloid-Z., 1930, 52, 243—248).—Viscosity measurements of boiled fruit juices have been made and the values compared with the loss of water. In general, high viscosity goes with high pectin content and of acid, so that unripe fruit juices gelate better. The tendency to gelate in the juices examined is in the following order: black currant → gooseberry (unripe) → tomato → apple (unripe) → gooseberry (ripe) → rhubarb. Rhubarb juice contains little pectin and does not gelate readily in spite of its high content of acid. The tendency to gelate can be influenced considerably by the presence of other substances, especially sugar, the addition of which causes an enormous increase in viscosity. Medium concentrations of acid (about 0.1*N*) have the most favourable effect on the gelation of mixtures of fruit juices and sugar. On keeping, the viscosity of fruit juices and their tendency to gelate diminish. Moulds were observed to grow only on preparations poor in sugar or in pectin, and even then not when exposed to free circulation of dry air.

E. S. HEDGES.

Chemical and bacteriological examination of lemonade. C. DUPONT (Ann. Falsif., 1930, 23, 141—145).—The results of examinations of seven samples are recorded and the necessity for adopting hygienic methods of manufacturing and bottling is stressed.

A. SHORE.

Desiccation of potatoes. LÜHDER and others.—See XVIII.

PATENTS.

Process of sterilisation. J. W. YATES, Assr. to GENERAL LABORATORIES, INC. (U.S.P. 1,754,251, 15.4.30. Appl., 9.5.28).—The sterilisation of plant used in connexion with milk, e.g., in creameries, condenseries, and bottling plants, is effected by atomising a solution containing 100—500 p.p.m. of available chlorine in close proximity thereto. The effective agent is hypochlorous acid, formed by the reaction of the chlorine with atmospheric carbon dioxide. It is claimed that the solution of hypochlorous acid so produced has also a

highly solvent action on the film of casein adhering to the surfaces in such plants. C. JEPSON.

Apparatus for the treatment [sterilising and drying] of fish materials. W. H. HASLAM (B.P. 332,241, 16.4.29).

Drying of grass (B.P. 327,409).—See XVI. **Pure, constant yeast** (B.P. 311,315).—See XVIII. **Preserving organic etc. matter** (B.P. 331,546).—See XXIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Toxicity of different samples of mercurochrome 220. J. H. BURN and G. K. ELPHICK (Quart. J. Pharm., 1930, 3, 178—186).—Determinations of the toxicity of eight commercial samples of mercurochrome by intravenous injection into mice showed a variation of the lethal dose (causing 50% mortality) from 37 mg. to 140 mg. per kg. There was no parallelism between high toxicity and high mercury content except in extreme cases. A limiting toxicity of 105 mg. per kg. is suggested for commercial products. A simple method of determining toxicity is described. R. K. CALLOW.

Evaluation of tincture of iodine. E. RUPP and G. HAMANN (Apoth.-Ztg., 1930, 45, 67—68; Chem. Zentr., 1930, i, 1982).—Potassium iodide is determined in the solid residue after evaporation; the residue is also tested for chloride and bromide. After the determination of free iodine an additional quantity of sodium thiosulphate is added; the appearance of turbidity within 10 min. indicates the presence of acid decomposition products. A. A. ELDRIDGE.

Evaluation of tincture of iodine. G. FRERICHS (Apoth.-Ztg., 1930, 45, 161; Chem. Zentr., 1930, i, 1982; cf. preceding abstract).—For the determination of total iodine the tincture is treated with ferric chloride solution; after 1 hr. it is diluted, treated with phosphoric acid and then with potassium iodide, and titrated. An acidimetric determination of hydrogen iodide is desirable. A. A. ELDRIDGE.

Constituents of aperient drugs. L. ROSENTHALER (Pharm. Acta Helv., 1929, 4, 128—132; Chem. Zentr., 1930, i, 1948).—Légers formula for barbaloin (A., 1917, i, 276) is criticised. An osazone could not be prepared, and the anticipated acid was not produced by heating with dilute nitric acid. The reduction of alkaline copper sulphate solution is attributed to reduction products analogous to anthranols. No methoxyl group could be detected, and the existence of a penta-acetyl derivative is not considered to be established. *as*-Phenylmethylhydrazine affords a derivative which does not exhibit the characteristics of an osazone.

A. A. ELDRIDGE.

Pharmacology of ergot with special reference to biological assay and standardisation. VII. **Changes occurring in crude ergot and fluid extract of ergot**, U.S.P. X, during storage. VIII. **Bio-assay standards for ergot and its preparations.** M. R. THOMPSON (J. Amer. Pharm. Assoc., 1930, 19, 436—449).—If the drug is kept dry the loss of alkaloidal activity in whole ergot during storage (1—2 years) is not serious. Airtight containers are not necessary, and

are to be avoided unless the drug is kept dry by a suitable dehydrating agent. Storage of powdered ergot results in an appreciable loss in activity unless the fixed oil is first removed. The acid value of the fixed oil increases during storage, but this bears no relationship either to the specific alkaloidal activity or the non-specific amine content. Storage of the dry material causes no significant change in the histamine or other proteinogenous amine contents, but damp samples rapidly develop these amines. The deterioration of fluid extract of ergot in specific alkaloidal activity is hastened by exposure to air and is decreased by storage in a refrigerator; its rate is more rapid during the first three months, after which it becomes slower and more constant, but even if the preparation is kept in partially evacuated ampoules, a slight but definite loss in activity cannot be avoided. From a consideration of the proposed standards for ergot, the author recommends that the fluid extract should have an alkaloid content of 0.05%. Ergotamine tartrate is a better standard than is either the phosphate or ethanesulphonate; it is more definitely crystalline and gives a better solution.

E. H. SHARPLES.

Approximate determination of alkaloid in fluid extract of ergot. H. ESCHENBRENNER (Apoth.-Ztg., 1929, 44, 1534; Chem. Zentr., 1930, i, 1665).—The extract (10 c.c.) is treated with alcohol (10 c.c.) and dilute hydrochloric acid (5 drops), and evaporated to 5 c.c. Water (10 c.c.) is added, and 10 c.c. of the filtrate are treated with 2 c.c. of Mayer's reagent and 4 g. of sodium chloride. The mixture is placed in a burette. The volume of the precipitate should reach at least 2 c.c. in 2 hrs.

A. A. ELDRIDGE.

Stability of tincture of digitalis, B.P., as estimated by the frog method. F. WOKES (Quart. J. Pharm., 1930, 3, 205—217).—Periodical assay by the frog method of tinctures of digitalis showed that the activity decreased initially by about 3% per month, reaching two thirds of the initial value after 16—17 months, whereas the author (*ibid.*, 1929, 2, 48) found no deterioration by the cat method. Variation in p_H from 5.47 to 5.93 did not appear to affect deterioration, contrary to the statement of Joachimoglu and Bose (B., 1924, 766), but the rate of deterioration was increased at p_H 8. The diminution of potency is not due to delayed absorption in the frog.

R. K. CALLOW.

Determination of pilocarpine. P. BOURGET (Schweiz. Apoth.-Ztg., 1930, 68, 381—382).—The ground and sifted jaborandi leaves (25 g.) are moistened with 200 c.c. of 10% sodium carbonate solution and extracted in a Soxhlet apparatus for 3 hrs. with benzene; the cold extract is immediately extracted with 1% sulphuric acid in portions of 30, 20, 20, and 10 c.c., and the combined acid extract is filtered, neutralised to Congo-red with ammonia, and treated with 1% potassium permanganate solution until one drop produces a rose tint which persists for a moment. After the addition of excess of ammonia the solution is extracted about 10 times with chloroform and the united chloroform extract (50—60 c.c. total), after filtration over dry and powdered sodium carbonate, is neutralised

with 2% nitric acid and then evaporated to dryness on a water-bath. The residue is treated with a small quantity of acetone, which removes the impurities without affecting the pilocarpine nitrate, and filtered through a Gooch filter, which is then dried below 100° and weighed. The white crystalline powder should have m.p. about 174—175°; if the assay is for the manufacture of pilocarpine, it should not be below 165°. If the benzene extract be kept for very long before extraction with sulphuric acid, the alkaloid content decreases rapidly. If before adding sodium carbonate the leaves are extracted with a hot solvent, the alkaloid content of the subsequent benzene extract is not affected on keeping. Apparently the substance which provokes the decomposition exists in the leaf and is removed by extraction.

E. H. SHARPLES.

Polish rhubarb. M. RUSZKOWSKI (Spraw. Prac. Państ. Inst. farm., 1925, 15 pp.; Chem. Zentr., 1930, i, 1339—1340).—The percentage of extract, the content of anthraquinone derivatives, and the ash content of Polish varieties of rhubarb are recorded.

A. A. ELDRIDGE.

Use of the bismuth-iodine reagent in the evaluation of galenical hemlock preparations. M. M. JANOT and C. FAVRE (Bull. Sci. pharmacol., 1929, 36, 529—541; Chem. Zentr., 1930, i, 1191).—The extract (2 g.) is dissolved in water (5 c.c.), treated with 10 c.c. of sodium hydroxide solution, and extracted twice with 25 c.c. of ether, the united extracts being acidified with sulphuric acid, treated dropwise with the reagent (prepared from bismuth carbonate 2 g., officinal hydrochloric acid 5 c.c., potassium iodide 12.5 g., and water 25 c.c.), and filtered after 5 min. The precipitate is collected, washed with water acidified with sulphuric acid, heated with 35 c.c. of sodium hydroxide solution at the b.p. for 10 min., distilled into 0.1*N*-sulphuric acid, titrated, and calculated as conine. Tests with guinea-pigs show that the fatal dose is 0.01—0.02 g. per kg.

A. A. ELDRIDGE.

Composition of cinchona febrifuge. J. A. GOODSON and T. A. HENRY (Quart. J. Pharm., 1930, 3, 238—248).—In applying the Howard and Chick process for the separation of cinchona alkaloids to "cinchona febrifuge," the composition of the fractions obtained has been checked by determinations of methoxyl content and optical rotation. The quinine does not as a rule crystallise as the sulphate, but is precipitated at the second stage with cinchonidine tartrate. The amount of quinine in the bases from the tartrate is calculated from the methoxyl content. For the determination of quinidine Chick's modification of the method ("Allen's Organic Analysis," 5th Ed., 1929, Vol. 6, p. 426) is satisfactory, the precipitate consisting of practically pure quinidine hydriodide, but the quality of the "cinchonine" isolated is variable, and may be roughly gauged by ascertaining the yield of "crystallisable cinchonine" obtainable from it.

R. K. CALLOW.

Camphor substitutes. H. GOLDSTEIN (Bull. Soc. chim. Belg., 1930, 39, 197—205).—A lecture.

A. I. VOGEL.

Sensitive colour reaction for rivanol, and its application. M. J. SCHULTE (Pharm. Weekblad, 1930,

67, 601—604).—The diazo reaction gives a deep red dyestuff, fast to wool and silk, but not to cotton. The reaction may be applied for colorimetric determination in pharmaceutical preparations. S. I. LEVY.

Homœopathic aconite and gelsemium preparations. H. NEUGEBAUER (Apoth.-Ztg., 1930, 45, 80—82; Chem. Zentr., 1930, i, 1983).—The tincture is evaporated with hydrochloric acid, made ammoniacal, and extracted with ether; the ethereal residue is titrated. The factor 0.03322 is recommended for gelsemium instead of 0.03222. A. A. ELDRIDGE.

Identification of homœopathic preparations by luminescence analysis. H. NEUGEBAUER (Pharm. Ztg., 1930, 75, 885—889).—Preparations which give identical results under normal conditions of capillary analysis (cf. B., 1930, 82) can, in many cases, be differentiated by treatment of the "capillary picture" with certain reagents before the examination under ultra-violet light. Many examples are given and also the results of the examination of various drugs at different dilutions. E. H. SHARPLES.

Determination of essential oils [in drugs]. H. GFELLER (Pharm. Acta Helv., 1929, 4, 200—201; Chem. Zentr., 1930, i, 1665).—The method of D.A.B. VI. gave consistent results. Preparations of juniper fruit containing at least 1% of essential oil could not be obtained; German and Swiss camomile gives a blue, Hungarian a green, and camomile smells a brown oil of unpleasant odour. A. A. ELDRIDGE.

Physiological aspects of the essential oils. G. M. DYSON (Perf. Essent. Oil Rec., 1930, 21, 287—314).—A review.

***Oleum deelinæ* [dee oil].** KESSLER.—See II.

PATENTS.

Production of monohydric phenols. BOOT'S PURE DRUG CO., and J. MARSHALL (B.P. 330,333, 26.3.29).—*n*-Amylcresols, having a phenol coefficient of 250—300, are prepared from tolyl *n*-valerates by treatment with aluminium chloride and reduction of the resulting ketone by Clemmensen's method. The preparation of 3-valeryl-*o*-cresol, m.p. 18°, b.p. 140—150°/15 mm., 5-valeryl-*o*-cresol, m.p. 103—104°, b.p. 200—205°/15 mm., 3-*n*-amyl-*o*-cresol, m.p. 32°, 5-*n*-amyl-*o*-cresol, m.p. 29°, 4-valeryl-*m*-cresol, m.p. 6°, b.p. 152—154°/15 mm., 4-*n*-amyl-*m*-cresol, m.p. 24—25°, b.p. 137—139°/15 mm., 3-valeryl-*p*-cresol, m.p. 32—33°, and 3-*n*-amyl-*p*-cresol, m.p. 10°, is described. C. HOLLINS.

Rubber derivatives (B.P. 313,919).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Comparison of resolving power and sensitivity of photographic plates with varying development. B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1930, 5, 1—11).—Five special developers recommended for reducing the graininess of photographic images were compared with pyrogallol and metol-quinol. Little useful improvement over the standard developers was obtained as increased resolution was accompanied by

decreased sensitivity. The results do not apply to pictorial photography. C. J. SMITHELLS.

Photography on copper. C. J. SMITHELLS (Nature, 1930, 126, 133).—Immersion of a copper or brass surface for 10 sec. in a 10% solution of copper chloride or of copper ammonium chloride renders the surface sensitive to light. Details for obtaining photographic images on such surfaces are given. L. S. THEOBALD.

Desensitisers. III. Desensitising properties of chrysoidine and some of its derivatives. (Miss) F. M. HAMER (Phot. J., 1930, 70, 232—233; cf. B., 1929, 1032).—The desensitising power of chrysoidine and of the hydrochlorides of six of its derivatives have been investigated and compared with that of safranin. The sample of chrysoidine used was as powerful as safranin (most samples are inferior), but it retarded development much more strongly. The methyl derivative was less powerful, whilst the bromo-derivative and benzene-3-azo-5-chloro-2:4-tolylenediamine hydrochloride were as efficient as safranin. The time of appearance of the image with these three compounds was approximately the same as with safranin; chloro-chrysoidine and its methyl derivative were more powerful than safranin, and whereas the former retards development to the same extent as chrysoidine, the latter shows even stronger retardation. The desensitising power of these compounds is shown to be a property of the hydrochlorides, since the corresponding bases and *p*-toluene-3-azo-5-chloro-2:4-tolylenediamine are very feeble desensitisers. Such compounds are therefore not satisfactory as practical desensitisers, since the desensitisation conferred in a preliminary bath is destroyed by the alkali of the developer. J. W. GLASSETT.

Anti-fogging and anti-sensitising effects. S. E. SHEPPARD (Z. wiss. Phot., 1930, 28, 85—90).—See B 1929, 151.

PATENT.

Manufacture of light-sensitive silver halide emulsions. O. MATTHIES and B. WENDT, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,758,576—7, 13.5.30. Appl., 28.1.29. Ger., 31.1.28).—See B.P. 305,143; B., 1930, 793.

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose service powders. M. VUKADINOVIC (Arhiv Hemiju, 1930, 4, 139—142).—The maximum pressure of the gaseous products of combustion of a powder is the greater the smaller is the solubility of the nitrocellulose. The disruptive power is the greater the higher is the nitrogen content, on which the quickness of the powder also depends. The degree of gelatinisation of a powder influences its density. C. W. GIBBY.

XXIII.—SANITATION; WATER PURIFICATION.

Sterilisation of water by ultra-violet light as emitted by the carbon arc. R. G. PERKINS and H. WELCH (J. Amer. Water Works' Assoc., 1930, 22, 959—967).—Small-scale experiments, using a carbon arc in place of the usual mercury lamp, show that an adequate sterilising effect can be obtained through a distance of 12 in. with an exposure of 4 sec. during which the column of water is constantly advancing towards

the source of light. Since the range of effective radiation is at least 12 in., it follows that a volume of water passing a single arc may have a radius of 12 in. provided the exposure towards and away from the source of light amounts to 4 sec.

C. JEPSON.

Zeolite [water]-softening plant experiences. D. E. DAVIS and J. T. CAMPBELL (J. Amer. Water Works' Assoc., 1930, 22, 952—958).—Comparison of operating results of a 2,000,000 gal./day pressure-type plant and a 4,000,000 gal./day open-type filter indicates that whereas the former was operated at twice the rate of the latter, rather less hardness was removed per cub. ft. of sand and more difficulty was experienced in preventing loss of the zeolite when washing. Average operating results of the open-type plant are as follows: hardness of raw water 194 p.p.m.; hardness of final water 63 p.p.m.; length of total softener run 559 min.; hardness removal 4004 grains/cub. ft.; salt required for regeneration 0.334 lb. per 1000 grains of hardness removed.

C. JEPSON.

Determination of hardness of water. E. F. TEBENIKHIN (J. Chem. Ind., Russia, 1929, 6, 222—236).—Clark's method, using soap solution standardised with barium chloride, gives low results for calcium and high results for magnesium salts. Hard waters should first be diluted to a hardness of 3—5°. Wartha and Pfeifer's method gives low results for waters below 3° hardness, whilst Blacker's method gives high results for soft corrected and boiler waters. The palmitic acid is preferably obtained from Japan wax, and is neutralised with potassium hydroxide in glycerol solution.

CHEMICAL ABSTRACTS.

Determination of sodium [in water]. E. R. CALEY and C. W. FOULK (J. Amer. Water Works' Assoc., 1930, 22, 968—976).—A rapid gravimetric and colorimetric method of determining sodium in water consists in precipitating sodium as magnesium uranyl sodium acetate with a solution of magnesium uranyl acetate in acetic acid, and either weighing the precipitate or redissolving it and comparing colorimetrically with a standard sodium solution treated in a similar way. The accuracy of the method is stated to be equal to, if not higher than, that of the standard method of the A.P.H.A.

C. JEPSON.

Determination of urea in water. M. H. MCCRADY (J. Amer. Water Works' Assoc., 1930, 22, 926—937).—The general assumption that urea in aqueous solution is rapidly converted into ammonium carbonate is shown not to be the case for dilute solutions unless acidified, and, moreover, free ammonia may be removed by boiling the sample without destroying any urea present. If the urea nitrogen be then converted into ammoniacal nitrogen by means of urease a measure of the urea originally present is obtained. Detailed instructions for making the determination are given, and, as it is capable of a sensitivity of the order of 0.004 mg. of urea nitrogen, it may prove a useful indicator of the presence of excretal pollution in sanitary water analysis.

C. JEPSON.

Variation of phenol coefficients of coal-tar disinfectants with different test organisms. B. G. PHILBRICK (Ind. Eng. Chem., 1930, 22, 618—619).—From a study of the phenol coefficients of coal-tar

disinfectants of various concentrations with different organisms it is possible, if the *B. typhosus* phenol coefficient is known, to ascertain the approximate efficiency of a disinfectant against *Staph. aureus*, *B. diphtheriae*, *Strep. hemolyticus*, and *Pneumococcus* in presence or absence of organic matter. This may be expressed as the phenol coefficient or as maximum dilution which will kill the given organisms in 5 min. in presence of organic matter. Determinations were made of the maximum dilutions of disinfectants which would kill *B. tuberculosis* in sputum in 1 hr., using intraperitoneal inoculations into guinea-pigs, so establishing the relationship between phenol coefficient for *B. typhosus* and power of sterilising tubercular sputum.

W. J. BOYD.

Theory of gas masks. II. W. MECKLENBURG (Kolloid-Z., 1930, 52, 88—103).—The theory of adsorption so far as it is applicable to gas masks is worked out and the conclusions reached explain the experimental results of Schilov, Lepin, and Wosnessensky (A., 1930, 27).

E. S. HEDGES.

Test for industrial lead poisoning; presence of basophilic red cells in lead poisoning and lead absorption. C. P. MCCORD (U.S. Bur. Labour Statistics, Bull., 1928, No. 460, 33 pp.).

PATENTS.

Purification of boiler feed water. P. JÓHANSSON (B.P. 331,778, 7.10.29).—A method and apparatus are claimed in which waste heat and waste steam are utilised to produce distilled water for use in the boiler. The waste steam is superheated by passing through a pipe coil placed in the furnace flue or funnel smoke box and its heat utilised to boil water in an evaporator. The steam produced is separated from the boiling liquor and passed to the feed water supply.

C. JEPSON.

Water purification. J. T. TRAVERS and O. M. URBAIN, ASSRS. to OHIO SANITARY ENGINEERING CORP. (U.S.P. 1,754,129, 8.4.30. Appl., 13.7.28).—Sewage or water is mixed with a non-putrescible colloidal marl or plastic clay and the p_H value raised, e.g., by calcium hydroxide, so that the colloids carry a negative charge. An electrolyte, characterised by its capacity to dissociate, is also added with the result that the positive ions so released are adsorbed by the colloids and the negative ions are able to release enough nascent oxygen from the water to satisfy the biochemical oxygen demand of the sewage or completely to sterilise the drinking water as the case may be.

C. JEPSON.

Water purification [from phenols etc.]. J. T. TRAVERS, C. H. LEWIS, and O. M. URBAIN, ASSRS. to OHIO SANITARY ENGINEERING CORP. (U.S.P. 1,754,068, 8.4.30. Appl., 27.8.28).—Phenolic substances may be completely removed from water by treatment with a mixture of the hydroxides of iron and calcium and marl clay (minimal amounts, 4.4, 14.8, and 30 grains per gal., respectively), or waste cement dust in which all free lime and calcium carbonate has been neutralised with sulphuric acid. To secure complete removal a reaction period of at least 10 min. and p_H above 10.8 are necessary.

C. JEPSON.

Air-purifying apparatus (B.P. 307,428).—See I. **Sterilisation** (U.S.P. 1,754,251).—See XIX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 26 and OCT. 3, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Spray dryers. W. KUHLES (Chem. Fabr., 1930, 293—296).—Dryers or evaporators using an atomised spray of liquid or solution are an extension of the principle of film evaporation. The temperature of the air used can be reduced owing to the large surface of contact. The most suitable arrangement has a vertical spray rising to meet a baffle-plate, from which it falls outwards against a countercurrent of heated air passing upwards in an annular exterior zone of the drying chamber. Such an air current has no eddies. Concrete is better than wrought iron as constructional material on account of its lower heat conductivity. In many cases the fine state of division of the product of such a dryer is an advantage. C. IRWIN.

Adsorption of gases and vapours on active charcoal and recovery of the adsorbed products. A. FOULON (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 235—237, 281—283).

Oil firing. HIND.—See VIII.

PATENTS.

Muffle furnace and method of operating same. W. W. KEMP, Assr. to C. M. KEMP MANUF. CO. (U.S.P. 1,752,800, 1.4.30. Appl., 29.8.27).—The combustion gases are prevented from entering the muffle by pumping air into it, so that the pressure inside the muffle is always slightly higher than that in the combustion chamber. The air is preheated by the waste combustion gases. D. K. MOORE.

Gas-fired furnaces. WELLMAN SMITH OWEN ENG. CORP., LTD., and H. W. SOWARD (B.P. 331,635, 9.5.29).—The gas is admitted through independently controlled inlets in the side and one of the end walls and the air downwards through the roof in the form of a number of small jets. Recuperator passages for the air and waste gases are also arranged in the roof structure. B. M. VENABLES.

Calcining apparatus. F. GELSTHARP, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,754,854, 15.4.30. Appl., 17.5.28).—A rotary, inclined, cylindrical kiln is described for the calcination of green copperas. Among other details, a stationary muller is provided at an intermediate point in the kiln to break up lumps. B. M. VENABLES.

Drying kilns. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 332,491, 2.11.29. Ger., 3.11.28).—Automatic electric controls are described for a drying kiln, used, *e.g.*, for lacquered articles, which must have doors closed during the periodical stoppages of the conveyor. B. M. VENABLES.

Drying ovens. S. L. GROOM (B.P. 332,067, 25.7.29).—A conveyor for carrying plate-like objects through a tunnel dryer is arranged so that both runs are effective, provision being made to charge articles at both ends simultaneously, on the top and bottom runs respectively. Hooked carriers may be attached to the conveyor chains, the hooks supporting the articles on the lower run and the conveyor itself on the upper.

B. M. VENABLES.

Spray-drying apparatus. W. S. BOWEN (U.S.P. 1,753,915, 8.4.30. Appl., 5.6.29).—The liquid is sprayed centrally in the upper part of the drying chamber or tower and the air is admitted through a false ceiling which is comparatively thick and is provided with apertures situated radially but inclined to the axis of the vessel, the thickness of the roof being necessary to give sufficient tangential motion to the incoming air. B. M. VENABLES.

[Indirect] heating or cooling of [divided] solid substances. N. AHLMANN (B.P. 332,226, 16.4.29).—The pulverulent solid substantially fills a container and is subjected to heating or cooling through the walls of the vessel and by means of a stirrer which may be, *e.g.*, in the form of a zigzag tube; an upward current of air or gas is provided to keep the material loose without producing whirling. The heating may be used to bring about chemical reactions between more than one solid substance. B. M. VENABLES.

Heat-exchange apparatus. A. D. HARRISON (U.S.P. 1,754,857, 15.4.30. Appl., 10.4.29).—A number of plates, alternately plain and ribbed on both sides to form sinuous passages, are assembled in a press. Packing is applied round the circumference only of the plates, and leakage from around the transfer ports is led out of the press by guard grooves leading to ports. When used for the pasteurisation of milk, the raw milk will first exchange heat with the treated milk in one section of the press and then be further heated by steam or other hot fluid in another section. B. M. VENABLES.

Heat-exchange device. G. KRÄNZLEIN, and SAMESREUTHER & Co. G.M.B.H. (B.P. 331,910, 9.4.29. Ger., 10.4.28. Addn. to B.P. 309,445; B., 1930, 798).—The heat exchanger is modified so that electrical heating elements can be placed in the pipes. The pipes are enclosed in two flanged plates and are in thermal contact with them. D. K. MOORE.

(A, C) **Rotary pivoted hammers [for pulverisers].**
(B) **Mill pulveriser.** J. P. POLSTER, Assr. to ILLINOIS STOKER CO. (U.S.P. 1,753,473—5, 8.4.30. Appl., [A] 14.3.28, [B] 2.5.29, [C] 5.8.29).—In (A) and (C) stirrup-shaped swing hammers for pulverisers are described.

* The remainder of this set of Abstracts will appear in next week's issue.

In (B) a pulveriser is described having two end-zones mainly for crushing, the beaters of which deflect the material into the centre pulverising zone, which is surrounded by a continuous bull-ring; both inlet and outlet of material is effected through the same openings near the ends, and air inlets may be provided through the ends of the machiné. B. M. VENABLES.

Pulverising or grinding mill. D. J. UHLE and J. V. DURNIN (U.S.P. 1,752,314 and 1,752,622—3, 1.4.30. Appl., 22.11., 30.3., and 22.11.28).—Balls are rotated by pusher elements attached by yielding supports to arms on the shaft. This avoids undue strain. By making these pushers rotatable, scoring and wear of the balls are reduced. D. K. MOORE.

Pendulum roll mill. F. L. BUCHANAN (U.S.P. 1,754,414, 15.4.30. Appl., 24.9.28).—A rotating drum is provided with liner plates and hollow trunnions, and through the latter and right through the mill extends a bar, which may be hollow and used to supply water, carrying grinding rollers in stirrup-shaped supports. The feed is introduced through separate conduits and screened discharge openings are formed in the ends or central part of the circumference. The mill may be operated wet or dry. B. M. VENABLES.

Grinding or crushing mills. G. H. ROBINSON (B.P. 331,877, 10.4.29).—The grinding rolls are fitted in a carriage attached by hinged joint to a carrier fixed to the shaft. Springs behind the carriages force the rolls against the bull-ring. A cone distributor is used for feeding the machine. A suitably shaped plough lifts coarse material from the lower part of the machine back to the grinding rolls. D. K. MOORE.

Gyratory crushers. W. W. TRIGGS. From TRAYLOR ENG. & MANUF. CO. (B.P. 332,348, 6.6.29).—A form of seal to prevent dust reaching the mechanism of such crushers is described. B. M. VENABLES.

Mixing apparatus. BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 331,606, 15.4.29).—A jacketed vessel is provided with downwardly depending fixed stirrers and with moving ones upstanding from a rotating arm, the distance of which from the bottom of the vessel may be varied. The vessel may have the inner shell of chemically resistant material, and may be provided with a lid having inlet and outlet openings, and with a draw-off valve of the type that opens by spring action and is closed manually. B. M. VENABLES.

Agitation and settling process. C. P. HOOVER and F. A. DOWNES (U.S.P. 1,752,795, 1.4.30. Appl., 7.8.25).—The top part of a cylindrical vessel is fitted with agitators and serves as a chamber in which the precipitate, by a suitable combination of agitator speed and time factor, is transformed into the best condition for settling. The lower part is made up of a number of superimposed settling compartments with inverted cone-shaped bottoms having central discharge for the sediment, and outlet pipes for the clarified liquor. D. K. MOORE.

Agitation and settling apparatus. [Water softener.] F. A. DOWNES, Assr. to DORR CO. (U.S.P. 1,752,789, 1.4.30. Appl., 7.8.25).—The apparatus is

suitable for water softening or other purposes where the weight of solids is small compared with the volume of liquid and the flocculation has to be carefully controlled. It comprises an agitation chamber superposed upon a number of settling chambers. In the former rather more violent agitation may be given in the earlier stages by spacing the stirrers closer in the upper part than in the lower; the settling chambers have a common central feed passage which also forms the outlet for settled sludge to the next chamber below. Of these stirrers only the lowest has a spigot or pump discharge for the sludge, but each has a conical bottom and set of rakes and immediately under the circumference of each bottom is a ring-pipe for clear overflow. The overflow from each compartment is separately controllable by valves or by small adjustment of the height of the ends of the overflow pipes, and the combined overflow may be passed upwards through a filter to a storage compartment situated above the rest of the apparatus; provision is also made for returning sludge to the feed to aid settling. B. M. VENABLES.

[Shaking-tables for] separation of dry materials. BIRTLEY IRON CO., LTD., and (A) C. W. H. HOLMES and I. L. BRAMWELL, (B) (HON.) B. L. BOURKE (B.P. 332,291 and 332,583, [A] 27.5.29, [B] 23.1.29).—Shaking-tables with decks pervious to air currents are described, the discharge of material being entirely over one edge. B. M. VENABLES.

Apparatus for effecting contact between gases and divided solids. HUMPHREYS & GLASGOW, LTD., Assees. of E. J. BRADY (B.P. 315,868, 10.7.29. U.S., 21.7.28).—The divided solid is allowed to fall through an annular space between two rotating, perforated walls and the gas passes zigzag across the space, the general flow being upward or countercurrent. The perforations slope downwardly towards the annular space and may be in the form of louvres, fingers or wires may be placed in the annular space, the baffles which cause the zigzag passes may be flushed by a liquid, and means may be provided by which the quality of the outgoing gas determines the rate of treatment. *E.g.*, in the removal of hydrogen sulphide from coal gas by iron oxide, a small burning jet of the gas, a ribbon of lead acetate paper, and a photo-electric cell may be used. B. M. VENABLES.

[Centrifugal] separation of mixtures. L. P. SHARPLES, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,754,774, 15.4.30. Appl., 16.11.27).—Two centrifuges of different types are used in conjunction to separate a mixture of solid and liquid: the first delivers a clear liquid and a sludge, the second delivers dried solid (from the sludge) and a muddy liquid which is returned as original feed. B. M. VENABLES.

[Centrifugal] extractor. A. PARTLOW, Assr. to VORCLONE CORP. (U.S.P. 1,753,215, 8.4.30. Appl., 31.3.28).—An under-driven extractor is arranged so that the basket can be easily lifted right out of the casing and, when out, the bottom can be completely opened by swinging two half-doors, one of which carries the female part of a driving boss, the male part remaining on the shaft. B. M. VENABLES.

Centrifugal machines. R. M. BERLINE (B.P. 311,248, 7.5.29. Luxembourg, 7.5.28).—A centrifugal

machine for sedimentation is long in comparison with its diameter and is divided into two zones: the first for a preliminary separation without plates, and the second having closely spaced cones or plates producing a more intense effect. B. M. VENABLES.

Centrifugal machines. J. L. RUSHTON (B.P. 332,288, 18.4.29).—A self-contained system of forced lubrication for such machines suitable, *e.g.*, for spinning rayon, is described. B. M. VENABLES.

[Sedimentation and] filtration apparatus [for solid-laden liquids]. C. T. CABRERA, Assr. to ELECTRO DIALYZER CORP. (U.S.P. 1,754,667, 15.4.30. Appl., 26.7.28).—The fluid, such as sewage, is admitted well down on the axis of a cylindrical tank provided with a conical bottom and with rakes to discharge settled sludge; the upper wall of the tank is formed of permeable material through which the partly clarified liquid overflows. These filter members are supported in frames in sections and are double everywhere so that they can be removed section by section for cleaning without stopping the action; the necessity for cleaning is postponed because when the lower part of the filters becomes choked the level of the liquid will rise and pass through a clean, higher surface. There is also no possibility of choked filters causing a material rise of pressure. B. M. VENABLES.

Filter. J. T. SHIMMIN (U.S.P. 1,752,758, 1.4.30. Appl., 4.5.25).—In a filter of the continuous rotary-drum type with the filtering medium fastened to the inside of the drum, vacuum assisted by gravity is used to effect filtration and air pressure and gravity to discharge the filter cake. D. K. MOORE.

Filter. A. HOPKINS, Assr. to HOPKINS-TULL MACHINERY Co. (U.S.P. 1,754,320, 15.4.30. Appl., 20.12.28).—A number of semi-circular bags are contained in the annular space between two cylindrical walls, the feed entering the bags through a distributing manifold and the filtrate leaving the annular space upwardly. B. M. VENABLES.

Tipping filter press. P. W. PRUTZMAN and V. C. BENJAMIN, Assrs. to CONTACT FILTRATION Co. (U.S.P. 1,754,401, 15.4.30. Appl., 4.1.28).—The filter is of the recessed-plate type with filter medium on the lower side only of the cells. The axis of the pile is vertical when in operation, but horizontal for cleaning. B. M. VENABLES.

Filter-cake loop dryer. Loop dryer [for fabrics]. A. O. HURXTHAL, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,755,005—6, 15.4.30. Appl., [A] 24.4.26, [B] 3.7.28).—(A) The dryer is suitable for material that will adhere to a foraminous belt that unwinds from a drum filter or other means of applying the material in a thin layer. The filter drum, belt, and a discharging device are driven by separate electric motors. While in the dryer the belt forms deep U-shaped loops hanging over cross-bars on a link conveyor and at the dry end the loops are straightened out and the dry material is beaten off. In (B) a dryer of similar construction to that described in (A) is adapted to web materials, such as cloth or paper, and a device is added which serves to steady the loops against air currents. B. M. VENABLES.

(A) Process, (B) machine, for treating materials. C. W. VOGT, and VOGT INSTANT FREEZERS, INC. (B.P. 332,577 and 332,593, 23.4.29).—(A) Materials, *e.g.*, ice-cream or edible oils, are subjected to pressure, stirring, heating, or cooling, and, if desired, injection of gas, simultaneously, so as to produce a plastic compound of smooth texture. The resistance to flow or to agitation, being a measure of the plasticity, is caused to regulate the supply of heat-exchanging medium (coarse adjustment) and/or the supply of the material (fine adjustment); usually the greater the resistance the larger is the feed to prevent overfreezing. (B) A machine for above purposes comprises a number of tubular chambers with elongated internal stirrers and external spiral passages for heat-exchanging medium; different media or different quantities of the same medium may be passed round different tubes. External means are provided for producing the pressure. B. M. VENABLES.

Apparatus for making emulsions or dispersions. IMPERIAL CHEM. INDUSTRIES, LTD., and R. B. F. F. CLARKE (B.P. 331,928, 13.4.29).—The constituents are sprayed through at least two nozzles so as to impinge on each other or on a common surface; at least one of the nozzles is multiple so that a motive fluid such as steam or compressed air may be used, and partly mixed materials are lifted back into the spray. For example, linseed oil may be sprayed by steam and ammonium oleate solution simultaneously by compressed air. B. M. VENABLES.

Preparation of emulsions, suspensions, and colloidal dispersions. P. FREEDMAN (B.P. 332,533, 15.4.29).—The mixture is subjected to vibration by means of piezo-electric crystals energised by electric pulsations, which either may be in direct contact or may transmit their vibrations through another hydraulic medium and flexible dividing wall. B. M. VENABLES.

Apparatus for production of chemical compounds. M. PÓLÁNYI and S. VON BOGDANDY (B.P. 303,900, 11.1.29. Ger., 12.1.28).—In order to effect reactions between a liquid or dissolved substance, which cannot be unduly heated, and a gas or vapour, which may have to be heated to produce volatilisation, the liquid is spread in a thin layer on the interior of centrifugal bowl and surrounding container, being kept in circulation by a pump forming part of the centrifuge, while the other constituent is fed to the interior of the bowl and, if necessary, volatilised there by an electric arc or other means. B. M. VENABLES.

Apparatus for bringing liquids and gas and/or vapours into intimate contact. S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (B.P. 331,592, 3.4.29).—The apparatus is of the type in which brushes are rotated alternating with fixed partitions in a casing, the liquor being distributed independently of the brushes by a pump for each compartment with suitable multiple outlet. B. M. VENABLES.

Lining vessels for carrying out operations with liquids and the like. IMPERIAL CHEM. INDUSTRIES, LTD., and F. H. BRAMWELL (B.P. 331,838, 9.1.29).—A vessel to which an incorrodible lining, *e.g.*, of rubber or lead, is to be applied is made with a double (corrodible,

shell, the inner one being perforated, and after the lining is applied a reduced pressure is maintained in the space between the shells, thus preventing creep of the lining.

B. M. VENABLES.

Evaporating method and means therefor. INDUSTRIKEMISKA AKTIEB. (B.P. 312,089, 17.5.29. Swed., 18.5.28).—The liquid to be evaporated or solid to be dried is sprayed into a series of evaporating chambers in the presence of air. This air passes through the chambers in series and has its temperature raised step by step so that it takes up more and more moisture. At the end of the series it is further heated by external means such as a steam-coil, additional water being added if desired, and then passes the reverse way through the heating chambers in conducting relation with the evaporators, thus giving up the latent heat of the moisture and causing a corresponding evaporation. Preferably the pressure of the operations is maintained at slightly above or below atmospheric. In some cases, while the evaporating chambers remain in series, some of the heating chambers may be connected in parallel.

B. M. VENABLES.

Rotary apparatus for cooling liquids. R. ZWERINA (B.P. 331,812, 5.3.29. Austr., 14.2.29).—The liquid is cooled by flowing through passages in the vanes of a fan. Deflector plates reduce eddying when entering and leaving the vanes.

D. K. MOORE.

De-aerating water at atmospheric pressure. W. BOBY, and W. BOBY & Co., Ltd. (B.P. 332,678, 23.5.29).—The water flows countercurrent to live and/or exhaust steam through a series of compartments, being caused to flow under and over baffles and to cascade from compartment to compartment. The steam is regulated so that only enough to raise the water to the b.p. is admitted.

B. M. VENABLES.

Apparatus for degasifying liquids. SOC. ITAL. PIRELLI, and L. EMANUELI (B.P. 332,542, 19.4.29).—The liquid, such as insulating oil, is sprayed, after heating if necessary, into a vacuum chamber and discharged therefrom preferably at an increased pressure. A suitable form of spray is described.

B. M. VENABLES.

Gas-washing apparatus. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 332,283, 18.4.29).—The gas is subjected to liquid sprays while passing through louver-like baffles; the dust is removed from the apparatus while still in suspension in the liquid, for which purpose the bottom of the apparatus is conical and flushed by sprays.

B. M. VENABLES.

Filters for gases. H. WITTEMEIER, Assec. of E. H. SCHÄFER (B.P. 331,754, 7.9.29. Ger., 27.10.28).—A filter composed of a stack of metal plates or grids is arranged so that the plates may be slid out for cleaning one group at a time; a spring holds the remainder of the pack up against the abutment at the discharge side.

B. M. VENABLES.

Air filters. I. L. BRAMWELL, C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 332,601, 23.1.29).—A system comprising a number of groups of bag-filters is maintained under reduced pressure by an induced-draught fan, even the inlets of the filters being under slight vacuum; to clean a group by reverse current

without stopping the operation of the others it is necessary only to cut off that group from the fan and open the clean or outlet side to atmosphere.

B. M. VENABLES.

Separator of liquids and gases, intended particularly for use in gas-compressing plants. H. W. WILDISH (B.P. 331,704, 18.7.29).—The separator is of the fixed type and has inclined baffles with the outlet above them.

B. M. VENABLES.

Apparatus and method for testing gases. R. B. WASSON (U.S.P. 1,753,675, 8.4.30. Appl., 24.10.25).—The sample of gas is held at a constant volume before, during, and after the test and the reduction in pressure due to the absorption of a constituent is determined. The apparatus comprises two concentric chambers, a pump, manometer, and the necessary multi-way cocks. In determining for instance, the carbon dioxide in combustion gases, the inner chamber is completely emptied of gas by pumping caustic liquor covered with a layer of oil into it from the outer chamber; the sample of gas is then drawn into the inner chamber at atmospheric pressure (independent of the pressure of supply) by simply allowing the liquid level to equalise in the inner and outer chambers. The inner chamber is then isolated and the pump used to draw liquid from the inner chamber and deliver it back in the form of a spray which will break through the oil film and absorb the carbon dioxide.

B. M. VENABLES.

Treatment of fluids with solid substances. T. T. GRAY, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,753,859, 8.4.30. Appl., 18.1.26).—Fluids such as petroleum products are treated with solid substances which are supported in separate containers within a shell, either above or below the level of the liquid, thus preventing the solid reagents from becoming diffused through the petroleum or mixing with each other. Provision is made for easily handling the containers through a man-hole.

B. M. VENABLES.

Apparatus for bringing liquids and gas or vapours and gas into intimate contact. S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & Co. (B.P. 331,818, 3.4.29).—The apparatus consists of a series of compartments each fitted with stationary brushes through which the liquid and gas pass.

D. K. MOORE.

Transferring liquefied gases from one container to another. C. W. P. HEYLANDT (U.S.P. 1,753,785, 8.4.30. Appl., 8.3.29. Ger., 2.3.28).—In the transport or bulk-storage vessel, the liquid is maintained at about its b.p.; when it is required to force liquid out, a small portion is allowed to pass through an evaporator coil and the vapour readmitted to the upper part of the tank.

B. M. VENABLES.

Control of proportions of components of gaseous mixtures. W. R. SCHOFIELD, JUN., and R. D. MILNER, Assrs. to LEEDS & NORTHRUP CO. (U.S.P. 1,753,319, 8.4.30. Appl., 7.3.24).—A change in composition and therefore of thermal conductivity of a sample of gas is caused to unbalance a Wheatstone bridge of which two arms are formed of wires (carrying heating currents) having a high temperature coefficient situated in the sample and a standard gas, respectively. The movement of the

galvanometer of the bridge is translated to control, *e.g.*, a valve or damper by known means. The sample and standard gas are situated in adjacent cells and the former is changed continuously by convection only, both the inlet and outlet pipes to the cell being arranged at the same cross-section of the sample pipe and perpendicular to the flow of gas in it. B. M. VENABLES.

Heating of fluids. C. W. STRATFORD (B.P. 305,205, 18.1.29. U.S., 2.2.28).—In a still or boiler having a stationary, internal, cylindrical heating surface, the liquid is projected against the heating surface by jets which are evenly spaced and are submerged in the body of the liquid. A portion of the liquid is withdrawn by a pump and continuously circulated through the jets.

B. M. VENABLES.

[Fractionating] still. Scrubber. J. A. CAMPBELL (U.S.P. 1,754,605 and 1,754,607, 15.4.30. Appl., [A] 19.4.27, [B] 5.11.28).—In (A) a fractionating column is fed at an intermediate height through an axial column pipe, the trays are perforated, and the downcomers are in the form of sharp-edged froth-breaking trays raised above the plates and provided with pipes leading to near the surface of the plate below. The froth collectors below the feed inlet are preferably larger than those above it. In (B), prior to entering a gasoline absorption plant, natural gas is cleaned by selective absorption in a tower provided with perforated conical trays above which are sharp-edged froth-breaking collectors or trays. It is intended that the crude oil, water, and solid impurities should run down the conical trays, and drains are provided from their apices direct to a separate discharge chamber at the bottom of the tower. The lighter absorption liquid which rapidly becomes saturated with the petrol vapour should form froth and, after being caught in the collectors, run down only one stage at a time to a sump, thence to be pumped either wholly to the top tray or partly to the others.

B. M. VENABLES.

Gaseous exothermal catalyses. O. PIETTE, and UNION CHIM. BELGE, SOC. ANON. (B.P. 332,532, 12.4.29).—A number of twin concentric tubes (the lower ends of the larger tubes having closed ends) are buried in the catalyst mass and the gas before the reaction is passed downwardly through the annular spaces, upwardly through the inner tubes, then through the catalyst downwardly, so that the flow of cold gas and reacting gas is concurrent and the temperature of the catalyst more uniform.

B. M. VENABLES.

Solidification of carbon dioxide or other gases. H. W. COLE and M. W. McLAREN (B.P. 312,231, 13.5.29 U.S., 22.5.28).—An apparatus is described by which liquid carbon dioxide (or other gas) may be expanded in an entirely closed chamber under carefully controlled pressure and temperature and the snow compressed in that chamber or in an adjacent ram cylinder, so that hard blocks of solid may be obtained. The pressure incidental to the original compression may be used to work the hydraulic ram.

B. M. VENABLES.

Hygrometers. W. LUBACH and E. GIESER (B.P. 332,135, 1.10.29).—The hygroscopic element of an electric hygrometer is made of glass, earthenware, or

other material with a glassy surface. An ammeter or electric-discharge lamp may be used as the measuring instrument.

D. K. MOORE.

Process for extinguishing fires. O. TREICHEL (U.S.P. 1,771,151, 22.7.30. Appl., 31.10.27. Ger., 15.11.26).—See B.P. 280,543; B., 1928, 628.

Filter press. H. JUNG (U.S.P. 1,771,928, 29.7.30. Appl., 1.12.28. Ger., 2.5.27).—See B.P. 322,091; B., 1930, 126.

Distillation or like columns. A. KUHN, Assr. to C. STILL (U.S.P. 1,772,399, 5.8.30. Appl., 15.3.26. U.K., 24.3.25).—See B.P. 239,791; B., 1925, 869.

Production of a cooling liquid of low f.p. F. WERNER (U.S.P. 1,771,517, 29.7.30. Appl., 3.11.25. Ger., 23.9.25).—See B.P. 260,760; B., 1927, 65.

Apparatus for washing, separating, or concentrating minerals and other granular materials. J. BRASS (B.P. 332,682, 27.5.29).

Pasteurising apparatus. P. N. GRØNBORG (B.P. 319,742, 24.9.29. Denm., 27.9.28).

Tanks for receiving and separating mixed solid and liquid materials. ASH CO. (LONDON), LTD. (B.P. 311,816, 26.4.29. U.S., 17.5.28).

Opening and closing of cylinders or capsules for compressed or liquefied gas. SOC. ANON. D'APPLICATIONS DES GAZ LIQUÉFIÉS (B.P. 318,546, 28.8.29. Fr., 5.9.28).

Production of foam for fire-extinguishing purposes. MINIMAX A.-G. (B.P. 320,020, 30.9.29. Ger., 29.9.28).

Separating particles of different densities (B.P. 331,841). **Fractional condensation** (B.P. 331,485 and 331,526).—See II. **Preventing corrosion of water-pipes** (B.P. 331,725). **Mercury-vapour boilers** (B.P. 310,880).—See X. **Grinding mills** (B.P. 331,762).—See XIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Ultimate analysis of coal. F. SCHULZ (Coll. Czech. Chem. Comm., 1930, 2, 486—488).—In the ultimate analysis of coal and coke only very small quantities of nitrogen oxides are formed during the combustion in oxygen, and the error due to their absorption does not exceed 0.1% C.

H. F. GILLBE.

Extraction of Miike coal [with tetralin and phenol]. K. FUJIMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 201—202 B).—After prolonged extraction with heated tetralin in an atmosphere of carbon dioxide, about 30% of the sample was dissolved. A further 10% of the residue was similarly dissolved by phenol. The residues from these extractions contained rather less volatile matter and were less strongly coking than was the original coal.

C. IRWIN.

Determination of volatile matter of Japanese coals by means of the high-temperature analytical balance. T. SOMIYA and S. HIRANO (J. Soc. Chem. Ind., Japan, 1930, 33, 252—254 B).—The change of weight of a number of coal samples in a nitrogen-filled electric furnace over the temperature range 800—1200°

was measured. Higher values were obtained if the coal was heated immediately to at least 800° without pretreatment at 550°, but results by the latter method were the more consistent. In this case all curves became practically straight lines at about 950°, and this temperature is recommended for volatile matter determinations in proximate analysis. C. IRWIN.

Micro-pyrometer and gas-furnace methods for determination of fusion point of coal ash. M. C. K. JONES, H. FARMER, J. E. BREWER, and H. C. PORTER (Ind. Eng. Chem. [Anal.], 1930, 2, 325—328).—Results of ash fusion-point determinations on ten samples of bituminous coal and three of anthracite, carried out in different laboratories, are compared and fairly satisfactory agreement is found between results obtained by the micro-pyrometer method and those obtained by the gas-furnace method. Buckwheat anthracites that clinkered in household furnaces using blowers were indicated by the standard gas-furnace test to have highly refractory ash, whilst the micro-pyrometer test indicated in some instances a moderate clinkering tendency.

E. S. HEDGES.

Coking coal mixed with iron minerals. N. N. ROGATKIN, L. P. UKHOV, and D. G. JOFFE (J. Chem. Ind., Russia, 1929, 6, 213—214).—When coal is coked with magnetic iron minerals the coke obtained possesses good resistance to pressure and a high reacting capacity.

CHEMICAL ABSTRACTS.

Purification of spectrum carbon. G. HEYNE (Z. angew. Chem., 1930, 43, 711—712).—Carbon to be used as carrier for spectrum analysis of powders, solutions, etc. is purified by heating in a current of nitrogen and hydrogen either in a charcoal tube to 2500° for 30 min., or between charcoal blocks electrically; by the latter means a temperature of 3000° may be attained when a shorter heat-treatment is needed. The sample is freed completely from iron and aluminium, and from most of the silicon, calcium, and magnesium.

H. I. DOWNES.

Calorific value of coconut husks. S. H. J. WILSON (New Zealand J. Sci. Tech., 1930, 12, 14—15).—A sample of husks from the Cook Islands contained 21.5% of outer skin, 49.6% of fibrous husk, and 28.9% of shell. Duplicate samples of these three materials gave average calorific values of 4113, 4113, and 4382 g.-cal., respectively, and for the whole material, 4192. As a fuel the material is excessively bulky.

E. HOLMES.

Nomogram for calculating calorific value of gases. L. J. ROGERS (Gas World, 1930, 93, 119).—Examination of the olefines in low- and high-temperature gases at the Fuel Research Station has shown that their calorific value varies from 2500 to 2200 B.Th.U. per cub. ft.; these figures are used in constructing a nomogram for calculations with mixtures.

R. H. GRIFFITH.

Water-gas tar. A. V. SIVOLOBOV (J. Chem. Ind., Russia, 1929, 6, 206).—Phenols, pyridine bases, and carbazole are almost entirely absent; the crystalline products (up to 16%) contain naphthalene (crude, 50%) and a considerable quantity of easily polymerisable unsaturated compounds. The anhydrous tar contains

benzene 0.7, toluene 0.03, crude naphthalene 8, anthracene 1.1, phenanthrene 2, and acenaphthene 0.8%.

CHEMICAL ABSTRACTS.

Determination of phenol and *m*-cresol in crude phenol oil. P. K. SAKMIN (J. Chem. Ind., Russia, 1929, 6, 890—892, 966).—The separation of phenols with alkali is described. Fractional distillation affords a fraction of b.p. up to 195°, containing nearly all the phenol, which is determined by a simplified Masse-Lerou procedure; in the fraction, b.p. 195—207°, the *m*-cresol is determined by Raschig's method of nitration. If much is present, however, 80% nitric acid must be employed.

CHEMICAL ABSTRACTS.

Purification of naphthalene. I. E. BESPOLOV (Nef. Choz., 1929, 17, 737—738).—Distillation is preferably preceded by removal of oil.

CHEMICAL ABSTRACTS.

Perm crude oil. R. A. VIRABYANTZ (Nef. Choz., 1930, 18, 196—201).—The composition is recorded; the sulphur content is high. The results of cracking tests are also given.

CHEMICAL ABSTRACTS.

Corrosive action of petroleum. M. BLAGODAROV and V. SHEVLYAKOV (Azerbaij. Nef. Choz., 1930, 106—113).—The copper strip test is satisfactory. Hydrogen sulphide must be removed with alkali before treatment with 90—94% sulphuric acid; otherwise it is oxidised to sulphur. Treatment with hypochlorite may be substituted for that with sulphuric acid.

CHEMICAL ABSTRACTS.

Decomposition of Surakhani crude oil and its distillates. I. E. BESPOLOV (Azerbaij. Nef. Choz., 1930, No. 3, 60—73).—Pipe-stills yield more unsaturated hydrocarbons than do shell-stills; deep cracking of mazout increases the yield of aromatic compounds. Vapour-phase cracking favours the formation in the tar of aromatic substances (optimum temperature 650°); the yield of gasoline and kerosene fractions is smaller and of gas and tar larger than in liquid-phase cracking. Lengthening of the cracking time favours the formation of aromatic substances at the expense of unsaturated hydrocarbons. Deep cracking of cracked gas oil yields a tar practically free from saturated hydrocarbons.

CHEMICAL ABSTRACTS.

True boiling crude analysis [of petroleum crude oils]. G. A. BEISWENGER and W. C. CHILD (Ind. Eng. Chem. [Anal.], 1930, 2, 284—287).—An improved method for determining the quantity and quality of products obtainable from the crude oils is described, and consists in distilling the sample in an efficient true b.p. apparatus, followed by correct blending and inspecting. The method has many advantages: it is inexpensive and rapid, maximum yields of more valuable products can be determined and yields may be correlated with refinery operation, and considerable additional information is obtainable, such as mol. wts. and flash temperatures necessary for pipe-still operations.

E. S. HEDGES.

Atmospheric cracking of residue from Apsheron crude oils. V. SHEVLYAKOV and M. VOLF (Azerbaij. Nef. Choz., 1930, 96—100).—Coke (9—19%), gasoline (10%), kerosene (24—30%), and residue of high m.p. (30—40%) were obtained. The gasoline contained

aromatic and unsaturated hydrocarbons, but little naphthenes.

CHEMICAL ABSTRACTS.

Crude oil resins. N. A. VASILIEV and L. V. ZHIRNOVA (Nef't. Choz., 1929, 17, 707—712).—After removal of asphaltenes and naphthenic acids the resins from Balakhani-Sabunchi crude oil were separated with silica gel; fuller's earth adsorbed little and caused polymerisation. Vacuum distillation prevents the formation of new resins if carried out in an atmosphere of carbon dioxide. The mol. wts. were 226 and 1112 (silica gel), or 504 and 927 (fuller's earth) for the resins in the lower and higher fractions, respectively. The composition corresponds with the formula $C_nH_{2n-m}O_p$, where n varies from 16 to 69, m from 8 to 40, and p from 1 to 3; less than one atom of sulphur is present. Grozni asphalt affords similar resins.

CHEMICAL ABSTRACTS.

Direct determination of oil in oil-water-soap emulsions. L. N. MARKWOOD (Ind. Eng. Chem. [Anal.], 1930, 2, 265).—The following method for determining oil in emulsions is based on the fact that emulsions of the oil-in-water type stabilised by soap are resolved by phenol. It has the advantages over the usual technical method that it is direct, can be completed in 20—30 min., and no inflammable solvents are required. About 5 g. of the sample are introduced into a 10-c.c. cylinder, 3 c.c. of 75% phenol solution are added in drops, and the contents of the cylinder gently mixed. The cylinder is then centrifuged and the volume of the oil layer read. The density of the oil is determined in a small pycnometer and the percentage of oil calculated.

E. S. HEDGES.

Coal from Kuznetski basin, Siberia. N. M. KARAVAEV and I. B. RAPOPORT (Izvest. Teploekhn. Inst., 1929, No. 8, 15—30).

Nature and properties of Siberian boghead [coal]. G. L. STADNIKOV (Proc. II. Int. Conf. Bit. Coal, 1929, 1, 625—631).

Ammonia. THAU.—See VII. Cresylic acids as flotation agents. LANDOLT and others.—See X.

PATENTS.

Manufacture of briquettes, fire-lighters, and other consolidated fuels. H. ALEXANDER and J. A. GREENE (B.P. 330,295, 6.2.29).—A mixture of a substance which contains the necessary binding agent, *e.g.*, bituminous coal or resinous saw-dust, with anthracite dust, coke dust, etc. is heated or carbonised in moulds provided with an anti-adhesive lining, *e.g.*, wood pulp or graphite. A relatively small quantity of an additional binding material, *e.g.*, water or dolomite, may be added.

A. B. MANNING.

Manufacture of charcoal fuel briquettes. C. DEMOULIN (B.P. 332,815, 23.9.29).—Powdered wood charcoal is mixed at about 100° with wood-tar pitch and aqueous (3% solution of) alkali. The resulting emulsion spreads over the solid particles and produces a paste which is then briquetted. Sodium nitrate may also be included.

R. H. GRIFFITH.

Separation of [coal] particles of different densities by means of [heavy] liquids. C. W. H. HOLMES,

and BIRTLEY IRON CO., LTD. (B.P. 331,841, 8.3.29).—In the separation of coal by floating it in a heavy liquid, the mixed material is first sprayed with or dipped in paraffin or other liquid that will prevent the heavy liquid (*e.g.*, crude varnish) from wetting it; it is then treated in a liquid of intermediate density, and only the part that sinks is further treated in the heaviest liquid.

B. M. VENABLES.

Stabiliser for fuel suspensions. H. G. C. FAIRWEATHER. From AMER. COALINOL CORP. (B.P. 332,343, 25.5.29).—A suspension of powdered coal, or similar solid material, in a mineral, vegetable, or animal oil, coal tar, etc. is stabilised by the addition of a soap-like substance made by oxidation of a high-boiling oil containing olefines and paraffins; air or oxygen is blown through the hot oil in the presence of a metallic salt of an organic acid, such as manganese oleate, and the reaction product is neutralised and saponified by means of alkali, before addition to the suspension.

R. H. GRIFFITH.

Coal-carbonising retorts etc. T. M. DAVIDSON (B.P. 331,906, 8.4.29).—A vertical retort is provided with a close-fitting internal structure, which forms a large number of small pockets close to the walls. The maximum depth of heat transfer through the coal is about 3 in. The retort widens towards the base, and the central structure is movable vertically, through a special lower door, by means of a hydraulic ram.

R. H. GRIFFITH.

Carbonisation of agglomerates of non-bituminous coals. G. M. F. F. MASCART (B.P. 331,850, 8.4.29).—In carbonising briquetted coals containing less than 14% of volatile matter, it is possible to use not more than 7% of pitch as binding material if heating is carried out initially at 500° and finally to about 900° during 10 hrs. The amount of unchanged pitch recovered with the tar depends on the initial temperature, decreasing from 60 to 70% at 500° to about half that amount at 750°. The resulting coke is of high bulk density and coherence.

R. H. GRIFFITH.

Carbonising coal and cracking liquid hydrocarbons. TRENT PROCESS CORP. (B.P. 313,158, 21.5.29. U.S., 9.6.28).—Coal is continuously carbonised in a vertical retort provided throughout its length with tubular off-takes. The residual hot coke descends into an oil-bath and is there quenched, the vapours produced passing upwards into the retort, where they are cracked and the reaction products escape by way of the tubular off-takes.

R. H. GRIFFITH.

Wet carbonisation. D. W. BERLIN and J. A. BRINCK (B.P. 317,401, 13.8.29. Swed., 15.8.28).—A paste of peat or similar substances is forced through the inner of two concentric tubes, and a liquid heating medium moves countercurrent in the other. Means are also provided for transfer of heat from the treated material to the circulating liquid, to which additional heat can be applied as desired. Gas evolved in the apparatus escapes through a valve, and the tubes are constructed to withstand an increase in pressure.

R. H. GRIFFITH.

Apparatus for low-temperature distillation, drying, etc. J. Y. JOHNSON. From I. G. FARBENIND.

A.-G. (B.P. 331,879, 11.2.29).—A rotating bundle of inclined tubes is mounted on a water-cooled shaft in such a way that the tubes can expand freely through end-wall borings. Heating is effected by passing gases round the tubes and through the support-plates. Gravity feed and extraction gear are employed to convey material, such as powdered coal, through the apparatus.

R. H. GRIFFITH.

Gasifying fuels in a molten-ash-type producer, and simultaneously treating ores or residues. H. J. F. PHILIPON (B.P. 332,366, 15.6.29).—Fuels with a high ash content, such as domestic refuse, schist from coal seams, etc., are gasified in a producer so that the clinker produced becomes thoroughly molten. The nature and quantity of the slag formed is regulated by previous addition of inorganic material, and by controlling the temperature by alterations in the degree of preheating of the blast air. The process can be adapted to working up of cast iron, cement slag, calamine and other ores, and to the recovery of phosphoric acid from phosphate ores. Conditions are adjusted to give a slag of optimum fusibility, and the process is designed to work most satisfactorily with an initial fixed carbon content of about 30%.

R. H. GRIFFITH.

Destructive hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,251, 17.1.29).—In order to avoid local overheating, high-pressure hydrogenation of carbonaceous material is carried out in a reaction vessel consisting of several gas-heated vertical tubes. The gases are not allowed to burn on or near the tubes and are previously mixed with effluent gases to lower their temperature before being circulated through the system. The heating media may be 100° hotter than the reaction temperature, provided that they do not exceed 800° and that they are uniform in character.

R. H. GRIFFITH.

Destructive hydrogenation of solid carbonaceous material. C. F. R. HARRISON, H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,246, 17.4.29).—Catalysts for assisting liquefaction of coal and similar solid materials are added in the form of a solution (preferably saturated) of a soluble salt so that they are evenly distributed throughout the mass. The amount of liquid added should be insufficient to form a paste with the powdered coal. The impregnated material may be dried and mixed with oil. Nickel nitrate and ammonium molybdate are mentioned as suitable salts.

R. H. GRIFFITH.

Destructive hydrogenation of brown coal and other oxygen-containing low-grade fuels. H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,222, 31.12.28).—The initial material is comminuted and then dried and heated, in two stages if desired, in the presence of a non-oxidising gas, so that not only is drying effected without oxidation, but oxygen compounds already present are partly eliminated as carbon dioxide and water. The product is formed into a paste with a suitable oil or tar and submitted to destructive hydrogenation.

A. B. MANNING.

Preparation of liquid products from coal or coal-containing materials and liquefaction of such

materials. II. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 330,223, 31.12.28).—The initial materials are heated with hydrogen under pressure to a maximum temperature (350–550°), with or without catalysts, and the heating is then immediately discontinued so that the mass begins to cool. The gaseous products are drawn off while still hot (e.g., at 325°), and are cooled; the non-condensed gases contain a large proportion of totally oxidised compounds, e.g., carbon dioxide. The residue is then strongly heated with hydrogen under pressure, with or without catalysts, and maintained for some time in contact with hydrogen at the maximum temperature; the gaseous products are drawn off while still hot, cooled, and the non-condensed gases, consisting chiefly of hydrocarbons of high calorific value, are used as fuel. The residue in the reaction vessel may, if desired, be again submitted to an analogous treatment. Substantially the whole of the sulphur present in the initial materials is removed in the first stage of the process as hydrogen sulphide. Molybdenum and iodine, or their compounds, are suitable catalysts.

A. B. MANNING.

Gas-manufacturing installations. A. H. LYNN and N. J. BOWATER (B.P. 330,250, 12.2.29).—The installation comprises a carbonisation plant, a water-gas plant, means for mixing the coal gas from the former with the "run" gases from the latter to produce a mixture of suitable quality for use as distribution gas, and means for mixing a portion of the "run" gases and/or a portion of the coal gas with the "blow" gases of the water-gas plant to produce a gas suitable for heating the carbonisation plant. When carburetted water-gas is required to augment the output of the carbonisation plant the water-gas plant may operate alternately to produce carburetted water-gas in one cycle of operations, and blue water-gas for admixture with the "blow" gases to produce heating gas in another cycle.

A. B. MANNING.

Gas and steam producer generator. J. BELLAY (U.S.P. 1,753,847, 8.4.30. Appl., 1.8.27).—A water-gas generator is fitted with an annular boiler, which is further heated by all or part of the gas produced, and supplies process steam to the plant.

R. H. GRIFFITH.

Production of water-gas. D. TYRER, H. G. WATTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,221, 31.12.28).—Finely-divided carbonaceous material is completely gasified in steam, or in steam and oxygen, by passing the mixture, at a velocity not exceeding about 10 cm. per sec., and with a stream-line motion, in contact with a highly heated surface. A relatively wide, vertical generator is used, preferably divided into a number of successive zones of temperature, e.g., a preheating zone, a reaction zone, and a hydrocarbon-decomposing zone. The carbonaceous material and the gasifying agent may be introduced together at either the top or bottom of the generator; or the former may be introduced at the top and the latter at the bottom, in which case the rate of gas supply should be less than the rate of settling of the material in the still gas.

A. B. MANNING.

Manufacture of producer gas. REID POWER DEVELOPMENT Co., and J. W. HINCHLEY (B.P. 332,007,

30.5.29).—Gas from a lime kiln, essentially carbon dioxide, is passed to a coke-filled producer so that the gas from the latter contains large amounts of carbon monoxide. The quantities of air and gas fed to the kiln and producer are regulated in accordance with the temperatures attained, to give a gas of the required composition. The supply of combustible gas for heating the kiln is controlled by a valve which responds to the producer temperature. R. H. GRIFFITH.

Purification of technical gases, more particularly coke-oven gases which are to be subjected to decomposition by low-temperature cooling. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 307,886, 15.3.29. Ger., 15.3.28).—Deposition of resinous materials during the low-temperature fractionation of coal gas is prevented by preliminary removal of cyclic diolefines. This is effected by treatment with hydrogen, halogens, hydrogen halides, oxygen, etc., or by washing with tetralin or similar high-boiling oils. In the former method a catalyst, such as aluminium chloride for chlorination, may be used, and the latter method is best carried out under increased pressure. R. H. GRIFFITH.

Removal of sulphur from fuel gases. S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (B.P. 332,147, 15.10.29).—In purifying crude gases from hydrogen sulphide by means of iron oxide, either dry or as an aqueous suspension, wetting of the solid or dilution of the suspension is prevented by previous drying of the gas. For this purpose a hygroscopic liquid is used such that in subsequent treatment at 35–40° no further deposition of water occurs. R. H. GRIFFITH.

Production of hydrogen [from methane]. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,571, 19.4.29).—Thermal decomposition of methane is carried out alternately with the water-gas reaction and the combustion of carbon in a coke-filled chamber. The temperature is initially raised to about 1500° by blowing with air, and the decomposition of methane then proceeds until the temperature has fallen to about 1100°. Between this point and 900° steam is added, after which the cycle is repeated. The different stages can be balanced so that no coke, but only deposited carbon, is consumed. R. H. GRIFFITH.

Production of acetylene. R. M. WINTER, P. H. HULL, J. FERGUSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,731, 12.7.29).—Methane is mixed with oxygen or air and passed rapidly through a tube at above 1000°, when appreciable amounts of acetylene are formed. The mixture should contain as much oxygen as is possible without explosion, and ignition of the gas must be prevented by the use of a gauze placed before the reaction zone. The yield of acetylene is increased by raising the temperature and by selecting an optimum velocity; a maximum of about 10% is obtainable. R. H. GRIFFITH.

Manufacture of acetylene and hydrogen from hydrocarbons or gas mixtures containing same. J. Y. JOHNSON. From I. G. FARBENTIND. A.-G. (B.P. 332,057, 12.7.29).—Hydrocarbons etc., preferably under slightly reduced pressure, are passed through one or more

electric arcs and the acetylene formed is wholly or partly separated, part of the residual gas being recirculated after admixture with fresh gas, whilst another part, together with an addition of hydrocarbons or other gases if desired, is treated in another arc or arc system. H. S. GARLICK.

Vaporisation of heavy hydrocarbons. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 316,552, 23.7.29. Fr., 30.6.28).—In order to prevent the formation of hard pitchy and carbonaceous deposits in a vaporiser for heavy oils, heating is carried out in two stages, but in a single vessel. Oil fed to the top of this vessel trickles over heated baffle plates, which are at such a temperature that partial volatilisation occurs but that the residue is sufficiently liquid to flow on to a packing of porous material. The deposit which forms in the lower, hotter zone can then be removed at intervals by combustion. R. H. GRIFFITH.

Cracking of oils. C. P. DUBBS (B.P. 331,569, 5.3.29).—Oil is heated to cracking temperature under cracking pressure in a heating zone and directly passed into an adjacent separating or reacting zone where the vapours are separated and dephlegmated. The residual oil is continuously removed without coming into contact with reflux condensate or with fresh oil. In order to obtain the maximum amount of useful fuel oil the polymerisation of the unvaporised residual oil is checked by maintaining the lowest possible level of the latter in the separating zone and by regulating both the rate of withdrawal of the residual oil and the dissipation of heat therefrom. H. S. GARLICK.

Cracking of hydrocarbons. J. H. BRÉGEAT (B.P. 331,531, 27.3.29).—Asphaltic, paraffinous, and resinous substances extracted from petroleum, low-temperature distillation tars, acid sludges from petroleum refining, waxes, and natural asphalt are subjected to cracking in a retort at 350–460° with continuous agitation, either with or without the addition of abrasive material. The level of the substance in the retort is maintained substantially constant throughout the treatment and the rate of distillation regulated so that the quantity of distillate collected hourly comprises 5–14% by wt. of the effective capacity of the retort. The distillate is subjected to reflux condensation, and that portion distilling above 300° is returned to the cracking circuit. H. S. GARLICK.

Manufacture of liquid hydrocarbons and hydrocarbon derivatives. J. Y. JOHNSON. From I. G. FARBENTIND. A.-G. (B.P. 331,558, 7.1.29).—Distillable carbonaceous materials are subjected to destructive hydrogenation under a pressure of 20 atm. or more in the presence of at least three different constituents, one being an oxide of a metal of group II or III or a compound thereof which is solid under the conditions of working, e.g., kaolin or Florida earth, and the other two being oxides, hydroxides, or sulphides of different metals of the type of cobalt, nickel, or metals of group V or VI. H. S. GARLICK.

Refining system [for hydrocarbon oils]. A. G. PETERKIN, JUN., ASSR. to ATLANTIC REFINING CO. (U.S.P. 1,752,455, 1.4.30. Appl., 8.4.26).—Hydrocarbon vapours are passed upwardly in contact and in interchange

relation with descending liquid reflux oil. Oil fractions are removed from the fractionating zones and a solid purifying agent, *e.g.*, fuller's earth, is suspended in at least a portion of a fraction so removed and the suspension passed in fractionating relation with the ascending vapours.

H. S. GARLICK.

Purification of gas, particularly such as results from gasification of heavy oils. C. CHILOWSKY (B.P. 317,332, 13.8.29. Fr., 13.8.28).—Gas is bubbled into a liquid contained in vertical tubes which are provided with means for circulating a cold fluid round them and which establish connexion between a tank beneath them, likewise filled with the bubbling liquid and provided with a means for feeding hot gas thereto and with a collector above them having means for discharging the cooled gas through a valve immersed in the bubbling liquid. Preferably the level of the bubbling liquid is above the top of the ends of the tubes. When the gasification of heavy oils is effected in the presence of steam the vessel in which bubbling takes place is connected by an overflow pipe to a settling vessel, the water necessary for the supply of the gas generator being withdrawn from the settling vessel by a pump through a filter.

H. S. GARLICK.

Apparatus for lye treatment of petroleum oils. C. K. PARKER, Assr. to STANDARD OIL Co. of CALIFORNIA (U.S.P. 1,752,350, 1.4.30. Appl., 12.7.26).—The oils are continuously mixed with an immiscible alkaline treating agent, heated and supplied under pressure to an enlarged, horizontal, pressure, settling chamber, through an inlet of gradually increasing cross-sectional area. Oil and lye are separately discharged through an outlet larger than the main section from vertically separated points.

H. S. GARLICK.

Fractionally condensing (A) composite vapours, (B) petroleum vapours and other vapour compounds. A. LOSEY, P. GOTTLIEB, and L. L. HAUPT (B.P. 331,485 and 331,526, 25.2.29. U.S., [A] 11.12.28, [B] 10.10.28).—(A) The vapours are passed into fractionating apparatus consisting of a series of compartments in which fuel oil, lubricating stock, distillate, and gas oil are successively condensed in separate compartments by causing the vapours to perform successive limited amounts of work, the resulting vapours being passed into an adjacent compartment and fractionally condensed by heat exchange with a fraction returned under increased pressure. (B) The heated vapours of crude oil are passed through a multistage expansion device, whereby they are fractionally condensed into a series of oil products.

H. S. GARLICK.

Breaking [water-petroleum oil] emulsions. J. W. BECKMAN (U.S.P. 1,753,641, 8.4.30. Appl., 21.12.25).—Natural crude oil emulsions containing an emulsifying agent are treated with a medium adapted to sustain the life of, and are inoculated with, living micro-organisms able to destroy such emulsifying agent, the mixture being agitated at intervals during the life action and the temperature maintained at 20–40°. H. S. GARLICK.

Stabilising absorption system [for gasoline]. J. A. CAMPBELL (U.S.P. 1,754,606, 15.4.30. Appl., 19.5.27).—The vapour-laden gases (casing-head gas) are passed upwardly through a downwardly moving stream

of absorbing liquid, and the vapour-laden liquid is removed from the lower end of the stream and heated to vaporise the absorbed liquids. The lean absorbing liquid is returned to the stream. The lower part of the absorbing stream is maintained at a higher temperature than the upper part, by introducing therein a certain amount of the heated absorbing liquid.

H. S. GARLICK.

Gasoline filtering system and apparatus. B. V. STOLL, Assr. to STOLL OIL REFINING Co. (U.S.P. 1,752,709, 1.4.30. Appl., 14.3.29).—Gasoline vapours from a cracking process are condensed, immediately filtered through a decolorising absorbent, and discharged into an alkaline mixer from which it passes into storage through pressure-release valves.

H. S. GARLICK.

Motor fuel. H. MENZ, W. STEFFEN, and E. JAAKS-MÜNCHBERG (B.P. 331,966, 30.4.29).—A vaporising fuel comprises 50–55% of a hydrocarbon, preferably hydrogenated, *e.g.*, tetrahydronaphthalene, about 10% of sulphonated vegetable or animal oil soluble in water, and about 35% of distilled water containing 5% of waste sulphite-lye dissolved therein. H. S. GARLICK.

Manufacture of non-knocking engine fuels of the benzene type. I. G. FARBERIND. A.-G. (B.P. 303,894, 5.1.29. Ger., 13.1.28).—Benzene (3–7 pts. by wt.) obtained from the destructive hydrogenation of brown coal, tars, or mineral oils are blended with 1–3 pts. of benzene isolated from the destructive hydrogenation of mineral coal, *e.g.*, anthracite.

H. S. GARLICK.

Treating fuel oils. A. H. ACKERMAN, Assr. to CATALYTIC CHEM. Co. (U.S.P. 1,754,297, 15.4.30. Appl., 16.2.27. Renewed 8.12.28).—Petroleum coke is ground with a small proportion of a composition comprising anthracene 10 pts., nitrobenzene 4 pts., sulphuric acid 2 pts., caustic soda 1 pt., and naphthalene 200 pts., and the resulting mixture is agitated with fuel oil until dissolved. A product having increased calorific value is claimed.

H. S. GARLICK.

Coke oven. H. SCHRÖDER (U.S.P. 1,770,998, 22.7.30. Appl., 28.11.25. Ger., 12.4.24).—See B.P. 254,960; B., 1926, 778.

Non-knocking motor fuel. A. C. EGERTON, Assr. to ASIATIC PETROLEUM Co., LTD. (U.S.P. 1,771,169, 22.7.29. Appl., 6.6.27. U.K., 29.7.26).—See B.P. 279,560; B., 1928, 292.

Valve-reversing devices for intermittent gas producers etc. BAMAG-MEQUIN A.-G. (B.P. 332,783, 22.8.29. Ger., 27.10.28).

Recovering oil and grease [from water etc.]. W. G. MORSE (B.P. 332,569, 23.2.29).

Contacting of gases and solids (B.P. 315,868). Testing gases (U.S.P. 1,753,675). Treatment of fluids (U.S.P. 1,753,859). Still and scrubber (U.S.P. 1,754,605 and 1,754,607).—See I. Activated iron oxide (B.P. 332,259).—See VII.

III.—ORGANIC INTERMEDIATES.

Determination of small amounts of acid in ether. L. P. HALL (Ind. Eng. Chem. [Anal.], 1930, 2, 244–246).—Formic and acetic acids occur among the products of

decomposition of ethyl ether, but pure ether is found to be neutral. The two-layer method of titration, using phenolphthalein as indicator, is subject to large errors when small amounts of acid are being determined, but a skilled operator can obtain satisfactory results by adding alcohol to keep the solution homogeneous. The ether-alcohol mixture exerts an influence on the colour of the phthalein indicators. The method now recommended is to substitute a sulphonephthalein indicator such as bromothymol-blue, taking care to avoid introduction of carbon dioxide. This indicator may be used satisfactorily also when the ether contains alcohol.

E. S. HEDGES.

Hygroscopicity of urea. S. OKA (J. Soc. Chem. Ind., Japan, 1930, 33, 227—228 B).—Hygroscopicity was measured by weight increase in a given time in contact with air of given moisture content. The hygroscopicity of urea was reduced by 25% by pressing into a cake. It was further lessened by melting with 10% of water and rapidly cooling, and further again by melting in absence of water. The last product is hygroscopic only in summer in Japan.

C. IRWIN.

Determination of the inorganic acidity of crude nitro-products. I. M. KARYAKIN and A. A. GOLUBEV (J. Chem. Ind., Russia, 1929, 6, 120—121).—Sufficient aniline to combine with the sulphuric acid is added, the salt being collected, washed with benzene or ether, and titrated. Crude nitronaphthalene is first dissolved in benzene; dinitrobenzene and dinitrotoluene are dissolved in aniline.

CHEMICAL ABSTRACTS.

Preparation of nitrosophenol and of indoles by means of aluminium nitrite. V. G. GULINOV (J. Chem. Ind., Russia, 1929, 6, 214—216).—A solution of aluminium nitrite is added, with stirring, to an aqueous solution of phenol and aluminium sulphate, stirring being continued for 30 min.; after 18—20 hrs. the precipitate of aluminium *p*-nitrosophenoxide (90—93% of theoretical yield) is collected, washed with cold water, and dried below 40°; alumina is removed by washing with hydrochloric acid (2—3%). The salt is treated with sodium hydroxide solution (5%), the nitrosophenol being precipitated with hydrochloric acid. In the preparation of indophenol for blue a solution of *o*-toluidine (10·7 pts.) in sulphuric acid (*d* 1·70—1·74) is treated at 1—3° with dry aluminium nitrosophenoxide (13·1 pts.) in small portions with stirring, which is continued during 1 hr. After 3 hrs. at 4° the mass is poured on ice and the acid neutralised; the precipitate is collected and washed with cold water. Herz's method (G.P. 199,963 of 1902) for obtaining the sulphur-blue dye is modified. In the preparation of indophenol for green, α -naphthylamine (14·3 pts.) is dissolved with cooling in sulphuric acid (180 pts.), aluminium nitrosophenoxide (13·1 pts.) being introduced in small portions, with stirring, at 3—5°, stirring being continued for 1 hr. After 2—3 hrs. at 5° the mixture is poured on ice, the precipitate being collected and washed with water at 0°. The preparation of the sulphur-green dye is described.

CHEMICAL ABSTRACTS.

Crude phenol oil. SAKMIN. Naphthalene. BESPOLOV.—See II.

PATENTS.

Manufacture of methyl alcohol [from carbon monoxide and hydrogen]. G. NATTA (B.P. 330,919, 11.3.29).—Carbon monoxide and hydrogen are passed under pressure (260—300 atm.) over smithsonite, especially smithsonite calcined at 400—450°, at about 400°. The conversion into methyl alcohol is 20%.

C. HOLLINS.

Removal of water from aqueous formic acid. H. T. CLARKE and D. F. OTHMER, Assrs. to KODAK, LTD. (B.P. 318,633, 5.9.29. U.S., 8.9.28).—*n*-Propyl formate is added to the aqueous formic acid, or is formed therein by addition of propyl alcohol. The mixture is heated below the b.p. of water so that an azeotropic mixture of water and *n*-propyl formate distils, the dehydrated formic acid being then separated from the remaining propyl ester by fractional distillation. W. J. WRIGHT.

Manufacture of arylacetic acids and substitution products thereof. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 330,916, 19.2.29).—Aromatic hydrocarbons, and such derivatives as contain no primary or secondary amino-groups, react with mono- or di-halogenated acetic acids or esters at 100—275° in absence of alkali to give arylacetic acids or esters. Chloroacetic acid heated with naphthalene at 165—185° for 3 days yields α -naphthylacetic acid, m.p. 129°. The preparation of 5-acenaphthylacetic acid, m.p. 187°, b.p. 180°/9 mm.; anthracylacetic acid, m.p. 189°; fluorenylacetic acid, m.p. 148°; 4-bromo- (m.p. 122—128°) and 4-chloro- (m.p. 124°) α -naphthylacetic acids; ethyl α -naphthylacetate, m.p. 129—130°; 2-hydroxy- α -naphthylacetic acid, m.p. 106—107°, b.p. 275°/13 mm.; and di- α -naphthylacetic acid, m.p. 216—218°, is also described.

C. HOLLINS.

Manufacture of sulphonation products of unsaturated hydroxy-fatty acids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,904, 18.3.29).—Air charged with sulphur trioxide is passed over the surface of a well-stirred solution of castor oil or ricinoleic acid in about 3 pts. of an organic solvent (carbon tetrachloride, trichloroethylene). Preferably the hydroxyl group is first sulphonated with 100% sulphuric acid at 30—40°, and the ester is then treated with sulphur trioxide at —10° to —3°. The products are not precipitated by acids or lime.

C. HOLLINS.

Manufacture of *NN'*-thio-derivatives of amines [*NN'*-disulphides]. IMPERIAL CHEM. INDUSTRIES, LTD., T. BIRCHALL, and S. COFFEY (B.P. 331,016, 18.4.29).—Secondary amines react with sulphur monochloride (disulphur dichloride) in neutral or alkaline aqueous solution or suspension to give *NN'*-disulphides. Products from diethylamine, ethylaniline, and piperidine are described.

C. HOLLINS.

Manufacture of phenolic compounds. I. G. FARBENIND. A.-G. (B.P. 308,220, 18.3.29. Ger., 19.3.28. Addn. to B.P. 288,308; B., 1929, 746).—The process of the prior patent is extended to the production of phenols generally, and the yields are improved by addition of copper, nickel, cobalt, or their oxides or nitrates to the silica gel catalyst. Examples are phenol from chlorobenzene and steam over silica gel and copper

oxide at 400°; *p*-cresol from *p*-chlorotoluene over silica gel and nickel at 380°. C. HOLLINS.

Manufacture of condensation products from aromatic aldehydes and phenols [mothproofing agents]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,893—4, 14.3.29).—Hydroxylated di- and triarylmethanes, valuable as mothproofing agents, are obtained by condensation of (A) a non-hydroxylated aromatic aldehyde with 2 mols. of a *p*-alkylated phenol having a free *o*-position (excluding benzaldehyde) with *p*-cresol, or (B) an aldehyde (excepting hydroxylated aromatic aldehydes) with a 3- and/or 5-halogenated phenol having a free 4-position. The condensations are preferably carried out in presence of acid agents (sulphuric acid, hydrochloric-acetic acid, or zinc chloride). The compounds may be applied to wool, fur, etc. together with a dye. Examples are the products from (A) benzaldehyde-*o*-sulphonic acid and 5-chloro-*p*-cresol or *p*-cresol, (B) formaldehyde and *m*-chlorophenol, benzaldehyde-*o*-sulphonic acid and 2:5-dichlorophenol.

C. HOLLINS.

Manufacture of 2-aminonaphthalene-3-carboxylic acid [2:3-aminonaphthoic acid]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,941, 20.3.29).—2:3-Hydroxynaphthoic acid or its zinc salt is mixed with zinc oxide or carbonate and ammonium chloride and then heated at 180—200°, preferably in a current of dry air or ammonia, for 3—4 hrs. in a ball-mill. A 70—80% yield of 2:3-aminonaphthoic acid is claimed.

C. HOLLINS.

Manufacture of oxygenated organic compounds. [Oxidation of aromatic side-chains, naphthalene, etc.] A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,100, 28.6.29).—Aromatic hydrocarbons or their halogen derivatives are oxidised in the liquid phase by oxygen or air under pressure at 150—250° in presence of water and an oxidation catalyst (oxide or hydroxide of copper, nickel, cobalt, iron, manganese, cerium, osmium, uranium, vanadium, particularly ferric oxide hemihydrate). The action may be moderated by addition of considerable amounts of carbon dioxide. Examples are the oxidation of toluene to benzaldehyde and benzoic acid, of *p*-xylene to *p*-toluic acid, of *m*-xylene to *m*-tolualdehyde and *m*-toluic acid, of ethylbenzene to acetophenone and phenylmethylcarbinol, of naphthalene to benzoic acid and phthalic acid, of anthracene to anthraquinone, and of *p*-chlorotoluene to *p*-chlorobenzaldehyde and *p*-chlorobenzoic acid.

C. HOLLINS.

Manufacture of diaminoazoanthraquinones. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,644, 16.3.29).—A 1:4-diaminoanthraquinone is oxidised, *e.g.*, with chromic acid, in an organic acid medium (acetic acid) to give a 4:4'-diamino-1:1'-azoanthraquinone. The product on benzylation and reduction with alkaline hyposulphite yields a 1-amino-4-benzamidoanthraquinone.

C. HOLLINS.

Manufacture of the 6-sulphonc acids of quinizarin and its homologues. I. G. FARBENIND. A.-G. (B.P. 308,359, 21.3.29. Ger., 22.3.28).—The boric ester of quinizarin or a homologue (*e.g.*, 2-methylquinizarin) is sulphonated in the 6-position by heating

with oleum at 170—180°, preferably in presence of a little mercuric sulphate. C. HOLLINS.

Production of hydrogenated phenol compounds. H. JORDAN, ASSR. to CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (U.S.P. 1,771,089, 22.7.30. Appl., 20.3.28. Ger., 14.7.26).—See B.P. 274,439; B., 1929, 164.

Carbazolequinones and process of preparing them. G. KRÄNZLEIN, H. GREUNE, K. ZAHN, and M. P. SCHMIDT, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,772,317, 5.8.30. Appl., 18.1.27. Ger., 15.1.26).—See B.P. 264,530; B., 1928, 635.

Acetylene (B.P. 332,731). **Hydrocarbons** (B.P. 331,558).—See II. **Anthraquinone derivatives** (B.P. 307,306).—See IV. **Activated iron oxide** (B.P. 332,259).—See VII. **Mercury from catalyst sludges** (B.P. 332,106).—See X. **Solvents** (U.S.P. 1,754,417).—See XXII.

IV.—DYESTUFFS.

Fastness of dyestuffs in ultra-violet light. II. G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930; 8, 184—190).—Results are given of further tests (cf. B., 1930, 453) with dyestuffs of the nitroso-, nitro-, azo-, stilbene, pyrazolone, diphenylmethane, and triphenylmethane groups.

T. H. POPE.

Nitrosophenol and indoles. GULINOV.—See III. **Dyeing tannins.** UYEDA.—See XV. **Sensitising dyes.** BLOCH and HAMER.—See XXI.

PATENTS.

Manufacture of dyes [for acetate silk] and application thereof. BRIT. CELANESE, LTD., and H. C. OLPIN (B.P. 330,602, 12.3.29).—*ar*-Tetrahydronaphthylamines are condensed with aromatic nitrocompounds containing reactive halogen, *e.g.*, *ar*-tetrahydro- α -naphthylamine with 1-chloro-2:4-dinitrobenzene or its 6-sulphonic acid, or the β -amine with 1:4-dichloro-2-nitrobenzene, to give yellow dyes for acetate silk. Preferably sulphonic groups are absent.

C. HOLLINS.

Manufacture of acid wool dyes [from nitronaphthalimides]. I. G. FARBENIND. A.-G. (B.P. 309,552, 11.4.29. Ger., 12.4.28. Addn. to B.P. 299,721; B., 1930, 455).—A nitro-1:8-naphthalimide, or its *N*-alkyl or -aralkyl derivative, is boiled with aqueous-alcoholic sodium hydrogen sulphite or hyposulphite, whereby the nitro-group is reduced and a sulphonic group is introduced. 4-Nitro-*N*-methyl-naphthalimide thus yields a yellow wool dye.

C. HOLLINS.

Preparation of [vat] dye derivatives. J. S. WILSON, L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 332,249, 16.1.29).—Halogenated vat dyes (3:3'-dichloro-flavanthrone or -indanthrone etc.) are treated with sulphites to replace halogens with sulphonic acid groups.

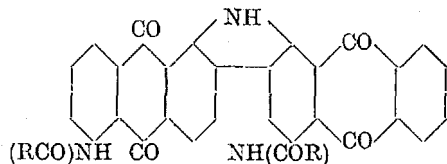
L. J. HOOLEY.

Manufacture of dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,203, 15.4.29).—Aminonitriles of the benzene series are condensed with 4-halogeno-1-aminoanthraquinone-2-sulphonic acids to yield dyes with good solubility and giving even shades. Thus 4-bromo-1-aminoanthra-

quinone-2-sulphonic acid with *m*-aminobenzonitrile gives a reddish-blue, and with 1-amino-4(or 5)-methyl-3-benzonitrile a more greenish dye (cf. B.P. 10,378 of 1914 and 7861 of 1915; B., 1916, 830, 829).

L. J. HOOLEY.

Manufacture of anthraquinone derivatives [carbazoles of the anthraquinone series]. I. G. FARBENIND. A.-G. (B.P. 307,306, 4.3.29. Ger., 3.3.28).—Hydrolysis of 4:5'-diacetamido- or -dibenzamido- $\alpha\alpha'$ -dianthraquinonecarbazole (annexed formula) under mild



conditions, e.g., with 96% sulphuric acid below 60°, or with 10% alcoholic caustic alkali at 110–115°, removes the acyl group from the 5-nitrogen atom only, giving a reddish-brown vat dye.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 307,838, 12.3.29. Ger., 14.3.28).—Carbazoles from α -amino- or $\alpha\alpha'$ -diamino-1:1'-dianthraquinonylamines are condensed with halogenoanthraquinones to give vat dyes. Examples are: 5:5'-diamino- $\alpha\alpha'$ -dianthraquinonecarbazole with 2-bromo-4:3-*p*-tolylaziminoanthraquinone (powerful red); 4:4'-diamino- $\alpha\alpha'$ -dianthraquinonecarbazole with 2-chloroanthraquinone (corinth); 5-amino-4'-benzamido- $\alpha\alpha'$ -dianthraquinonecarbazole (cf. B.P. 307,306, preceding) with 4-chloro-1-benzamidoanthraquinone (black-brown).

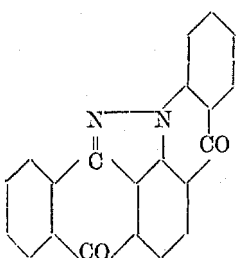
C. HOLLINS.

Manufacture of vat dyes of the anthanthrone series. I. G. FARBENIND. A.-G. (B.P. 307,481, 9.3.29. Ger., 10.3.28).—The halogen in mono-, di-, tri-, or tetra-bromoanthranthrones is replaced by cyano-groups by heating with cuprous cyanide in benzyl cyanide. The product from 4:10-dibromoanthanthrone is a reddish-orange vat dye.

C. HOLLINS.

Manufacture of compounds of the pyrazolanthrone series and vat dyes therefrom. A. CARPMAEL.

From I. G. FARBENIND. A.-G. (B.P. 332,316, 1.5.29).—A 1-halogenoanthraquinone-2-carboxylic acid is condensed with hydrazine, preferably in presence of a



diluent such as pyridine, to yield *pyrazolanthrone-2-carboxylic acid*, m.p. above 300°, which is converted into the acid chloride and condensed with an aromatic hydrocarbon or halogenated hydrocarbon using an acid condensing agent, e.g., thionyl chloride. By treatment with an acid-binding condensation agent these

derivatives yield dyes probably having the annexed constitution and dyeing cotton red shades from the vat. The pyrazolanthronecarboxylic acids or aroylpyrazolanthrones may be alkylated or aralkylated.

L. J. HOOLEY.

Manufacture of pink to red [thioindigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 318,595, 6.9.28. Ger., 6.9.28).—A 6-halogeno-3-oxythionaphthen and a

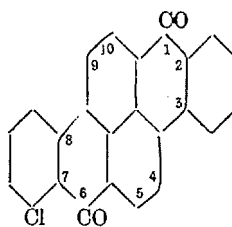
4-methylthionaphthen, halogenated or not in the 6-position (one of these thionaphthens containing a 2-anil group) are condensed to form dyes giving shades faster to light and exposure and nearly as bright as those given by 6:6'-dihalogeno-4:4'-dialkylthioindigo dyes. 6:6'-Dichloro-, 6:6'-dibromo-, and 6'-chloro-4-methyl bithionaphthenindigos are exemplified.

L. J. HOOLEY.

Manufacture of water-soluble leuco-esters of vat dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,579, 11.3.29. Addn. to B.P. 324,119; B., 1930, 364).—The leuco-compounds of vat dyes are esterified with sulphonyl halides of benzoic acid, preferably in presence of a tertiary base. *m*-Chloro-sulphobenzoic acid reacts, e.g., in pyridine, with leuco-indigo, indanthrone, or Caledon-jade-green, to give water-soluble esters which are hydrolysed to vat dyes by heating with alkali.

C. HOLLINS.

Manufacture of vat dyes [from halogenated dibenzpyrenequinones]. I. G. FARBENIND. A.-G. (B.P. 308,617, 23.3.29. Ger., 23.3.28).—Halogenated dibenzpyrenequinones are condensed with suitable amino-compounds (excluding amino-dibenzanthrones and -isodibenzanthrones) in presence of a solvent (nitrobenzene, *o*-cresol), sodium acetate or carbonate, and copper or copper acetate to give vat dyes. The products may be modified by heating with condensing agents. Examples are: bromo-2:3:6:7-dibenzpyrene-1:8-quinone with α -aminoanthraquinone (dull red), heated with sodium aluminium chloride (red-brown); or with anthranilic acid (claret-red), heated with sulphuric acid (brown-orange); dibromo-2:3:7:8-dibenzpyrene-1:8-quinone with α -aminoanthraquinone (violet); or with benzamide (violet); chloro-2:3:7:8-



dibenzpyrene-1:6-quinone (annexed formula) with phthalimide, heated with sulphuric acid (violet); or with benzamide (scarlet); tetrachloro-2:3:7:8-dibenzpyrene-1:6-quinone with β -aminoanthraquinone (brown-black); or with α -aminoanthraquinone (blue-black); or with anthranilic acid (pure green),

heated with sodium aluminium chloride or chlorosulphonic acid (brown).

C. HOLLINS.

Manufacture of monoazo dyes [for wool]. I. G. FARBENIND. A.-G. (B.P. 307,303, 4.3.29. Ger., 2.3.28).—*o*-Toluidines or *o*-anisidines, containing in position 4 or 5 an acylamino-group and in position 5 or 4 an alkoxyl or (in the case of *o*-anisidines) a methyl group, are diazotised and coupled with 1:8-acylaminonaphtholmono- or -di-sulphonic acids to give red-violet to green-blue level wool dyes fast to alkalis and acids. Examples are: 4-amino-2:5-dimethoxyacetanilide \rightarrow acetyl-H-acid (reddish-blue); 3-amino-4:6-dimethoxyacetanilide \rightarrow acetyl-H-acid (violet); 5-acetamido-4-methoxy-*o*-toluidine \rightarrow acetyl-H-acid (reddish-violet); 4-amino-3-methoxy-6-ethoxyacetanilide \rightarrow *p*-toluenesulphonyl-H-acid (bluish-violet). [Stat. ref.]

C. HOLLINS.

[Tris]azo dyes and their application to the dyeing of regenerated cellulose rayon. IMPERIAL

CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 330,607, 13.3.29).—A tetrazotised 3:4'-diaminoazobenzene is coupled with 2 mols. of the same or different coupling components (excepting hydroxynaphthyl aryl ketones and 2:3-hydroxynaphthoic arylamides) chosen so that the resulting dye contains at least two carboxylic or sulphonic groups or one of each. The same dyes may be produced from 3-nitro-4-aminoazobenzenes; they give level shades on viscose silk. Examples are: *m*-nitroaniline → aniline → salicylic acid, reduced, → γ -acid (reddish-brown); *m*-nitroaniline → cresidine → Schäffer acid, reduced, → 2S-acid (blue); 4-nitro-*o*-anisidine → cresidine → N.-W.-acid, reduced, → 2S-acid (greener-blue); 4-nitro-*o*-anisidine or *o*-toluidine → aniline → *o*-cresotic acid, reduced, → phenyl- γ -acid (in alkali; yellowish-brown); *m*-nitroaniline → *o*-toluidine → salicylic acid, reduced, → γ -acid (yellowish-brown); 3:4'-diamino-5'-methoxy-2'-methylazobenzene, m.p. 164–165°, → 1 mol. of γ -acid and 1 mol. of *N*-methyl- β -naphthylamine-7-sulphonic acid or 2R-acid (brownish-black), or → 1 mol. of benzoyl- γ -acid and 1 mol. of M-acid (violet-blue). The last-mentioned diazo component is made by reduction of 3-nitro-4'-amino-5'-methoxy-2'-methylazobenzene, m.p. 163°; 3-nitro-4'-aminoazobenzene, m.p. 208–209°, gives 3:4'-diaminoazobenzene, m.p. 210° (decomp.). C. HOLLINS.

Vat dyes and process of making same. B. MAYER and H. SIEBENBÜRGER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,771,802, 29.7.30. Appl., 23.11.26. Switz., 8.12.25).—See B.P. 262,774; B., 1928, 517.

Manufacture of stable, dry, easily soluble leuco-preparations of vat dyes. E. BAUDER, Assr. to J. R. GEIGY SOC. ANON. (U.S.P. 1,771,763, 29.7.30. Appl., 8.8.27. Ger., 16.8.26).—See B.P. 276,023; B., 1929, 124.

Azo dyes of the pyrazolone series. G. BONHÔTE, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,771,767, 29.7.30. Appl., 20.11.28. Switz., 24.11.27).—See B.P. 301,096; B., 1930, 454.

Diazo-type prints (B.P. 316,563 and 318,108).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Photomicrographs of wool fibres: new method. J. MANBY (J. Text. Inst., 1930, 21, T 231–233).—Mounting media, stains, and methods of illumination are described. A 3% solution of celluloid in amyl acetate, having n_D^{20} 1.4049, is found to be the best medium, whilst celluloid in acetone, and celloidin in a mixture of absolute alcohol and ether, are not so good. Suitable stains are a 5% aqueous solution of acid-fuchsin, and a saturated aqueous solution of picric acid, to both of which 2% of acetic acid is added. B. P. RIDGE.

Bamboo. II. Bamboo-lignin. K. SISIDO (J. Cellulose Inst., Tokyo, 1930, 6, 167–170; cf. B., 1930, 812).—Bamboo is specially suitable for a study of lignin formation in vegetable material because the shoots grow extraordinarily quickly. Shoots 5–8 m. long were divided into three parts, each part being then dried and pulverised, and the dry powder extracted exhaust-

ively with alcohol-benzene and dried *in vacuo*. Lignin was removed with 72% sulphuric acid, and its methoxyl number determined by the Zeisel method. The methoxyl numbers for the samples were also calculated on the assumption that this number represents lignin. Both calculated and observed values show a decrease from the base to the tip of the shoots, but good agreement is not found. It is considered probable that in the samples more remote from the base of the shoot the whole of the methoxyl is not fixed to lignin, and it is concluded that the lignin is methylated gradually according to the growth of the bamboo, or combines with the residue which contains methyl in order to give the higher values determined. Some properties of bamboo-lignin have been compared with those of wood-lignin, and it is found that they behave similarly. Thus carbon, hydrogen, ash, and methoxyl contents are similar in amount to those from spruce-wood lignin. Also both spruce-wood and bamboo chips give a green coloration on immersion in concentrated hydrochloric acid, whilst, on separation, the lignin gives a greyish-violet coloration before, and a salmon-pink after, washing with boiling water. This suggests that bamboo-lignin also has a heterocyclic structure. The distribution of oxygen in the lignin as hydroxyl, methoxyl, etc. is discussed.

B. P. RIDGE.

Suitability of Brazilian woods for pulp manufacture. E. OFFERMANN and G. A. FELDTMANN (Papier-Fabr., 1930, 28, 461–471).—Experimental digestions of 14 Brazilian woods are described. The highest yields were obtained by the sulphite process, but the sulphate process gave more uniform and more readily bleached pulps. Paper-making trials were not practicable owing to lack of material, and the paper-making properties were judged by microscopical methods. Photomicrographs are given. T. T. POTTS.

Wood-pentosan. I. K. NISHIDA and T. TAKAGI (J. Cellulose Inst., Tokyo, 1930, 6, 170–173).—Wood-meals from a number of different coniferous and deciduous trees were extracted with 5% sodium hydroxide solution, the extract was treated with Fehling solution in order to separate pentosan as the copper compound, the latter was decomposed with hydrochloric acid, and the yield and composition of the pentosan were determined. The pentosan content (from the copper compound) of the deciduous woods examined is found to be characteristic, whilst coniferous woods contain much smaller quantities of pentosan. Frequently the wood-pentosan was converted into two different modifications; deciduous woods contain both the α - and β -varieties, whilst only the α -type is present in coniferous wood. The composition of the β -pentosan (% xylan etc.) from the deciduous woods examined is given; that of the α -variety will be given in part II. B. P. RIDGE.

Hygroscopic moisture of cellulose. S. OGURI and M. NARA (J. Soc. Chem. Ind., Japan, 1930, 33, 267–270 B).—The water-absorption time curves have been determined for wood, bamboo, and cotton celluloses and for nitrocellulose, exposed to saturated water vapour at various temperatures. The equilibrium (maximum) moisture content depends on the origin of the cellulose, and decreases as the temperature rises; e.g., the

moisture content of standard cotton cellulose is 16.8% and 15.2% (dry basis) at 20° and 30°, respectively, which is less than that of bamboo or wood cellulose, and greater than that of nitrocellulose. The absorption is very slow.

E. LEWKOWITSCH.

Viscose. XXXIV. Effect of chlorination on the properties of cellulose. S. MASUDA and J. MURAKAMI (J. Cellulose Inst., Tokyo, 1930, 6, 173—174).—Previous work by the authors has shown that the properties of sulphite-pulp are improved by chlorination of the material at 15°, and similar results are now found for cotton. The copper number of the purified cotton cellulose used was reduced from 5 to 4 as a result of chlorination for 15 min. at 24° and washing with water, under standard conditions, and to 3 by similar chlorination for 30 min.

B. P. RIDGE.

Imbibition phenomena and viscose silk. A. WEHRUNG (Cellulosechem., 1930, 11, 170—173).—The difference in behaviour of viscose yarn obtained by variations of procedure is discussed from a theoretical point of view.

T. H. MORTON.

Beating of [paper] pulp. XII. Improvement of absorbency of blotting paper. M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 164—167; cf. B., 1930, 813).—Blotting papers of high absorbency are obtained by the use of pulps treated with concentrated sodium hydroxide solutions, and the absorbing velocity of such products is, on the average, about 40% higher than that of paper made from untreated pulp. Hence pulps can be transformed into "freer" states than their natural ones, and this effect is the reverse of that obtained by beating.

B. P. RIDGE.

Determination of moisture in pulp and paper. K. G. JONAS (Papier-Fabr., 1930, 28, Fest- u. Auslandsheft, 111—112).—Drying in a stream of dry air at 65° is preferred to oven-drying and distillation methods. Carbon tetrachloride used in distillation methods is held to vitiate results owing to the formation of hydrochloric acid.

T. T. PORTS.

Examination of half-stuffs and paper with the quartz lamp. H. G. KLEIN (Papier-Fabr., 1930, 28, 477—480).—The influence of moisture content, action of light, and chemical treatment on the fluorescence of half-stuffs and papers has been investigated, and tables showing the fluorescence colours of a number of samples are given. Slight changes in moisture content do not influence fluorescence greatly, whereas short exposures to light and heat have appreciable effect. The effect of chemical treatment on fluorescence is held to afford a means of distinguishing pulps prepared by the various processes.

T. T. PORTS.

Use of dextrose in the manufacture of imitation parchment papers. H. WREDE (Papier-Fabr., 1930, 28, Fest- u. Auslandsheft, 118—119).—The addition of 3% of dextrose to the beater furnish is claimed to improve manufacturing conditions and the quality of the finished sheet. The pulp hydrates more rapidly, less power is required for beating, greater transparency and gloss are obtained, and the sheet lies flat. The pliability of papers may be increased by spraying with dextrose solutions before calendering.

T. T. PORTS.

Oxide inclusions as cause of working difficulties in copper half-stuffs. R. W. SCHULZE (Dingler's Polytech. J., 1929, 344, 233—236; Chem. Zentr., 1930, i, 1209).

Sinkage [of pulp-wood]. SCARTH and JAHN.—See IX.

PATENTS.

Removing marks on wool. G. J. M. and R. H. M. STEINS (U.S.P. 1,754,338, 15.4.30. Appl., 13.8.27. Belg., 18.8.26).—Wool is soaked at 30—70° in heavy tar oils (b.p. above 100°) and then extracted with a mixture of benzine and light petroleum.

F. R. ENNOS.

Spools for textile industries. I. G. FARBENIND. A.-G. (B.P. 311,287, 8.5.29. Ger., 8.5.28).—Metal or other spools are coated with cellulose esters which are resistant to acids and alkalis. The coating consists of one or more cellulose derivatives of fatty acids containing more than 5 carbon atoms, and is applied direct or over a coating of natural or artificial resin.

B. P. RIDGE.

Manufacture of a cork product. C. TECCA and C. R. DEL TURCO (B.P. 312,672, 30.5.29. Ital., 30.5.28).—Ground cork is mixed with 20—30 wt.-% of alkali in aqueous solution, and after 10—15 hrs. a further quantity of cork sufficient to neutralise the excess of alkali is added, together with fillers, colouring matter, etc. The plastic mass is moulded, stamped, or pressed and hardened by evaporation of the water.

F. R. ENNOS.

Production from cellulose hydrates of bodies such as capsules, foils, tubes, etc. which are capable of reversibly swelling. WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 316,580, 29.7.29. Ger., 31.7.28).—Such articles, made from regenerated cellulose, are treated with a sucrose solution either before drying or after pre-drying until shrinking commences.

B. P. RIDGE.

Manufacture of cellulose hydrate films. KALLE & Co. A.-G. (B.P. 312,076, 16.5.29. Ger., 19.5.28).—Cellulose hydrate films are freely suspended in moist air so that they absorb water, and are afterwards dried by suspending in air under normal atmospheric conditions, whereby their internal stresses become compensated whilst the material remains smooth.

F. R. ENNOS.

Manufacture of viscose. F. STEIMMIG, and ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (B.P. 332,628, 28.2.29).—Organic substances such as multivalent alcohols, sugars, etc., which serve to coagulate viscose and diminish the oxidation of the hydrogen sulphide produced, and sulphites, amides, or imides, which retard ripening, or arsenites, which do not promote ripening, are added to the viscose before spinning. [Stat. ref.]

B. P. RIDGE.

Manufacture of fibres, ribbons, films, or the like from viscose. I. G. FARBENIND. A.-G. (B.P. 309,147, 5.4.29. Ger., 5.4.28. Addn. to B.P. 306,971; B., 1930, 74).—An alkali salt of an organic oxy-acid (citric, tartaric, lactic), instead of oxalic acid, is added to the viscose, which is precipitated by means of a solution of sulphuric acid and sodium sulphate.

F. R. ENNOS.

Manufacture of artificial silk. L. M. V. GROS-LAFOND, V. M. L. LAFOND, E. M. V. LAFOND-PANSU, and P. BANZET (B.P. 308,267, 19.3.29. Fr., 20.3.28).—Artificial silk threads are dry-stretched on bobbins to an extent sufficient to ensure that they pass beyond their so-called "dead-zone," in which they have no elasticity. [Stat. ref.] B. P. RIDGE.

Replacing the water in a water-moist cellulose by an organic liquid. E. H. A. RÖST-GRANDE and E. CORRENS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,752,596, 1.4.30. Appl., 16.3.27. Ger., 17.3.26).—A water-miscible organic liquid (glacial acetic acid) is forced through a cake of moist cellulose; the acetic acid may then be expelled by means of another organic liquid (benzene) under pressure, which is miscible with the glacial acetic acid but immiscible with water.

F. R. ENNOS.

Manufacture of artificial yarns, threads, or filaments. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,527, 21.3.29).—The residual solvent contained in the artificial yarns is extracted by subjecting the material immediately after spinning to the action of a vacuum at 60–70°, the solvent so removed being recovered in a suitable plant. The material is subsequently conditioned while still in the vacuum chamber by the admission of a humidified atmosphere.

B. P. RIDGE.

Production of cellulose acetate. A. RUSSELL and J. F. HEGEMAN (U.S.P. 1,752,853, 1.4.30. Appl., 15.2.29).—Cellulose is acetylated with acetic anhydride, acetic acid, and sulphuric acid under a pressure of 50–75 lb./in.², the temperature being gradually raised from about –5° to 40°, and the subsequent hydration is effected by adding sufficient water and acid at 100–200 lb./in.², while maintaining the temperature. F. R. ENNOS.

Preparation of cellulose acetate. RUTH-ALDO Co., Assees. of H. L. BARTHELEMY (B.P. 305,096, 28.1.29. U.S., 30.1.28).—Cellulosic material is submitted first to direct oxidation, *e.g.*, by alkali or hydrogen peroxides, persalts, in the presence of moderating agents, *e.g.*, alkali carbonates, silicates, soaps, or sulphuric acid, and afterwards to a softening treatment with at least 50 wt.-% of hot vapours of acetic acid containing a small amount of halogen. The product is then acetylated in at least four stages by additions of acetic acid, acetic anhydride, and sulphuric acid so that in the earlier stages (up to formation of cellulose acetate of acetyl value about 25%), during which a small quantity of halogen or oxidising agent is added to reduce the amount of sulphuric acid required, only slight rises of temperature occur (up to 18–26°), whilst in the later stages the temperature is allowed to rise to 55–65°. The proportion of catalyst (sulphuric acid) in the successive baths is increased progressively, whilst that of the acetic anhydride is reduced until the final stage when the bulk is added. Finally, the acetic ester is partly saponified and the sulphuric esters are decomposed by adding aqueous formic or acetic acid, first alone and later with addition of hydrochloric and hydrofluoric acid equivalent to the quantity of sulphuric acid used as catalyst, the amount of water in the final mixture being adjusted to 5–15% of the total organic acids present.

F. R. ENNOS.

Cellulose acetate compositions. E. I. DU PONT DE NEMOURS & Co. (B.P. 306,911, 27.2.29. U.S., 27.2.28).—Carboxylic esters of a phenoxyethyl alcohol, *e.g.*, β -phenoxyethyl phthalate (or laurate) or its methyl derivative, are added as softeners or plasticisers for cellulose acetate. The products are suitable for the manufacture of lacquers, leather-cloth, etc.

B. P. RIDGE.

Treatment of cellulosic materials and production of cellulose esters therefrom. BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 332,607–8, 17.4.29).—Purified cellulosic materials are more readily esterified if they are treated (A) first with organic anhydrides (acetic anhydride), either alone or mixed with a liquid hydrocarbon, or in vapour form alone or mixed with an inert diluent, and then with an organic acid with or without anhydride (*e.g.*, acetic acid and acetic anhydride), or (B) first with one or more organic acids (*e.g.*, formic or acetic), alone or mixed with inert diluents, and then with one or more organic anhydrides, without or with organic acids, a condensing agent being present in each case, if desired.

B. P. RIDGE.

Manufacture of cellulose ethers. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 306,125, 21.1.29. Ger., 17.2.28).—Cellulose ethers, insoluble in pure solvents but soluble in mixed solvents, such as benzene-alcohol or dichloroethylene-alcohol, are produced by treatment of alkali-cellulose with alkyl halide in the presence of water but without further addition of caustic soda.

B. P. RIDGE.

Manufacture of cellulose ether esters. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,903, 28.3.29).—A cellulose ether containing about $\frac{1}{2}$ to $1\frac{1}{2}$ alkyl groups for each $C_6H_{10}O_5$ molecule, including 0.5–2% of benzyl or substituted benzyl cellulose, is acetylated by means of acetic anhydride in the presence of acetic acid as solvent and a small quantity of a catalyst (0.1–3% of sulphuric acid, or 10–30% of zinc chloride, calc. on the cellulose ether).

F. R. ENNOS.

Manufacture of soluble cellulose esters of higher organic acids. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 330,575, 11.3.29).—Cellulose is esterified by treatment with anhydrides of acids above C_7 , preferably in presence of an aliphatic hydrocarbon sulphonic acid (methanesulphonic acid) and a solvent (carbon tetrachloride, toluene) at moderate temperatures (15–65°). Esterification is facilitated by a pretreatment, *e.g.*, with dichlorohydrin at 90° for 16 hrs., with chloroacetic acid at 80–90°, or with vapours of the appropriate acid. The acid anhydrides exemplified are lauric, palmitic, stearic, and phenyl-acetic; the esters obtained soften or melt at 65–135° without decomposition.

C. HOLLINS.

Manufacture of shaped objects from cellulose derivatives. I. G. FARBENIND. A.-G. (B.P. 315,275, 10.7.29. Ger., 10.7.28).—Cellulose derivatives of naphthenic acid, or of saturated or unsaturated higher fatty acids containing more than 8 carbon atoms, are rolled or calendered at a raised temperature before being shaped. This treatment increases the elasticity of the material and permits of shaping and bending without loss of its pliancy.

B. P. RIDGE.

Manufacture of paper. KODAK, LTD., Assees. of G. T. LANE (B.P. 332,743, 22.7.29. U.S., 1.3.29).—Size consisting of a saponified saturated fatty acid of high mol. wt. (*e.g.*, stearic or palmitic acid) and having an iodine value below 40 (preferably below 5), is added to the paper pulp in the beater and precipitated on the fibres by the addition of aluminium or magnesium sulphate, or of potassium or chrome alum. Paper so produced causes no appreciable fogging of a photosensitive emulsion kept in contact with it, and has improved strength and flexibility, a smooth surface, and high resistance to chemicals, moisture, oils, heat, and light.

B. P. RIDGE.

Cellulose solutions. L. LILIENFELD (U.S.P. 1,771,460—2, 29.7.30. Appl., 10.5.24. Austr., 5.6., 25.5., and 5.6.23).—See B.P. 217,166 and 216,475; B., 1924, 977; 1925, 37.

Isolation of benzylcellulose. E. DORR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,771,529, 29.7.30. Appl., 20.9.26. Ger., 17.8.25).—See B.P. 265,491; B., 1927, 296.

Spinning boxes for use in the manufacture of artificial silk etc. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (B.P. 310,009, 19.4.29. Fr., 19.4.28).

Treating and utilising the black liquor obtained in the soda-pulp process. E. HÄGGLUND (U.S.P. 1,772,216, 5.8.30. Appl., 13.7.25. Ger., 16.7.24).—See B.P. 258,035; B., 1927, 165.

Centrifugal machines (B.P. 332,288). **Loop dryer for fabrics** (U.S.P. 1,755,005—6).—See I. RUBERISED FABRIC (B.P. 331,594).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Influence of p_H on dyeing phenomena. I. Uneven dyeing of viscose. II. Influence of p_H , salt, and variations of temperature in the dyeing of viscose with direct dyes. III. Dyeing viscose, cotton, and mercerised cotton in the same bath. IV. Rinsing of dyed cotton. V. Light-fastness of dyed cotton. C. E. MULLIN (Tiba, 1929, 7, 853—867).—The necessity for control of the p_H in dyeing and rinsing is exemplified, and the effect of addition of sodium chloride and of temperature on shade is discussed.

CHEMICAL ABSTRACTS.

PATENTS.

Rapid bleaching of vegetable fibres. C. G. SCHWALBE and H. WENZL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,909, 15.4.30. Appl., 19.1.25. Ger., 15.7.21).—The fibres are rapidly bleached by impregnation with a hypochlorite solution containing 10—30 g. of active chlorine per litre instead of the usual 1 g. per litre, then pressed to remove excess liquor, and shortly afterwards washed; the material may be steamed before, during, or after bleaching.

A. J. HALL.

Bleaching and stripping dyes from textile fibres and materials. BRIT. RES. ASSOC. FOR THE WOOLLEN & WORSTED INDUSTRIES, and A. T. KING

(B.P. 332,389, 6.7.29).—Wool materials are treated with a cold or warm solution containing mixtures of sodium bisulphite and sulphite such that the mol. ratio $SO_2/NaOH=1/1.25-1.35$ (substantially $2NaHSO_3, Na_2SO_3$); stripping is particularly efficient with azo dyes having an α -naphthol or substituted α -naphthol component. Examples of dyes not stripped by acidified bisulphite, but stripped by this new process, are Carmoisines WS and L9156K, Benzyl Bordeaux B, and Coomassie Scarlet 9012K.

A. J. HALL.

Bleaching of fabrics in open width. F. E. and P. V. BARKER (B.P. 332,718, 3.7.29).—Fabric is suspended in open width in festoons (looped form) on a wagon which can be successively run into kiers and washing chambers where the fabric is subjected first to showers of boiling alkali-liquor, and then to washing, chemicking, souring, and washing liquors.

A. J. HALL.

[Preparation of] non-aqueous dye-bath. E. G. SCHLATTER, Assr. to DIP-IT, INC. (U.S.P. 1,754,285, 15.4.30. Appl., 13.3.26).—Solutions of acid, basic, or direct dyes in glycerin are diluted with an organic liquid (*e.g.*, an aliphatic alcohol or acetone) which is a solvent for glycerin but not for the dye.

A. J. HALL.

Immunising textiles to direct-dyeing dyestuffs. SOC. CHEM. IND. IN BASLE (B.P. 315,434, 13.7.29. Switz., 13.7.28).—Vegetable fibres are immunised by esterification with maleic anhydride without a catalyst.

A. J. HALL.

Dyeing shearlings [wool skins] and the like. J. G. SHOEMAKER, Assr. to A. C. LAWRENCE LEATHER CO. (U.S.P. 1,754,287, 15.4.30. Appl., 4.4.28).—Wool skins or furs are given a resist of molten tallow or wax (preferably a tallow of m.p. 43° is applied at 49°), mordanted in the usual manner at a moderate temperature, dyed, and washed.

A. J. HALL.

Vat dyeing. A. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,563, 7.3.29).—Vat dyes may be vatted by titanous compounds in alkaline medium. Preferably a complex salt, formed, *e.g.*, by addition of sodium formate, lactate, or tartrate to titanous chloride solution, is used, or the alkali and the titanous salt are mixed in presence of glue or other protective colloid.

C. HOLLINS.

Colouring of wool and other animal fibres [with vat dye sulphonic acids]. J. S. WILSON, L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 332,232, 16.1.29).—Sulphonic acids of vat dyes (indanthrone, Caledon Red BN, Caledon Brilliant Purple RR) are dyed on wool or other animal fibres by a vatting process as distinct from an acid process. The increased solubility given by the acid group appears to aid the dyeing of animal fibres from the vat. The sulphonic acids may be prepared in stable soluble leuco-form.

L. J. HOOLEY.

Manufacture of evenly-dyed viscose fabrics. I. G. FARBENIND. A.-G. (B.P. 330,592, 12.3.29. Addn. to B.P. 306,908; B., 1930, 859).—Viscose silk is dyed evenly with the dyes: 5-nitro-*o*-aminophenol \rightarrow β -naphthylamine-5-sulphonic acid, prechromed (green);

4-chloro-*o*-aminophenol-6-sulphonic acid \rightarrow 1-*m*-sulphophenyl-3-methylpyrazolone, prechromed (pink); 1 : 2 : 4-aminonaphtholsulphonic acid \rightarrow α -naphthol-8-sulphonic acid, prechromed (blue); anthranilic acid \rightarrow β -naphthylamine-7-sulphonic acid, prechromed (violet); 5-nitro-*o*-aminophenol \rightarrow *p*-anisyl-J-acid, zinc compound (violet); 5-nitro-*o*-aminophenol \rightarrow *p*-phenetyl-J-acid, copper compound (reddish-blue). C. HOLLINS.

Coloration of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., and H. C. OLFIN (B.P. 330,591, 12.3.29).—Acetate silk is dyed with azo dyes containing an *ar*-tetrahydronaphthyl group, preferably having no sulphonic group. Examples are: *ar*-tetrahydro- β -naphthylamine \rightarrow phenol (yellow), or 2 : 3-hydroxynaphthoic *m*-nitroanilide (orange); *p*-nitroaniline \rightarrow *ar*-tetrahydro- α - or - β -naphthylamine (orange or golden-yellow), *m*-nitroaniline-*ar*-tetrahydro- α -naphthylamine (marigold); *p*-nitroaniline-*o*-sulphonic acid \rightarrow *ar*-tetrahydro- α -naphthylamine (orange-red). C. HOLLINS.

[Photochemical] dyeing and figuring of woven, knitted, and plaited fabrics, leather, and other fibrous material. W. HAENDEL (B.P. 309,166, 8.3.29. Ger., 7.4.28).—Light-sensitive diazosulphonic acids are applied locally to the material, *e.g.*, by printing, with or without simultaneous application of the coupling components, and the material is then exposed to light and the colour developed. A series of different colours may be successively printed, each being developed before the next printing. The diazosulphonates are made by addition of sodium sulphite to the diazonium chlorides from aniline (with phenol for yellow), benzidine (with resorcinol for red), tolidine (with 2S-acid and β -naphthylamine for blue). The depth of shade may be varied by varying the concentrations or the length of exposure to light. C. HOLLINS.

Manufacture of azo dyes on the fibre [stable diazo preparations]. I. G. FARBENIND. A.-G. (B.P. 306,844, 25.2.29. Ger., 25.2.28).—The compounds of diazo- or tetrazo-compounds with secondary amines (piperidine, dialkylamines) prepared by the method of Baeyer and Jäger (A., 1875, i, 273) or Wallach (A., 1887, 137) are stable in aqueous solution even at 100°, but yield diazo solutions when treated with cold dilute acid. They are used for development of ice-colours; *e.g.*, cotton padded with 2 : 3-hydroxynaphthoic α -naphthylamide is treated with an acidified solution of the piperidine compound of diazotised *m*-nitro-*o*-toluidine (for red) or of tetrazotised dianisidine (for blue). C. HOLLINS.

Manufacture of an azo dye on the fibre [ice colour]. I. G. FARBENIND. A.-G. (B.P. 307,704, 11.3.29. Ger., 10.3.28).—2 : 3-Hydroxynaphthoic β -naphthylamide is coupled on the fibre with diazotised 2 : 4-dichloro-6-amino-*m*-cresol methyl ether to give a bluish-red fast to light and to kier-boiling with 0.4% sodium hydroxide solution. C. HOLLINS.

Production of azo dyes on the fibre [ice colours]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 331,056, 23.5.29. Cf. B.P. 286,274; B., 1929, 675).—

In the production of blue ice-colours from 4-amino-diphenylamines and 2 : 3-hydroxynaphthoic arylamides, the diazo solution is mixed with excess of (acetic) acid and after development the goods are passed through an alkaline bath (hot aqueous sodium carbonate).

C. HOLLINS.

Dyeing and printing textile goods etc. [with solubilised vat dyes]. DURAND & HUGUENIN SOC. ANON. (B.P. 310,478, 26.4.29. Ger., 26.4.28).—The development of the shade by oxidation of solubilised vat dyes (B.P. 220,964; B., 1925, 879) printed on textiles is effected by drying in presence of a copper salt, either before or at the time of printing.

A. J. HALL.

Dyeing and printing with [leuco-ester] vat dyes. DURAND & HUGUENIN SOC. ANON. (B.P. 319,021, 14.9.29. Ger., 15.9.28).—Development of leuco-ester salts of vat dyes is effected with iron alum, particularly ammonium iron alum. These developers are suitable for all esters on animal fibres and for esters having sufficient affinity on vegetable fibres. They avoid over-oxidation with dyes such as Indanthrene Blue RS, and as they can be used without addition of acid, less water is required in rinsing.

L. J. HOOLEY.

Production of fast dyeings and prints [with indigosols etc.]. DURAND & HUGUENIN SOC. ANON., Assees. of J. C. LIVINGSTON (B.P. 314,350, 25.6.29. Ger., 25.6.28).—The acid oxidising conditions necessary for developing leuco-ester salts of vat dyes are produced by electrolysis (cf. B.P. 203,681; B., 1924, 982).

L. J. HOOLEY.

Discharge printing. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,646, 7.3.29. Cf. B.P. 330,563; B., 1930, 901).—A titanous salt, preferably a complex salt such as may be obtained by addition of sodium formate, tartrate, or lactate to aqueous titanous chloride, or, if a simple salt be used, with the addition of a protective colloid such as sulphite-cellulose liquor, is employed as reducing agent in alkaline discharge pastes, especially for vat dyes. The presence of iron is not objectionable.

C. HOLLINS.

Discharge printing on wool. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,202, 15.4.29).—Wool is dyed with a monoazo dye obtained by coupling diazotised *p*-nitroaniline-*O*-sulphonic acid with a β -naphthylaminesulphonic acid or *N*-alkyl derivative thereof, *e.g.*, β -methylaminonaphthalene-7-sulphonic acid, and discharge-printed in the usual way.

L. J. HOOLEY.

Production of printed [cellulose ester] fabrics. BRIT. CELANESE, LTD. (B.P. 317,455, 16.8.29. U.S., 16.8.28).—Fabrics with warp print effects are obtained by weaving a warp or weft of a treated (*e.g.*, hydrolysed) cellulose ester with an untreated ester and then printing a dye with an affinity for only one of the esters.

L. J. HOOLEY.

Apparatus for mercerising textile fabrics. W. MYCOCK & Co., LTD., and W. MYCOCK (B.P. 332,751, 31.7.29).

Dyeing of acetate silk (B.P. 330,602). **Trisazo dyes on rayon** (B.P. 330,607).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Chamber [sulphuric acid] process. XX. Empirical formula and nomogram for composition of nitrous vitriol. M. MATSUI (J. Soc. Chem. Ind., Japan, 1930, 33, 248—251b; cf. *ibid.*, 1925, 28, 165 b).—If H is the percentage sulphuric acid content, N the content of sodium nitrate in g. per litre, and p and T are the nitrous acid tension and temperature, respectively, then $H = 5.26 \log N - 5 \log p - 10,105/T + 96.710$. A nomogram is constructed for the solution of this equation within the limits for H , N , and T met with in practice.

C. IRWIN.

Manufacture of synthetic nitric acid. V. I. MAL-YAREVSKI and V. V. PAPKOV (J. Chem. Ind., Russia, 1929, 6, 849—853).—The operation of the Libinon process under 10—30 atm. pressure is advocated.

CHEMICAL ABSTRACTS.

Extraction of phosphoric acid from low-grade phosphorites with a mixture of sulphuric acid and ammonium sulphate. S. I. VOLFKOVICH and L. E. BERLIN (Min. Syr. Tzvet. Met., 1929, 4, 396—407).—The optimal mixture contains 16.6—22.2% of ammonium sulphate, which diminishes the amount of lower oxides extracted.

CHEMICAL ABSTRACTS.

Change of water-soluble phosphoric acid content of superphosphate during storage in bulk. Effect of temperature. T. SHOJI, E. SUZUKI and S. HIRABAYASHI (J. Soc. Chem. Ind., Japan, 1930, 33, 204—206 b).—Superphosphate made from Christmas Island rock showed no loss in water-soluble phosphate when stored in bulk in winter when the temperature in the interior of the heap was always below 40°. In summer pyrometers in different positions showed temperatures between 50° and 60°, and it was found that while material on the surface was unchanged, loss of water-soluble phosphate had occurred in the interior in proportion to the temperature reached. Laboratory experiments showed that a slow degradation also occurs at or below 40°.

C. IRWIN.

Economics and developments of ammonia production. E. A. THAU (Gas- u. Wasserfach, 1930, 73, 767—775).—A review of the present position concerning by-product and synthetic ammonia, obtained by a variety of methods. Attention is called to the influence of the cost of sulphuric acid on the ammonium sulphate process.

R. H. GRIFFITH.

Special hydrometer for aqua ammonia. D. F. FARRAR (Ind. Eng. Chem. [Anal.], 1930, 2, 293).—A hydrometer graduated in percentages of ammonia, ranging from 18 to 28%, with attached thermometer and a chart for temperature corrections has been constructed.

E. S. HEDGES.

Manufacture of sodium hydroxide from sodium sulphate. V. I. KURIKOV (J. Chem. Ind., Russia, 1929, 6, 119—120).—A mixture of wood charcoal and barium sulphate is calcined at 1000—1200°, the barium sulphide treated with steam at a temperature above 100°, and the barium hydroxide solution added to boiling sodium sulphate solution.

CHEMICAL ABSTRACTS.

Pyrogenic decomposition of gypsum. K. I. LOSEV and S. N. NIKITIN (J. Chem. Ind., Russia, 1929,

6, 169—173).—So-called fusion of gypsum is an optical illusion. Decomposition is noticeable above 1000° and maximal at 1420°; the decomposition temperature may be lowered by addition of silica, alumina, or ferric oxide. Reduction by charcoal is best effected at 1000°.

CHEMICAL ABSTRACTS.

Manufacture of bleaching powder. M. N. GURVICH, G. A. DMITRIEV, B. A. SASS-TISOVSKI, and D. V. STEPANOV (J. Chem. Ind., Russia, 1929, 6, 853—858).—The lime was calcined at 1000—1100° and slaked with 3 pts. of water; at the optimal rate of passage of chlorine into milk of lime an easily filterable paste is obtained. The temperature of the external bath should be 14°. The filtered, pressed, and dried precipitate contains 52—55%, and the filtrate 6—7%, of active chlorine.

CHEMICAL ABSTRACTS.

Identification of barytes, quartz, and silicates. H. WAGNER (Farben-Ztg., 1930, 35, 2174—2175).—The barium flame test as a means of identifying barytes is not satisfactory. Barytes can be distinguished from gypsum, but not from quartz, by its appearance in polarised light. Adsorption of dyestuff (brilliant-green) is not a successful means of identification. The immersion of the specimen in a refractive medium and examination under a low microscopical magnification is advocated. With phenylthiocarbimide as the medium barytes appears blue and can be clearly identified, although the method may fail under some special conditions.

J. O. CUTTER.

Manufacture of ferric chloride. I. E. ADADUROV (J. Chem. Ind., Russia, 1929, 6, 203—204).—A mixture of iron filings (20 pts.) and charcoal (1 pt.) is heated in an iron pipe in a current of chlorine containing a little air. The yield of ferric chloride is 83—92%. Pyrite residues from the manufacture of sulphuric acid may be used (yield 85—92% of the theoretical), provided that the sulphur content does not exceed 1.5%.

CHEMICAL ABSTRACTS.

Process of decomposing chromite. Y. KATO and R. IKENO (J. Soc. Chem. Ind., Japan, 1930, 33, 225—226 b).—Methods which have been proposed for the extraction of chromic oxide from chromite are reviewed.

C. IRWIN.

Preparation of pure chromic anhydride. V. V. POLYANSKI (Trans. Inst. Pure Chem. Reagents, Sci. Tech. Dept. U.S.S.R., 1929, No. 300, 143—154).—Calcium chromate is prepared by addition of calcium hydroxide paste, followed by calcium chloride solution, to aqueous sodium dichromate; the mixture is concentrated on the water-bath, the precipitate of calcium chromate being collected and dried. A solution in water (2 pts.) is treated with sulphuric acid (d 1.84, 1 pt.), kept on the water-bath, decanted, and this procedure repeated until no more calcium sulphate is precipitated, when chromic anhydride is crystallised. The product is treated with nitric acid, d 1.4, and dried at 60—100°.

CHEMICAL ABSTRACTS.

Utilisation of the gases produced by distillation of phosphorus in the blast furnace. E. V. BRITZKE, N. E. PESTOV, and N. N. POSTRIKOV (Min. Syrje Zwet. Met., 1929, 4, 375—387; Chem. Zentr., 1930, i, 1020—1022).—The difficulties encountered in Liljenroth's process, and due to the poisoning of the metallic catalysts

by phosphorus, phosphine, etc., are avoided by using phosphorite, limestone, sylvine, and other natural rocks as catalysts, the phosphoric acid formed by the action of water vapour being absorbed at high temperatures. Phosphite is not produced. Experiments in which a mixture of carbon dioxide and water vapour was passed through a vessel containing molten phosphorus and then through a heated porcelain tube containing the catalyst are described; phosphoric acid fog was precipitated electrostatically. In the absence of a catalyst and at 800° phosphoric, phosphorous, and hypophosphorous acids were obtained, the relative proportions depending on the experimental conditions, together with hydrogen and phosphine. Phosphoric acid free from phosphorous and hypophosphorous acids is formed only in presence of catalysts at about 600°. When the ratio of phosphorus to water is 1:150 the hydrogen contains no phosphine. Oxidation of phosphorus by carbon dioxide proceeds analogously. The gaseous products always contain carbon monoxide and dioxide, even when excess of phosphorus is employed; the solid products consist of oxides of phosphorus and (with deficiency of carbon dioxide) elementary phosphorus. At normal pressure the reaction begins at 800°. For ratios $P : CO_2 = 1 : 22-15$ the chief reaction is $P_4 + 8CO_2 = P_4O_{10} + 8CO$. Phosphine is oxidised by water vapour or carbon dioxide in an analogous manner. When a copper-nickel-pumice (Liljenroth) catalyst was used at 600° the acids contained H_3PO_4 98.75, H_3PO_3 0.91, H_3PO_2 0.34%; with active carbon (Urbain) reaction commenced at 400°. The use of phosphorite (composition recorded) in granular form leads by absorption of phosphoric acid to the production of a compact mass; chalk does not suffer from this disadvantage. The limiting velocity to avoid the collection of phosphorus and phosphine is the greater the smaller are the granules, and falls as the carbonate is converted into metaphosphate. At high temperatures chalk and limestone, at low temperatures active carbon and copper-nickel-pumice, give the best results. In presence of manganese dioxide the activity of the chalk catalyst at 500–600°, but not at higher temperatures, was somewhat increased.

[With M. N. BOLOTIN.] The calcium metaphosphate was converted, in the same apparatus, into $CaH_4(PO_4)_2$ by treatment with orthophosphoric acid and steam.

A. A. ELDRIDGE.

Petroleum method of iodine extraction. O. Y. MAGIDSON (J. Chem. Ind., Russia, 1929, 6, 699–702).—Polemical against Panteleimonov (B., 1929, 812). The presence of nitrites or of considerable acidity does not permit the direct titration of iodine with thiosulphate.

CHEMICAL ABSTRACTS.

Soluble copper from ores. SULLIVAN and SWEET; GUGGENHEIM and SULLIVAN.—See X. **Analysis of red lead.** AMPT.—See XIII. **Phosphate fertilisers.** VOLFKOVICH and KAMZOLKIN. **Natural aluminium phosphate.** KIDA.—See XVI.

PATENTS.

[Catalyst for] oxidation of ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,728, 17.8.29).—The catalyst comprises platinum-gauze netting

which has been coated with finely-divided rhodium by spraying it with an alcoholic solution of rhodium trichloride and heating in a hydrogen flame or by electro-deposition from a 1% solution of sodium chlororhodite.

A. R. POWELL.

Regeneration of carbonated ammoniacal solutions. SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.), ASSRS. to L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 306,947, 14.2.29. Fr., 29.2.28).—The solution is preheated in a heat exchanger by means of regenerated solution to about 65°, at which temperature it does not dissociate. It then passes down a plate column in countercurrent to vapours from solution undergoing dissociation and becomes rectified, while at the same time it is heated by regenerated solution in the bottoms of the plates. From the bottom of the column it then passes to a compartment where it is heated by a steam coil, and thence is pumped into the bottoms of the plates in an opposite direction to that of the ascending solution. It is thus again brought to 65°, and passes from the top of the tower to the heat exchanger, where, in its turn, it serves to preheat entering solutions to be regenerated.

W. J. WRIGHT.

Kilns or furnaces for calcining, burning, or roasting limestone, iron ore, chalk, etc. C. F. PRIEST (B.P. 332,037, 25.6.29).—In vertical kilns or furnaces the burners are cooled by water-jackets or other means, and project transversely through the wall into the interior, the orifices in the burners being directed vertically downwards. The combustible gases are thus brought into intimate contact with the whole of the material in the kiln.

W. J. WRIGHT.

Working-up of natural and industrial salt mixtures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,236, 25.3.29).—Potassium chloride or nitrate or sodium nitrate may be extracted from admixture with other salts, e.g., sodium chloride, by treatment of the mixture with liquid ammonia. A. R. POWELL.

Production of barium monoxide. G. T. SHINE (B.P. 331,878, 11.2.29).—Reduction of barium carbonate is effected continuously in a series of chambers of refractory material, placed in an elongated retort setting having a central main flue extending through the upper portion. This flue communicates with inlet ducts at each end, through which producer gas is admitted, and with a central exit for exhaust gases. The chambers are charged at the top and have movable bases for discharging, the charging apertures opening into an air-duct of heat-insulating material through which a current of cold air is caused to pass during the charging of the chambers. By this means gases are carried off, and continuous charging can be effected. W. J. WRIGHT.

Manufacture of calcium hypochlorite. R. B. MACMULLIN and J. A. GUYER, ASSRS. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,754,473, 15.4.30. Appl., 4.8.26).—Calcium hypochlorite, formed by chlorination of a lime slurry, is salted out by the addition of sodium chloride. The hypochlorite is separated by filtration or other means, and the solution remaining is treated with lime to precipitate basic calcium hypochlorite,

which may be used for making up lime slurry. Part of the solution, after addition of sodium chloride, may be used as a diluent in adding the sodium chloride to the lime slurry, thereby giving a thinner slurry and promoting the chlorination. W. J. WRIGHT.

Production of clear leach liquors. A. W. ALLEN (U.S.P. 1,731,450, 15.10.29. Appl., 24.11.25).—Extraction of nitrate from caliche is improved by promoting the formation and distribution of small crystals of sodium chloride, which act as a filter and assist the retention of slime, so that the effluent solution is free from suspended matter. To ensure formation of these crystals, the caliche must be finely crushed so as to restrict and cause more even distribution of the voids between the particles, and to form channels in which the crystals will be deposited from the leaching solution. The solvent is heated out of contact with the charge in order to prevent release of slime, and leaching is effected in an upward direction through a false bottom. W. J. WRIGHT.

Production of molybdates of alkaline-earth metals and of magnesium. I. G. FARBENIND. A.-G. (B.P. 331,472, 27.11.29. Ger., 8.12.28).—Roasted molybdenite is made into a pulp with water and milk of lime added at such a rate that the solution always remains only feebly alkaline. Lime may be replaced by baryta, strontia, or magnesia. A. R. POWELL.

Preparation of highly-activated iron oxide. G. E. SEIL (B.P. 332,259, 15.4.29).—Iron, or an iron-bearing material such as aniline sludge, is intimately mixed with an alkaline substance such as "fished salt," which consists of potassium and sodium carbonates. The mixture is introduced into a rotary kiln and heated either at 200–400° or, preferably, at 760–1200°. At these ranges of temperature a product of high activity is obtained, whereas at 400–760° only low activity results. The hot mixture is quenched with water and finely ground in a ball-mill while still wet, the ferric oxide and other insoluble material being subsequently filtered off. W. J. WRIGHT.

Manufacture of aluminium chloride. A. M. BULEY and H. BLUMENBERG, JUN. (U.S.P. 1,734,200, 5.11.29. Appl., 31.8.26).—Aluminium together with charcoal or other reducing agent, added for the purpose of combining with oxygen present, is introduced into a rotatable cylindrical container having radial blades to promote agitation. The mixture is heated to 700° to melt the aluminium, and chlorine is admitted. Aluminium chloride sublimes and is condensed in another vessel, from which the carbon monoxide or dioxide escapes through an outlet pipe. W. J. WRIGHT.

Manufacture of titanium compounds [white oxide pigment]. TITANIUM PIGMENT Co., INC., ASSEES. of L. W. RYAN (B.P. 308,725, 25.3.29. U.S., 27.3.28).—A slightly acid sulphate solution containing 7% Ti is added slowly with vigorous sliming to a solution of oxalic acid at 90–98°, whereby a basic sulphate is precipitated. Calcination of the product at 700–1000° affords a white titania pigment. Addition of a small quantity of phosphoric acid to the oxalic acid solution improves the colour of the calcined oxide. A. R. POWELL.

Manufacture of solid titanyl and titanate sulphates. I. G. FARBENIND. A.-G. (B.P. 309,047, 3.4.29. Ger., 3.4.28).—A concentrated titanium sulphate solution is atomised in a Krause dryer by a current of air at 300° and the product mixed with anhydrous sodium, barium, or magnesium sulphates. Alternatively, an organic binder may be added to the solution prior to drying. A. R. POWELL.

Production of pure sulphur. W. GLUUD, R. SCHÖNFELDER, and W. RIESE, ASSRS. to BERGWERKSVERBAND ZUR VERWERTUNG VON SCHUTZRECHTEN DER KOHLENTÉCHNIK (U.S.P. 1,771,293, 22.7.30. Appl., 23.7.26. Ger., 6.8.25).—The process described in B., 1927, 521 is claimed.

Gradual production of gases [e.g., chlorine]. W. E. KEMMERICH (U.S.P. 1,729,043, 24.9.29. Appl., 8.9.24).—The production of definitely determined quantities of chlorine, gradually and uniformly diffused for therapeutic purposes, is effected by causing a suitable chlorine-producing compound to react with acid in such a manner that the chlorine generated causes gradual gravitational contact of unattacked surfaces of the reagents. W. J. WRIGHT.

Converting hafnium and zirconium salts. J. H. DE BOER, ASSR. to N.V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,771,557, 29.7.30. Appl., 18.10.24. Holl., 15.11.23).—See B.P. 225,187; B., 1925, 545.

Extraction of krypton and xenon from oxygen. A. J. A. BLARINGHEM, ASSR. to SOC. ANON. D'ECLAIRAGE ET D'APPL. ELECTRIQUES (U.S.P. 1,772,202, 5.8.30. Appl., 5.11.24. Fr., 26.12.23).—See B.P. 226,783; B., 1925, 671.

Calcining apparatus (U.S.P. 1,754,854). **Solid carbon dioxide** (B.P. 312,231).—See I. **Hydrogen** (B.P. 332,571). **Hydrogen from hydrocarbons** (B.P. 332,057).—See II. **Flux for soldering etc.** (B.P. 314,400). **Nickel for catalytic hydrogen** (B.P. 314,944).—See X. **Fertilisers** (B.P. 332,639, 332,750, 332,803, and 332,864). **Fertilisers and sodium bicarbonate** (B.P. 331,451). **Manures** (B.P. 313,612).—See XVI.

VIII.—GLASS; CERAMICS.

Change of quartz into tridymite in silicate blocks in the presence of mineralisers and a partial substitution of quartzites by sand in a silicate bed. P. P. BUDNIKOV and I. C. SMELYANSKI (Res. Inst., Silicate Ind., Kharkov, 1929, 2, 51 pp.).—Addition of Martin slag assists the transformation of quartz into tridymite. Substitution of quartzite by sand (25%) does not impair the properties of silicate block.

CHEMICAL ABSTRACTS.

Effect of alumina and silica on some properties of glass. M. A. BESBORODOV (Glashütte, 1930, 60, 5–9; Chem. Zentr., 1930, i, 1353).—An increase in the alumina and silica contents retarded the devitrification of a boron-silica-alumina-lime-potash-soda glass, and increased the resistance towards water and sodium carbonate. The resistance towards sodium hydroxide and sulphuric acid rises with an increase in the silica content, but falls with an increase in that of alumina.

L. S. THEOBALD.

Radioactive method for testing the properties of glasses. O. HAHN and H. MÜLLER (Glastech. Ber., 1929,

7, 380—383; Chem. Zentr., 1930, i, 1355).—The emanating power of Jena glass and of a glass of the composition $\text{Na}_2\text{O}, \text{BaO}, 6\text{SiO}_2$, each melted with 0.1% of their weight of a substance containing radium, has been followed with respect to time and humidity of the atmosphere. In dry air the emanating power is small, especially in the case of Jena glass; at 83% humidity this glass remained unchanged whilst the barium glass quickly increased its emanation to 5.7% in 11 months. In saturated air this value rose to 19% in 11 months. The emanation is a measure of the surface development of a glass. The emanating power of Jena glass suddenly increased to a marked extent at 570°. L. S. THEOBALD.

Acceleration of the melting of glass. I. I. KITAI-GORODSKI (Keram. i Steklo, 1929, 5, 362—363).—Addition of ammonium salts (1—2%) facilitated the melting of a glass batch. CHEMICAL ABSTRACTS.

Cement for silica-glass joints. C. G. MAIER (Ind. Eng. Chem. [Anal.], 1930, 2, 337).—Silver chloride has many disadvantages when used for making detachable joints between silica and glass. A highly satisfactory joint is made by the eutectic mixture of thallous and silver chlorides, containing 60 mols.-% of thallous chloride and melting at 210°. When chilled rapidly the fused mixture solidifies as a fairly flexible, horny mass. For convenience of working it may be cast into sticks in chilled moulds. E. S. HEDGES.

Oil firing [of ceramic ware]. Report I. S. R. HIND (Trans. Ceram. Soc., 1930, 29, 149—168).—The results with coal- and oil-firing of the kilns of four firms are compared and discussed. It appears that, considering cost of fuel only, with oil at 90s. per ton, the competitive price of coal ranges between £2 and £3, but there are incidental advantages of oil, such as reduction of spoilt ware, of labour, more rapid firing and cooling, and more correct dimensions of the fired ware. About 24 hrs. can be saved in cooling, with improvement in the brilliance of glost ware. In an appendix a description is given of the fuel oil, of burners for it (all using steam), of viscosimeters, and a diagram showing loss of heat with excess air. B. M. VENABLES.

Apparent sp. gr. and moisture content of clay. F. K. CAMERON and R. A. LINEBERRY (Coll. Symp. Ann., 1929, 7, 179—189).—Addition of water to clays (100-mesh) caused no change in apparent d below 2.5% H_2O , above which balling began to cease and d to rise. Analogous phenomena occur with barium sulphate and pumice. A larger proportion is required of organic liquids which do not readily wet clay. Plasticity in a multi-component system is due mainly to a high surface tension, which is related to the apparent sp. gr.

CHEMICAL ABSTRACTS.

PATENTS.

Making laminated glass. J. KOLB, Assee. of A. G. WORRALL (B.P. 317,476, 29.7.29. U.S., 17.8.28).—Two sheets of glass cemented by a suitable binder are heated under partial vacuum to soften the binder and are finally pressed together. C. A. KING.

Laminated glass. H. A. GARDNER (B.P. 332,234, 11.3.29).—A toluenesulphonamide-aldehyde (*p*-toluenesulphonamide-formaldehyde) resin with nitrocellulose is used as the cement for laminated glass. C. A. KING.

Manufacture of refractory articles. HARTFORD-EMPIRE Co., Assees. of P. G. WILLETTS (B.P. 315,856, 7.6.29. U.S., 21.7.28).—A firebrick or refractory made from 55% of white bauxite, 35% of kaolin, and 10% of a white clay is claimed. The fired article has a composition between the following limits: 39.90—34.89% SiO_2 , 61.39—56.38% Al_2O_3 , 0.89—1.11% Fe_2O_3 , 2.65—2.72% TiO_2 , 0.25—0.27% MgO , 0.28% CaO , and 0—0.06% Na_2O . The batch is fired at such a temperature that the material is almost completely converted into mullite and has a porosity of about 27%.

A. R. POWELL.

[Tunnel] kilns. H. M. ROBERTSON (B.P. 305,025, 23.1.29. U.S., 28.1.28).—See U.S.P. 1,683,807; B., 1928, 839.

Continuous [tunnel] kilns. H. M. ROBERTSON (B.P. 332,619, 23.1.29).—See U.S.P. 1,737,540; B., 1930, 222.

Sealing of edges of laminated glass. L. J. KOLB, Assee. of R. W. HACKETT and A. G. WORRALL (B.P. 312,368, 24.5.29. U.S., 25.5.28).

IX.—BUILDING MATERIALS.

Thermal inefficiency of the cement rotary kiln. G. MARTIN (Trans. Ceram. Soc., 1930, 29, 143—148).—The paper comprises theoretical thermal data and deductions therefrom, mainly directed to show the transcendent importance of high flame temperature from the point of view of fuel economy. Figures showing the connexion between flame temperature and clinker output are tabulated, covering the range 2966° down to 805°; at the former point the consumption of coal is 6.36 tons per 100 tons of clinker, and at the latter point it is infinite. In a practical test the consumption agreed very well with the observed flame temperature. As a practical standard a fuel consumption of more than 22 tons of standard coal per 100 tons of clinker is considered highly inefficient.

B. M. VENABLES.

Hydration of Portland cement. Influence of monocalcium phosphate. I—VII. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 276—277 B; cf. B., 1930, 769).—Monocalcium phosphate if present in large proportions arrests the hydration of calcium aluminate, which is the principal factor in the setting of cement. Monocalcium phosphate interacts with lime whether originally present in the free state or produced by the hydration of calcium silicate, giving the insoluble bi- or tri-calcium salts. A small proportion of the mono-salt therefore has no injurious effect on setting. C. IRWIN.

Mixed Portland cements. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 192—196 B).—“Neosolidit” is a mixture of 70—75 pts. of Portland cement clinker, 10—15 pts. of calcined granite etc., and 10—15 pts. of siliceous matter containing soluble alumina and ferric oxide. The mixture is ground with 3—5% of gypsum. Chemical analyses are given. The material shows greater contraction on curing than straight Portland cement. On heating, the free lime attains a maximum at 400—600° and then diminishes. The curing process is slow, much free lime remaining after 28 days. C. IRWIN.

Revised procedure for determination of uncombined lime in Portland cement. W. LERCH and R. H. BOGUE (Ind. Eng. Chem. [Anal.], 1930, 2, 296—298; cf. B., 1926, 709).—Full details of an improved technique are given. The essential features of the method are the dissolution of the uncombined lime in a hot solution of glycerol and alcohol and the subsequent titration of the dissolved lime with an alcoholic solution of ammonium acetate. Some experimental results are given and the probable errors are represented graphically.

E. S. HEDGES.

Calcium ferrites and iron cements. III, IV. S. NAGAI and K. ASAOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 190—192 B, 256—259 B; cf. B., 1930, 769).—III. The compound $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$ is slowly decomposed by water, yielding calcium hydroxide, but $\text{CaO} \cdot \text{Fe}_2\text{O}_3$ is not acted on. The strength of the former is considerably decreased by water-curing.

IV. Mixtures of silica, ferric oxide, and lime, containing 10—12% Fe_2O_3 and having lime:silica ratios of 2.0—3.0:1, when heated together gave calcium ferrite as the first product, followed in succession by mono-, bi- and tri-calcium silicate. A similar result was obtained if part of the iron was replaced by aluminium. The presence of calcium ferrite or aluminate lowers the temperature of formation of tricalcium silicate by 100—150° as compared with that necessary in ordinary Portland cement.

C. IRWIN.

Why cannot aluminous cement and Portland cement be used as a mixture? K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 277—282 B).—*Ciment fondu* and Portland cement when mixed reduce the setting time of both, the shortest time of setting occurring with 70% of Portland cement. It was observed that this effect was more marked with badly-burned Portland cement, which suggested that the lime content of the latter was of importance. Experiments were made with cement to which lime had been added, and the setting of *ciment fondu* was followed microscopically using (1) water, (2) milk of lime; chemical tests were also made on the interaction of water and aluminous cement. It was concluded that the hydration of the latter by water proceeds as follows: $2(\text{CaO}, \text{Al}_2\text{O}_3) + 11\text{H}_2\text{O} = 2(\text{CaO})\text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O} + 2\text{Al}(\text{OH})_3$, and that this reaction is comparatively slow and reversible. If, however, lime is present the reaction is $\text{CaO} \cdot \text{Al}_2\text{O}_3 + \text{Ca}(\text{OH})_2 + 7\text{H}_2\text{O} = 2\text{CaO}, \text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O}$ and this reaction is rapidly complete and irreversible.

C. IRWIN.

Lime-alumina cement. II. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 196—198 B).—The partial replacement of alumina by ferric oxide reduces the absolute strength but diminishes the deterioration on water-curing and prevents the dusting of clinker. This latter is also an effect of the addition of 0.5—1.0% of borax. Further tests show that the deterioration of lime-alumina cement on water-curing is due to the dissolution of hydrated calcium aluminate. In this quality and in strength it is inferior to alumina cement.

C. IRWIN.

Laitance of cement mortars and concrete. II. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 260—

262 B; cf. B., 1930, 327).—The laitance on 1:2:4 concrete of low water-cement ratio is similar in composition to the cement, but with higher water ratios the lime content is increased relative to the silica. Other samples were found to contain large quantities of sand particles. Laitances from alumina cement or blast-furnace slag cement were found to contain 25—60% Al_2O_3 on the ignited sample. C. IRWIN.

Use of gypsum waste products. P. P. BUDNIKOV and V. M. LECHYEV (Keram. i Steklo, 1929, 5, 432—435). Cement obtained by heating gypsum at 500—700° has a shrinkage of 1—1.5%; shrinkage is prevented and the setting time reduced by addition of sodium hydrogen sulphate (0.6%) or copper sulphate (0.8%).

CHEMICAL ABSTRACTS.

Sinkage [of pulp-wood.] I. Mode of penetration of water into logs. G. W. SCARTH and E. C. JAHN (Canad. J. Res., 1930, 2, 409—424).—The distribution of water in logs floated on a lake is similar to that of living trees. The woods examined have a relatively dry heartwood, becoming wetter in the order jack pine, spruce, poplar, and balsam, the sapwood in all these cases being wet. In birch the heart- and sap-woods are equally wet. The rate of radial penetration increases in the order birch, jack pine, spruce, balsam, poplar, the penetration being very slow, even into sapwood. The advantage of a high proportion of dry heartwood is mainly in the initial buoyancy conferred. The rate of penetration is decreased by density and narrow outer rays. In air-dry logs, saturation of cell-walls precedes penetration of free water. The natural buoyancy of the logs is due to enclosed gases, which, being surrounded by water, can only escape in solution; further amounts of gas are probably formed by fermentation.

T. T. PORTS.

Electrical conductivity method for determining the moisture content of wood. A. J. STAMM (Ind. Eng. Chem. [Anal.], 1930, 2, 240—244).—The electrical conductivity of wood varies very greatly with its content of moisture, and a simple, compact, and portable electrical conductivity apparatus based on this fact has been designed for determining the average moisture content of wood. A special feature is the pin-point type of contact. The conductivity measurements can be translated directly into terms of average moisture content, not only when the moisture is uniformly distributed, but also when it is distributed according to any normal drying gradient. Experimental results obtained with 160 specimens of 25 kinds of wood show a mean deviation of 0.6% absolute moisture content and a maximum deviation of 1.7% from the values determined through loss of weight on drying in an oven. Surface moisture temporarily increases the moisture content value, but the normal value is regained after a short time. The usual surface-finishes of wood do not affect the readings.

E. S. HEDGES.

Pigments for façades. RASQUIN.—See XIII.
Rubber cements. BUSSE and DOGGETT.—See XIV.

PATENTS.

Manufacture of cement. F. S. WATT (B.P. 332,322, 7.5.29).—In a kiln the slurry is either injected through

a central nozzle surrounded by a number of flame jets which impinge on to the atomised slurry, or the respective flow of slurry and fuel is reversed, the object in either arrangement being to cause the spray to be injected into the hottest part of the furnace chamber.

C. A. KING.

Manufacture of magnesian cement. J. C. G. SPERNI (B.P. 332,282, 18.4.29).—An oxide of manganese (5–25%) is mixed with a magnesian cement composed of burned magnesite and clay, or the constituents may be calcined together and ground to powder. Sodium silicate may be incorporated in the mixture, and the cement is suitable, when gauged with water, for flooring or moulding into artificial stonework. C. A. KING.

Production of cellular solid bodies. J. W. BATTERSBY (B.P. 331,989, 17.5.29).—A solid, colloidal, soluble silicate of the alkali metals still containing about 25% of water is heated at 200–500°. The liberation of water vapour causes a cellular structure and moulded shapes may be formed. Alternatively, the silicate may be initially in the form of powder, and fillers, *e.g.*, coke, asbestos, or pumice, may be incorporated before heating.

C. A. KING.

Composition for making, maintaining, consolidating, and repairing highways or roads. "SAPONITE" (B.P. 310, 414, 11.4.29. Fr., 25.4.28).—A consolidating cement is prepared from a mixture of calcareous sand, hydraulic lime, and sodium silicate solution.

C. A. KING.

Progressive dry kiln and method of drying lumber. J. B. WELCH (U.S.P. 1,753,974, 8.4.30. Appl., 30.10.25).—The timber is moved progressively through a long chamber and subjected to a gradually increasing temperature, the atmosphere being maintained very moist at the green end by means of steam jets and the air recirculated in a number of zones by means of the convection currents produced by heaters aided by steam aspirators; each circulation is upwards through the lumber towards the dry end and downwards towards the green end.

B. M. VENABLES.

Preservatives for wood. GRUBENHOLZIMPRÄGNIERUNG GES.M.B.H. (B.P. 315,860, 21.6.29. Ger., 20.7.28).—Wood is impregnated with a solution at 70° containing 2% of a salt mixture consisting of 40% of ferrous sulphate crystals, 40% of sodium fluoride, 10% of arsenious acid, and 10% of sodium bisulphite. Alternatively, zinc or copper sulphates can be used instead of ferrous sulphate, sodium arsenate instead of arsenious acid, and potassium dichromate instead of the bisulphite.

A. R. POWELL.

Imitating the grain of wood or the like. MASA GES.M.B.H. ZURHERSTELLUNG KÜNSTLICHER OBERFLÄCHEN (B.P. 308,371 and 308,373–6, 22.3.29. Ger., 22.3.28).—(A) Printing blocks for intaglio printing are prepared from a photograph of natural wood-grain taken with the aid of ultra-violet rays having a wave-length less than 3700 Å. (B) The softer parts of the wood are first removed by chemical, physical, or mechanical means so as to throw the grain into relief. (C) The wood specimen to be copied is coated or impregnated with a coloured solution, a moistened gelatin paper is applied, and from the impression produced on the paper printing

plates are prepared by any known photo-mechanical process. (D) The specimen is soaked in a solution of a barium salt, then in a sulphate solution, and exposed to X-rays after a light-sensitive plate has been placed behind it. Alternatively, the wood is rubbed with a phosphorescent substance, exposed to the sun for some time, then laid on a photographic plate. (E) The wood is photographed by the aid of light from a quartz mercury-vapour lamp.

A. R. POWELL.

Bituminous composition. T. F. KING (U.S.P. 1,771,795, 29.7.30. Appl., 30.8.29. U.K., 3.8.28).—See B.P. 322,431; B., 1930, 178.

[Reinforced] composition flooring. H. L. TUCKER (B.P. 332,719, 3.7.29).

[Sectional] paving and like surfaces. L. COOKE (B.P. 332,741, 18.7.29).

Reproducing grain of wood [on printing plates]. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 308,372, 22.3.29. Ger., 22.3.28).

Kilns (B.P. 332,037).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Calorific value, heat and gas flow, the physical bases of metallurgical processes. H. BANSSEN (Stahl u. Eisen, 1930, 50, 668–678).—Diagrams have been constructed to show the heat balance in various stages of iron smelting—the blast furnace, gas producer, acid and basic open-hearth furnaces, converter and sponge iron producer—and the effect of preheating the gas and air is illustrated. A knowledge of the calorific value of the fuel and the rate of flow and heat content of the various gases is shown to be of paramount importance in the correct and efficient operation of the various processes. The resistance of the column of charge in the blast furnace to the flow of gas through the furnace determines to a large extent the output of the furnace, and any factor which increases this resistance reduces the efficiency of the furnace. One of these factors is the deposition of carbon from the flue gases in the upper part of the blast-furnace shaft, and an apparatus is described and illustrated for the determination of the magnitude of this deposition from blast-furnace gas.

A. R. POWELL.

Substitution of limestone by rock salt in blast-furnace practice. E. V. BRITKZE, A. N. KRESTOVNIKOV, and I. V. SCHMANENKOV (Min. Ssyrye Zwet. Met., 1929, 4, 359–375; Chem. Zentr., 1930, i, 1031–1032).—The charge of sodium chloride passes partly into the slag as sodium oxide, and partly into the gases as chloride; the proportion of the former (20–70%) increases, but not in simple ratio, with the charge, and is the greater the more finely divided and the more completely mixed are the materials. The reaction is represented by $2\text{NaCl} + \text{CO}_2 + 2\text{CO} \rightarrow \text{Na}_2\text{CO}_3 (\rightarrow \text{Na}_2\text{O} + \text{CO}_2) + \text{COCl}_2 + \text{C}_2$ (at 800–900°). A slag of optimal composition contained SiO_2 42.72, FeO 2.25, Al_2O_3 15.46, CaO 33.76, MgO 2.56, Na_2O 5.04%. The slag, the m.p. of which is depressed by 300°, is vitreous and separates well from the metal. A. A. ELDRIDGE.

Evaluation and testing of the properties of raw materials and key-products in the iron industry.

A. WAGNER (Stahl u. Eisen, 1930, 50, 655—668).—The present state of our knowledge of the properties of coal, coke, and iron ore used in the production of iron and steel and of the pig iron produced from various types of ore is reviewed and modern methods of determining the value of these materials for their particular purposes are briefly discussed. Laboratory tests of the coking power of coal and chemical analyses of iron ore do not give sufficient information as to the value of the coke and ore for metallurgical purposes, as no account is taken of their physical condition, which is of considerable importance in determining their behaviour in smelting. Similarly, chemical analysis of pig iron affords little information as to its value for subsequent operations, but, in combination with an examination of its micro- and macro-structure and a determination of its mechanical properties after remelting under standard conditions, the analysis gives a fairly accurate idea of the commercial value of the metal.

A. R. POWELL.

Practical importance of the influence of different cooling conditions on the structure of steel ingots.

F. LEITNER (Stahl u. Eisen, 1930, 50, 1081—1086).—The effect of varying wall thicknesses of the mould on the structure of plain carbon and nickel-chromium steels has been examined. The results show that a more fine-grained and homogeneous macrostructure is obtained by retarded cooling, and hence thin-walled moulds are to be preferred. Even better structures are produced by retarding the radiation of heat from the mould by covering it with a heat-insulating material or by using a double-walled mould with an annular air-space between the walls.

A. R. POWELL.

Theory of the malleabilising process. N. G. GIRSHOVICH and E. K. VIDIN (Trans. Inst. Met., Moscow, 1929, No. 4, 3—117).—The mechanism of graphitisation and decarburisation of white cast iron is discussed.

CHEMICAL ABSTRACTS.

Testing of sands for steel castings. S. PRÉARD (Bull. Soc. chim. Belg., 1930, 39, 252—270).—A description of methods of chemical analysis, and of the determination of grain size, binding power, permeability to air, and refractoriness of sands used for casting steel is given.

C. W. GIBBY.

Deoxidisation of steel with aluminium. C. H. HERTY, JUN., G. R. FITTERER, and G. N. BYRNS (Min. Met. Invest. Carnegie Inst. Tech., U.S. Bur. Mines Co-op. Bull., 1930, No. 46, 1—43).—Ferrous oxide and alumina coalesce slightly to form inclusions which are fusible below the m.p. of iron; inclusions high in alumina tend to group. In small ingots group-segregation of inclusions, undetected by analysis, caused cracking during forging. Equal weights of aluminium and silicon reduce the ferrous oxide content of steel to the same extent, but the reaction with aluminium is the more rapid.

CHEMICAL ABSTRACTS.

Influence of nickel and manganese on the properties of high-speed tool steel. V. EHMOKE (Arch. Eisenhüttenw., 1930—1, 4, 23—35; Stahl u. Eisen, 1930, 50, 1131—1132).—The hardness of high-speed

tool steel immediately after quenching is decreased by the addition of nickel; steels with more than 5% Ni have a purely austenitic structure, apart from the carbide segregations due to decomposition of the ledeburite eutectic, and their hardness is therefore relatively low, being only 300 Brinell units for the 5% Ni alloy. By subsequent annealing at 600—900° the austenite is converted into martensite and the hardness increases greatly, *e.g.*, to 575 Brinell units for the 5% Ni steel. With increasing nickel content the stability of the austenite becomes greater so that the temper hardness diminishes; a steel with 12% Ni is purely austenitic and cannot be hardened by tempering. Dilatometric tests on nickel high-speed tool steels show that the transformation from austenite into martensite takes place during a critical temperature range on cooling; this starts at 600° in the case of the 3% Ni steel and at 700—900° for the 5—12% Ni steels. In the case of the same steels without nickel a secondary hardening effect occurs at 580°, at which temperature the residual quenching austenite is converted into martensite. At temperatures up to 600—700° the tensile strength and hardness of nickel tool steels are much less than those of ordinary tool steels, but at higher temperatures nickel improves the strength and hardness. Manganese behaves in a similar manner to nickel in decreasing the hardness at the lower temperatures and increasing it above 600°.

A. R. POWELL.

Mechanical properties of copper steels with especial reference to the effect of heat treatment.

F. NEHL (Stahl u. Eisen, 1930, 50, 678—686).—The addition of more than 0.6% Cu to mild steel increases appreciably the yield point, but tends to make the metal brittle, a tendency which is overcome by the addition of 0.4% Cr. This alloy has a much higher tensile strength and yield point at high temperatures than silicon steel and is more resistant to corrosion. Quenching from above 800° and tempering at 450—550° increases the tensile strength of steels with more than 0.6% Cu (*cf.* following abstract) without seriously reducing the ductility and resistance to shock. Tables and graphs are given showing the effect of various heat treatments on the mechanical properties of several copper steels after varying mechanical treatment.

A. R. POWELL.

Temper-hardening of steel containing copper.

H. BUCHHOLTZ and W. KÖRNER (Stahl u. Eisen, 1930, 50, 687—695).—The solid solubility of copper in α -iron is 0.4% at 0—600° and then increases according to the equation $\log x = -4125/T + 4.32$ to 3.4% at the eutectoid temperature 810°. On cooling a copper-steel from the γ -solid solution range supersaturated solid solutions of copper in α -iron having a structure resembling that of martensite may be obtained; these supersaturated solutions as well as those containing 0.6—3.4% Cu quenched from 600—800° undergo age-hardening on tempering at 450—600°, but the separation of the excess copper in a highly dispersed condition and the subsequent coagulation of the particles take place very slowly. When the steel contains both copper and carbon in supersaturated solid solutions both elements separate in a disperse form on ageing at high temperatures, the two reactions taking place independently

of one another. Thus on tempering a quenched steel with 0.05% C and 2% Cu at gradually increasing temperatures the hardness rises sharply from 160 to 210 at 20° during 3 days, then to 220 at 100° due to separation of excess carbon; the hardness then falls rapidly to 150 at 300° owing to coagulation of the cementite, and rises again to 205 at 500–550° owing to precipitation of the copper. The properties of temper-hardened forgings of steel with 0.8% Cu and 0.4% Cr resemble those of a chromium–nickel steel with a tensile strength of 70 kg./mm.²; both steels are characterised by the regularity of their mechanical properties throughout large cross-sections and by their stability on prolonged storage after tempering.

A. R. POWELL.

Ternary system iron–nickel–sulphur. R. VOGEL and W. TONN (Arch. Eisenhüttenw., 1929–1930, 3, 769–780; Stahl u. Eisen, 1930, 50, 1090–1091).—This ternary system has been investigated by thermal and magnetic analysis within the region nickel–iron–FeS–Ni₃S₂. This part of the system includes only one ternary compound, (FeS)₂Ni₃S₂, which forms a continuous series of mixed crystals with Ni₃S₂ and which decomposes below 520° into a eutectic mixture of FeS and Ni₃S₂ after undergoing a polymorphic transformation at 615°. In the region studied only three primary crystal phases separate on cooling the liquid, viz., binary iron–nickel solid solution, either in the α - or γ -crystal phase according to the composition; FeS; and ternary solid solution of Ni₃S₂ in (FeS)₂Ni₃S₂ with excess nickel and iron. The metal solid solution forms eutectics with both sulphide phases. There is a ternary transformation point at 825° at which the liquid containing 38% Fe, 35% Ni, and 27% S reacts with FeS to form a ternary solid solution with 39% Fe, 32% Ni, and 29% S and a binary solid solution with 64% Fe and 36% Ni. Solid solutions rich in nickel and sulphur decompose at 480° forming a ternary eutectoid containing 13% Fe, 27% S, and 60% Ni and consisting of Ni₃S₂, (FeS)₂Ni₃S₂, and nickel–iron solid solution with 34% Fe. Ferrous sulphide reacts at 585° with the mixture containing 26% Fe, 46% Ni, and 28% S to form α -(FeS)₂Ni₃S₂ and an iron–nickel solid solution with 57% Fe. Thus at the ordinary temperature the system contains five fields having the composition (a) FeS + α - (or γ)-Fe–Ni, (b) FeS + γ -Fe–Ni + (FeS)₂Ni₃S₂, (c) γ -Fe–Ni + (FeS)₂Ni₃S₂, (d) (FeS)₂Ni₃S₂ + Ni–Fe + Ni₃S₂, and (e) Ni–Fe + Ni₃S₂. These results elucidate completely the composition of nickel matte and explain the presence therein of free metal similar to the hair-copper found in copper matte. This free metal occurs as leaflets when it has separated directly from the liquid mass and in hair-like crystals when it has been formed by the decomposition of ternary solid solution.

A. R. POWELL.

Determination of gases in metals, especially oxygen in iron and steel, by the hot extraction method. H. DIERGARTEN (Arch. Eisenhüttenw., 1928–9, 2, 813–828; 1929–1930, 3, 577–586, 627–635; Stahl u. Eisen, 1930, 50, 911–913).—By heating iron or steel in a graphite crucible in a high-frequency induction furnace or a carbon spiral furnace under a high vacuum at 1550–1650° all the oxygen is evolved as carbon

monoxide no matter in what form it is present in the steel. If more than 0.7% Mn or 0.3% Al is present part of these elements sublimes and reacts with the carbon monoxide, thus causing low results for oxygen; otherwise the method gives exceedingly accurate results. The oxygen content of cast iron lies between 0.002 and 0.014% under normal conditions, but much greater variations occur throughout an ingot than is the case with carbon, manganese, and phosphorus.

A. R. POWELL.

Determination of silica in the presence of silicon in ferrosilicon. A. STADELER (Arch. Eisenhüttenw., 1930–1, 4, 1–6; Stahl u. Eisen, 1930, 50, 1132–1133).—The finely-powdered sample (10 g.) is heated in a current of pure dry chlorine in such a way that the temperature rises to 300° in 30 min., 400° in 90 min., and to 500° in 150 min. Heating is then continued for 3 hrs. at 550°. The residue in the combustion boat is extracted with concentrated hydrochloric acid and the insoluble material collected, ignited, and weighed. The loss in weight after treatment with hydrofluoric and sulphuric acids is calculated as silica; the residue from this treatment consists of carborundum which is analysed in the usual way after fusion with sodium carbonate and nitrate. The chlorine is freed from oxygen by passing it over glowing wood charcoal. Blast-furnace ferrosilicon with 11% Si usually contains 0.06–0.10% SiO₂, 45% ferrosilicon about 0.14–0.26% SiO₂, and 90% ferrosilicon 1–1.32% SiO₂.

A. R. POWELL.

Zinc as a protective coating against corrosion fatigue in steel. W. E. HARVEY (Met. and Alloys, 1930, 1, 458–461).—The corrosion fatigue limit of uncoated steel in air is the same as that of electroplated specimens in water. With sherardised and galvanised coatings the presence of hard, brittle, intermetallic compounds caused premature failure.

CHEMICAL ABSTRACTS.

Corrosion tests of iron and steel in distilled, tap, and sea water. K. HASEGAWA and S. HORI (J. Eng. Assoc., Manchuria, 1930, 7, No. 35, 27 pp.).—The loss by corrosion was largest in distilled, less in sea, and least in tap water. In distilled water the corrodibility of steel increases proportionally to the carbon content; in tap and sea water it is maximal at 1% C. The corrodibility of cast iron is similar to that of medium-carbon steel; wrought iron shows the greatest resistance.

CHEMICAL ABSTRACTS.

X-Ray examination of commercial galvanised iron by a modified reflection method. C. W. STILLWELL and G. L. CLARK (Ind. Eng. Chem. [Anal.], 1930, 2, 266–272).—A modified method for obtaining X-ray diffraction patterns by reflection is described, and the method is especially suitable for the examination of the phases present in metals and alloys. It is sufficiently accurate to distinguish between several crystal forms, the lattices of which have already been established. The method has been applied to galvanised iron wire and has served to show that (1) there is no solid solution of zinc in iron adjacent to the steel base; (2) the compound next to the steel base is not FeZn₃, as formerly supposed, but is Fe₃Zn₁₀, (3) the outer layers of the coating contain the compound FeZn₇, a solid solution

of zinc in FeZn_7 , a solid solution of iron in zinc, and a phase with a body-centred cubic lattice the composition of which is unknown. A similar analysis of hot-dipped, Armco, sheet steel made it evident that (a) there is again no solid solution of zinc in iron adjacent to the steel base; (b) there is no evidence of the compound $\text{Fe}_3\text{Zn}_{10}$; (c) the intermediate layers consist of a solid solution of zinc in FeZn_7 and the body-centred cubic phase, the latter being more concentrated near the surface of the coating; (d) the surface of the coating is pure zinc, the crystals being oriented. The body-centred cubic lattice has not been observed in systems in equilibrium.

E. S. HEDGES.

Factors governing removal of soluble copper from leached ores. J. D. SULLIVAN and A. J. SWEET (U.S. Bur. Mines, Tech. Paper 453, 1929, 26 pp.).—The rate of removal of copper sulphate during the leaching of various ores with sulphuric acid has been studied under a variety of conditions. Although the time required to saturate crushed rocks with leach liquor is not a linear function of the particle size, the time of diffusion is almost so; therefore it is advisable to crush the ore reasonably small consistent with the production of a minimum amount of slime before feeding it to the leaching vats. When apparent equilibrium is reached in the vat 15% more of the total copper is retained in the residue at 2–3° than at 20°, but penetration of the leach liquor into the ore particles is somewhat greater and more rapid at the lower temperatures; hence it is recommended that leaching solutions should be added to the vats in the evening and washing carried out in the daytime when the temperature is higher. Alternate wetting and drying of the ore results in complete extraction of the copper in 15–20% of the time required in flood-washing. Tests made on the impregnation of rocks with copper sulphate solution show that the salt is disseminated throughout the rock especially along fractures and cleavage planes; on drying the impregnated rock the crystals of copper sulphate concentrate at or towards the surface, a fact which explains the beneficial action of alternate wetting and drying on the extraction by leaching. A low concentration of salts in the leaching solution does not appreciably improve the rate of diffusion of the copper, but there appears to be some relation between this rate and the viscosity of the solution. Thus 6% sulphuric acid is more sluggish in its action than 0.5% acid and a 4% ferric sulphate solution, which has twice the viscosity of the dilute acid, effects a complete removal of the copper in a time 50% greater than that required by the acid.

A. R. POWELL.

Acceleration of extraction of soluble copper from leached ores. M. GUGGENHEIM and J. D. SULLIVAN (U.S. Bur. Mines, Tech. Paper 472, 1930, 30 pp.; cf. preceding abstract).—The effect of alternate wetting and drying tests on the rate of extraction of copper sulphate from rocks previously impregnated with a solution of this salt has been studied. Maximum extraction was obtained by alternately washing for $\frac{1}{2}$ or 1 hr. and drying for $\frac{1}{2}$ or 1 hr. In chemical diffusion tests 50% of the total water-soluble copper was removed in 8 hrs. and 90% in 71%, whereas in the alternate

wetting and drying tests the same extractions were obtained in 2 and 17 hrs., respectively. Drying appears to bring the copper more rapidly to the surface, so that the smaller are the pieces of ore the more rapid is the extraction. Rise of temperature and drying the ore in a current of air increase the rate of dissolution of the copper.

A. R. POWELL.

Effects of silicon on the properties of brass. I. H. W. GOULD and K. W. RAY (Met. and Alloys, 1930, 1, 455–457).—Silicon (up to 2.8%) forms homogeneous α -solid solutions with 85:15 brass, but not with 60:40 brass; it forms a homogeneous γ -solid solution with the former at 8.6% and with the latter at 4.2%. All other brasses in the two series show two solid phases. Silicon lowers the f.p. of 85:15 and 60:40 brasses when present in amounts up to 10% and 7%, respectively. A tentative equilibrium diagram is advanced.

CHEMICAL ABSTRACTS.

Analysis of antifriction alloys. L. VENIKOV (J. Chem. Ind., Russia, 1929, 6, 209–210).—The alloy is dissolved in nitric acid; the residue after repeated evaporation with nitric acid is dissolved in oxalic acid, antimony trisulphide being precipitated from the solution; tin is determined electrolytically in the filtrate. The antimony sulphide is dissolved in sodium hydroxide solution, a small residue of lead and copper sulphides being dissolved in nitric acid and added to the original solution, in which lead and copper are determined in the usual way.

CHEMICAL ABSTRACTS.

Nickel casting alloys. J. McNEIL (Metal Ind., London, 1930, 36, 275–278, 305–306).—The effect of casting temperature and composition on strength and elongation are described. The solubility of lead in molten copper is increased from 35 to 60% by the addition of 2.5% Ni; with 2.5–3% Ni the two metals are miscible in all proportions.

CHEMICAL ABSTRACTS.

Coarse crystallisation of aluminium. R. KARNOP and G. SACHS (Metall.-Wirt., 1929, 8, 1115–1118; Chem. Zentr., 1930, i, 1209).—The tendency of aluminium to coarse crystallisation diminishes with increasing content of impurities. Iron is of greater importance than silicon.

A. A. ELDRIDGE.

Effect of iron on the properties of aluminium. S. A. POGODIN (Min. Syr. Tzvet. Met., 1929, 4, 616–624).—Addition of iron up to 5.4% increases the hardness, electrical resistance, and corrodibility of aluminium; up to 2.6% it increases the tensile strength and decreases the elongation and compressibility.

CHEMICAL ABSTRACTS.

Cleaning aluminium. R. W. MITCHELL (Metal Ind., N.Y., 1930, 28, 171–172).—The solution (of silicate or chromate), 2–6% by wt., should not evolve more than 2 c.c. of gas, nor dissolve more than 1 mg. of metal, per sq. in. of surface at 93–99°. No visible deposit should remain after rinsing, and the sample should not gain in weight.

CHEMICAL ABSTRACTS.

Solders for soldering aluminium. M. M. CHRUSHCHOV and M. V. SHAROV (Trans. Inst. Automotive Inst., Moscow, 1929, No. 9, 5–40).—Comparative tests were performed.

CHEMICAL ABSTRACTS.

Complete analysis of magnesium-aluminium alloys. S. S. SINGER (Ind. Eng. Chem. [Anal.], 1930, 2, 288).—A rapid method of separation of aluminium and iron from magnesium is based on the fact that iron and aluminium are not precipitated by sodium ammonium phosphate in the presence of tartaric acid, whereas magnesium is precipitated quantitatively. The precipitation of magnesium is greatly accelerated by chilling the solution in ice-water in addition to the usual vigorous stirring and scratching with a glass rod.

E. S. HEDGES.

Evaluation of stibnite. II. Determination of antimony. W. N. McNABB and E. C. WAGNER (Ind. Eng. Chem. [Anal.], 1930, 2, 251—256).—Titration of trivalent antimony by bromate, iodine, or permanganate gives practically identical results. The iron present as an impurity in stibnite is in the ferrous condition at the time of titration of the antimony, and affects the result. The permanganate method is recommended and a correction introduced by making a separate determination of the iron, and a method of effecting this is described. The Frankford Arsenal method (Cushman, Ind. Eng. Chem., 1918, 10, 380) gives results which are about 0.5% too low, due to loss of antimony by volatilisation. The authors' evolution procedure described formerly (B., 1929, 286) has been extended to include the iodometric determination of sulphide sulphur and the titration of antimony with permanganate. The analysis is rapid and accurate, and both determinations can be made on a single sample.

E. S. HEDGES.

Refining of tin. R. THEWS (Metallbörse, 1929, 19, 2553—2554, 2610—2611, 2666; Chem. Zentr., 1930, i, 1209).—The most undesirable impurities are iron, arsenic, antimony, zinc, and cadmium. Methods for the purification of tin are discussed.

A. A. ELDRIDGE.

Ternary alloys of lead. S. J. NIGHTINGALE (Brit. Non-Ferrous Metals Res. Assoc., Develop. Rep. D 3, 1929, 12 pp.).—The properties of lead alloys containing (a) 0.25% Cd and 0.5% Sb, and (b) 0.25% Cd and 1.5% Sn are compared with those of pure lead as regards their utility for the manufacture of piping. The tensile strength of the alloys is 1.62 tons/in.² compared with 0.9 ton/in.² for pure lead, the ductility 52—55% against 80%, and the fatigue limit 0.74—0.57 ton/in.² against 0.18. The alloys are on the whole more resistant to corrosion than is lead, and withstand a considerably higher bursting pressure, so that a lighter pipe can be used for the same service than is the case with lead. The working and soldering properties of the alloys are equally as good as those of lead and taking into account their superior properties are relatively less costly than lead; they do not undergo intercrystalline corrosion or fatigue.

A. R. POWELL.

Relative [flotation] activity of the various constituents of crude "cresylic acid." G. L. LANDOLT, E. G. HILL, and A. LOWY (Eng. Min. J., 1930, 129, 351—352).—The order of decreasing activity for a lead-zinc ore was: *m*-, *p*-, *o*-cresol, 1 : 3 : 4-, 1 : 4 : 5-, 1 : 2 : 4-, xylene, phenol, 1 : 3 : 2-xylene. Propylphenol is more active than *o*- or *p*-cresol. CHEMICAL ABSTRACTS.

Magnesite: its application in assaying. R. J. B. KETHEL (J. Chem. Met. Min. Soc. S. Afr., 1930, 30, 323—328).—South African magnesite deposits contain an average of 90% MgCO₃ and 5% SiO₂. For making cupels for gold assaying the material is calcined at 600°, ground to a fine powder, mixed with water, and moulded into bricks which are heated up to 1250° in 3—7 days in reverberatory or muffle furnaces. The calcined magnesite thus obtained is crushed so that 70—75% passes 200-mesh, mixed to a damp powder with a solution of glue or sugar, and pressed into the desired cupel shape in hand or compressed air presses. The cupels are dipped into a 1% solution of sodium silicate and dried for 12 hrs. at 100°, then set aside in a dry warm place to mature. A good cupel should absorb 86—90% of its weight in lead.

A. R. POWELL.

Influence of atmosphere, temperature, and nature of protection tube on the stability of platinum thermo-elements. F. STÄBLEIN and J. HINNÜBER (Arch. Eisenhüttenw., 1929—1930, 3, 781—785; Stahl u. Eisen, 1930, 50, 1089—1090).—Prolonged heating of platinum or 10% rhodium-platinum thermocouple wires in air, hydrogen, or coal gas results in a slow but only slight increase in the *E.M.F.* produced against new wire of the same type, but if the wire is enclosed in a protection tube much more serious changes take place which are shown to be due to the volatilisation of impurities from the sheath into the wires. At 1100° contamination is greatest in hydrogen, followed by carbon monoxide and carbon dioxide, and is least in air. Porcelain and refractory masses as protection tubes cause more serious contamination than iron, but fused silica is by far the most satisfactory. That the tube is the cause of the trouble is shown by the gain in weight of the wires and by an increase in their electrical resistance. When a couple is used in coal gas a heavy deposit of carbon is formed on the rhodium alloy wire at 850°, but the platinum wire remains quite bright, although at higher temperatures it, too, becomes covered with carbon.

A. R. POWELL.

Determination of inhomogeneities in metal specimens of any form by means of X-rays. E. Z. KAMINSKI and N. Y. SELYAKOV (Trans. Phys.-Tech. Lab., Leningrad, 1926, No. 3, 33—37).—Powdered media can be used provided that its absorption coefficient is of the same order as that of the specimen. Two or more powders must be well mixed and of similar coefficients of absorption.

CHEMICAL ABSTRACTS.

Anodes for the production of electrolytic zinc. H. R. HANLEY, C. Y. CLAYTON, and D. F. WALSH (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 321, 3—8).—The characteristics of several lead alloys for use as anodes have been examined. Calcium-lead anodes exhibit remarkable lowering of the anode potential, amounting to about 50% below that of pure lead. The alloy became passive on addition of 1% Ag. Thallium (more than 4%) causes remarkable passivity, but the anode potential is only slightly lowered.

CHEMICAL ABSTRACTS.

Resistance of electrodeposits to corrosion, with special reference to cadmium and zinc. W. S.

PATTERSON (J. Electroplaters' Dep. Tech. Soc., 1930, 5, 91—106).—The various factors governing corrosion are discussed. Steel is effectively protected against indoor corrosion by zinc or cadmium; the corrosion products of zinc do not affect subsequent corrosion, but cadmium is partly protected by its products. In a highly saturated and polluted indoor atmosphere the corrosion was at first similar for the two metals, zinc subsequently being the less vigorously attacked. In an outdoor atmosphere cadmium was corroded more readily than zinc, owing to the difficulty of formation of a less soluble basic cadmium sulphate. The mechanism of the corrosion is discussed. CHEMICAL ABSTRACTS.

Pressure die cast aluminium alloy test-pieces. J. D. GROGAN (Inst. Metals, Advance copy, 1930, No. 530, 18 pp.).

Rolled gold: its origin and development. E. A. SMITH (Inst. Metals, Advance copy, 1930, No. 539, 25 pp.).

Copper in brewing. STEWARDSON.—See XVIII.

PATENTS.

Apparatus for heat-treatment of metalliferous materials [roasting furnace]. E. M. S. INDUSTRIAL PROCESSES, LTD., R. A. STOKES, and E. G. L. ROBERTS (B.P. 330,930, 20.12.28).—A reverberatory furnace has a hearth formed of a series of troughs of arcuate cross-section over which the material to be roasted is passed by means of a series of paddles worked from water-cooled shafts lying parallel to the direction of the troughs. Discharge from the last trough is effected longitudinally by means of a screw conveyor and the heating gases are passed over the hearth countercurrent to the ore stream. A. R. POWELL.

Rendering utilisable cupriferous and zinciferous ores. A. L. MOND. From METALIGES. A.-G. and HOCHOFENWERK LÜBECK A.-G. (B.P. 318,232, 26.6.29).—The hot product obtained from the chloridising roasting of pyrites is cooled to about 100° by spraying it with the minimum amount of water, then air-cooled to below 75°, preferably to 50°, and leached with a small quantity of water or the acid liquor obtained by washing the roaster gases, whereby about 90% of the extractable zinc and only 15% of the extractable copper is removed in the leach liquor. The remaining zinc and the greater part of the copper are removed in subsequent leaches. The first leach liquor is worked up separately from the remainder by known methods. A. R. POWELL.

[Electric] furnace for reducing metals. G. C. COX and H. K. MCINTYRE (U.S.P. 1,753,822, 8.4.30. Appl., 5.10.26).—In an electric furnace in which the greater part of the hearth is inclined, the cathode is situated at the bottom of the low portion, and an anode for direct current at a higher level. Auxiliary electrodes for alternating current are provided to maintain the electrolyte at the optimum temperature. C. A. KING.

Production of metals and alloys by exothermic reduction. W. B. HAMILTON and T. A. EVANS (B.P. 331,892, 5.2.29).—The incipient heat required to induce reaction in a mixture of ore and a thermo-reducing

agent is provided by a high-frequency induction furnace. The reducing agent may consist partly of an energetically reacting mixture and partly of a slower acting mixture to maintain the heat when propagated. C. A. KING.

Apparatus for reduction of impurities contaminating molten metal. L. JONES (U.S.P. 1,753,891, 8.4.30. Appl., 19.7.28).—Molten metal direct from a reduction furnace is filtered through a layer of slag in an open refractory-lined container. An upwardly inclined discharge spout of increasing cross-sectional area is provided at the bottom of the container and the definite quantity of metal maintained therein is covered by a layer of molten slag through which fresh metal falls from a perforated covering disc. The used slag may be run off at intervals as necessary. C. A. KING.

Annealing furnaces. C. F. KENWORTHY (B.P. 332,436, 26.8.29. U.S., 14.1.29).—The furnace comprises a chamber opening at the bottom into a container for holding the material to be annealed and mechanical means for moving this container below the chamber and for raising the articles into the furnace; means are also provided for maintaining a non-oxidising atmosphere in the furnace. A. R. POWELL.

Production of pig iron. M. NEUMARK (B.P. 331,190, 22.3.29).—The fluxes used in the blast-furnace operation are so proportioned that the resulting slag contains >30% Al_2O_3 and <10% SiO_2 and is rich in lime. The resulting iron has a low content of sulphur and phosphorus (cf. F.P. 629,277; B., 1930, 149). A. R. POWELL.

Pickling process [for iron and steel]. J. C. VIGNOS (U.S.P. 1,736,332, 19.11.29. Appl., 29.8.28).—The metal is pickled in an acid ferrous sulphate bath through which chlorine is passed. A. R. POWELL.

Prevention of incrustation and corrosion in waterpipes etc. GROECK WASSERVEREDLUNG GES. M.B.H., Assees. of H. GROECK (B.P. 331,725, 14.8.29. Ger., 14.11.28).—Chromic acid or a soluble dichromate is added to the water of heating or cooling installations so as to form a light, non-adherent deposit instead of the usual hard crust. A. R. POWELL.

Non-corrodible alloys of iron and articles produced therefrom. K. M. TIGERSCHÖLD (B.P. 331,414, 2.8.29).—The alloys consist of iron with 15—30% Ni, 0—3% Cr, 0.05—1.5% C, 2.5—10% Si, and 0.2—2.5% Cu. Part or all of the silicon may be replaced by molybdenum and part or all of the nickel by cobalt. Sufficient carbon must be present to give the alloy an austenitic structure. A. R. POWELL.

Method of soldering [iron-chromium alloys]. H. S. GEORGE, Assr. to ELECTRO-METALLURG. CO. (U.S.P. 1,753,509, 8.4.30. Appl., 2.5.27).—Alloys such as stainless steel are soldered by applying a molten solder in conjunction with a piece of sodium to the metal faces. Ordinary silver solders containing about 80% Ag, 13% Cu, and 7% Zn are suitable. C. A. KING.

Coating metal bodies [e.g., iron] with metal [e.g., aluminium]. W. BAUR (B.P. 331,550, 3.4.29).—The metal is coated with aluminium paint which is allowed to harden and then heated to destroy the varnish

vehicle. The aluminium film is subsequently rolled or hammered into the surface so as to fill up the pores and other surface irregularities. A. R. POWELL.

Melting and refining of copper. C. A. JENSEN. From AMER. METAL Co., LTD. (B.P. 331,353, 7.6.29).—Copper is melted continuously below a layer of slag in a reverberatory furnace, new metal being charged into the flue of the furnace in such a manner that it is preheated practically to the m.p. before it is pushed into the bath of molten metal. The molten metal is tapped from below the slag layer at the opposite end of the furnace. A. R. POWELL.

Rendering articles of copper or copper alloys resistant to corrosion and oxidation. J. LAISSUS (B.P. 308,353, 18.3.29. Fr., 22.3.28).—The articles are packed in a box filled with powdered chromium, tungsten, molybdenum, tantalum, manganese, silicon, nickel, cobalt, vanadium, titanium, uranium, or zirconium, either alone or in the form of alloys, with or without the addition of an inert diluent, *e.g.*, silica or alumina. The box is placed inside a second box which is filled with grey-iron turnings and the whole is heated at just below the m.p. of the articles for a sufficient time to allow the case-hardening element to diffuse into the surface to the desired extent. For electrolytic copper and brass a cementation mixture comprising 65–60% of 80% ferrotungsten and 35–40% of alumina is preferred. A. R. POWELL.

Melting down easily fusible and easily oxidisable metals and alloys. C. BRACKELSBURG (B.P. 331,791, 7.11.29. Ger., 20.8.29).—A rotary tube-furnace heavily heat-insulated is heated by means of an oil or gas flame to a high temperature, the heat is shut off, the material to be melted is introduced, the furnace closed, and the charge allowed to melt by the heat of the furnace. A. R. POWELL.

Purifying light-metal [magnesium] alloys. J. A. GANN, ASSR. to DOW CHEM. Co. (U.S.P. 1,754,788, 15.4.30. Appl., 13.7.23).—The molten metal or alloy is agitated with a flux containing magnesium and sodium chlorides. Several treatments are advisable, but the flux from any treatment can be used for an earlier treatment of another batch of metal. A. R. POWELL.

Preventing corrosion of metallic surfaces. H. SUTTON, A. J. SIDERY, L. F. LE BROcq, and C. BRAITHWAITE (B.P. 331,853, 8.4.29).—Magnesium, or its alloys, is protected by immersing it in a neutral or acid solution containing the chromate or dichromate of an alkali metal and an aluminium salt. After drying, the metal is dipped in a solution of lanoline in benzene or coated with lacquer. C. A. KING.

Welding [rods for ferrous metals]. W. B. MILLER, ASSR. to OXWELD ACETYLENE Co. (U.S.P. 1,754,566, 15.4.30. Appl., 27.1.26).—The rod comprises soft iron, steel, or a chromium-iron alloy coated with a mixture of silica, ferromanganese, ferrosilicon, manganese ore, and a binder. The manganese and silicon may also be added as a manganese-silicon alloy with or without a proportion of magnesium which effectively removes gas from the joints. A. R. POWELL.

Welding-rod coating. W. J. HAMMERS, ASSR. to LIVERANCE & VAN ANTWERP (U.S.P. 1,752,793, 1.4.30.

Appl., 9.4.28).—A 2 : 1 mixture of iron oxide and felspar mixed to a cream with a 60% solution of sodium silicate is used for coating welding electrodes. C. A. KING.

Manufacture of saline flux coating compositions for use in soldering or welding. ALLOY WELDING PROCESSES, LTD., ASSEES. of SOUDURE ELECTRIQUE AUTOGENE Soc. ANON. (B.P. 314,400, 26.6.29. Fr., 26.6.28).—A mixture of sodium and potassium chlorides is ground with hydrofluosilicic acid and a solution of lithium chloride is added, followed, after 1 hr., by potassium hydroxide solution to cause peptisation of the precipitated fluosilicates. The mass may be ground in a colloid mill and other crystalloids added, *e.g.*, sodium sulphite or phosphate. The peptised fluosilicates act as protective colloids for the finely-divided crystalloids present in excess of their solubility in water. The mixture is suitable for coating aluminium and other light-alloy welding rods. A. R. POWELL.

Flux for welding. W. REUSS (B.P. 332,048, 3.7.29. Ger., 1.11.28. Addn. to B.P. 296, 923; B., 1928, 821).—The flux previously described may contain in addition ammonium and sodium chlorides and sulphuric or other mineral acid. All or part of the sodium chloride may also be replaced by alkali sulphates or nitrates. C. A. KING.

Mercury-vapour boilers. TRENT PROCESS CORP., ASSEES. of W. E. TRENT (B.P. 310,880, 26.4.29. U.S., 2.5.28).—In a mercury-vapour boiler the heating surfaces with which mercury is in contact are lined or coated with a non-ferrous metal, *e.g.*, silver, which will be "wetted" by mercury. Heating tubes may be of iron or steel plated with silver or of solid non-ferrous metal or alloy. C. A. KING.

Recovery of mercury [from catalyst sludges]. R. RILEY, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,106, 21.8.29).—Mercury sludge from acetylene hydration towers is chlorinated in a closed reaction vessel at the ordinary temperature, and the resulting mercuric chloride solution is treated with sodium hydroxide to precipitate mercuric oxide, which is redissolved in sulphuric acid to form mercuric sulphate for use again. A. R. POWELL.

Recovery of volatilisable metals [zinc, lead, bismuth, antimony, etc.] as oxides. Treatment of ores, metallurgical products, slags, etc. F. JOHANNSEN, ASSR. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,754,169 and 1,754,170, 8.4.30. Appl., [A] 4.1.26, [B] 14.10.26. Ger., [A] 26.5.25, [B] 5.11.25).—(A) After separation of the fine material by screening, the coarse material is passed downwards through a rotating tube-furnace into the middle of which is directed, from an injector at the lower end, a blast of compressed air carrying in suspension a mixture of powdered fuel and fine ore. In this way the latter is injected into the hottest zone of the furnace, thereby preventing caking and losses by dusting. (B) Easily fusible ores and metallurgical products containing volatilisable metals are fed into an inclined, rotating tube-furnace having an upper oxidising zone, and the fuel is introduced in a powdered form into the lower half of the furnace by means of an air blast. In this reaction zone the volatile metals are liberated and

the vapour is converted into oxide as it passes through the upper oxidising zone.

A. R. POWELL.

[Lead-antimony] bearing metal. P. KEMP (U.S.P. 1,754,364, 15.4.30. Appl., 11.1.28. Poland, 17.1.27).—The alloy contains 65–77% Pb, 3–14% Sn, 10–27% Sb, 0.7–2.5% As, and 0.5–3.5% Cd or In. The preferred composition is 66.8% Pb, 5% Sn, 25% Sb, 1.2% As, and 2% Cd.

A. R. POWELL.

Manufacture of [A] lead alloy products and [B] lead yarn. W. T. BUTCHER, W. CARROTT, and LOCKE, LANCASTER and W. W. & R. JOHNSON & SONS, LTD. (B.P. 330,925 and 330,960, 18.3.29).—(A) Lead alloys containing 4–12.5% Sb are rolled at 100–130° in a heavy rolling mill, which reduces the thickness of the casting by 50% in one pass, whereby the crystal structure is broken down without mechanical disintegration taking place. (B) Lead or lead alloys containing a small percentage of copper are extruded at 150–200° through triangular or polygonal holes in a metal die.

A. R. POWELL.

Manufacture of nickel in a finely-divided state in particular for the catalytic manufacture of hydrogen from a mixture of methane and water vapour. N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 314,944, 31.5.29. Holl., 5.7.28).—A solution of nickel nitrate in which a carrier may be suspended is treated at 40° with a slight deficiency of sodium hydroxide solution, so that the filtrate from the nickel hydroxide is perceptibly green. The carefully washed product is dried and reduced in hydrogen at 350°. A 72% yield of hydrogen is obtained by passing a mixture of methane and steam over the catalyst at 650°.

A. R. POWELL.

Precipitating system [for metals]. A. G. MCGREGOR (U.S.P. 1,753,015, 1.4.30. Appl., 4.5.28).—In plant for the replacement of metals from solution by other metals, *e.g.*, copper from copper sulphate solution by scrap iron, the solution is circulated through a series of launders containing scrap iron, the newest iron coming into contact with the most dilute liquor. Each launder in turn is cut out of service for discharging and recharging without stopping the process. The precipitated copper and unattacked iron in the launder discharge are separated by washing through a rotary screen.

D. K. MOORE.

Heat-insulating compositions for safes etc. A. E. WHITE. From MOSLER SAFE CO. (B.P. 332,356, 11.6.29).—A mixture of a vermiculite material, *e.g.*, jefferisite, kerrite, or maconite, diatomaceous earth, and plaster of Paris is run in between or applied to the walls of the safe.

C. A. KING.

Removal of carbon impurities from tantalum. C. W. BALKE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,754,453, 15.4.30. Appl., 2.9.26. Renewed 28.2.29).—Powdered tantalum containing small amounts of graphite is purified by heating it, *in vacuo*, with magnesia at a temperature at which the carbon will be oxidised and the resulting magnesium volatilised.

A. R. POWELL.

Separation of metallic values [precious metals from ores]. E. W. STEVENS (U.S.P. 1,754,408, 15.4.30. Appl., 20.12.29).—The ore pulp is fed through a rotating

circular feeder in the vertical axis of a centrifuge into a Venturi nozzle from which it is ejected into the bowl of the centrifuge. The heavier metal particles settle against the sides of the bowl, whereas the light gangue is caused to circulate in the middle of the bowl by the action of the nozzle.

A. R. POWELL.

Pen-point alloys. J. E. WOODWARD, Assr. to KASTENHUBER & LEHRFELD (U.S.P. 1,753,161–2, 1.4.30. Appl., [A] 19.12.27, [B] 22.5.29).—(A) An alloy consisting of 70–85 (80)% Os, 5–15 (10)% Pt, and 5–25 (10)% of a base alloy which contains 65–85 (75)% of a metal of the tungsten group and 15–35 (25)% of a metal of the nickel group is claimed. (B) An alloy containing 75% W and 25 Ni is first formed by gradually adding tungsten to molten nickel at about 1900°. Osmium is then added to the base alloy in the proportion of 80% of osmium to 20% of the latter until the temperature reaches 2400° (the m.p. of the ternary alloy) and the product is finally chilled.

C. A. KING.

Metallisation of thermally unstable substances, more particularly of organic electrically insulating substances. SIEMENS & HALSKE A.-G. (B.P. 306,902, 20.2.29. Ger., 27.2.28).—The material is heated at 100–400° and sprayed with the vapour of a metal carbonyl either alone or admixed with hydrogen or nitrogen. [Stat. ref.]

A. R. POWELL.

Coated metallic articles particularly in electrical apparatus and machinery. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 331,203, 18.3., 9.8., and 22.8.29).—Wires and metallic parts for electrical machinery are coated with resinous products made by the process of B.P. 327,722 (B., 1930, 624), to which, if desired, one or more colouring agents, fillers, or softening agents are added. The dried coating has a high dielectric constant and is highly elastic and pliable.

A. R. POWELL.

Testing metallic coatings. K. PITSCNER, Assr. to AMER. CHAIN CO., INC. (U.S.P. 1,753,301, 8.4.30. Appl., 22.12.26. Renewed 16.5.28).—Plated metal, *e.g.*, nickel or copper on iron, is tested for pinholes, cracks, etc. in the plating by applying a strip of paper treated with ferroxyl solution, made by stirring agar-agar into water, boiling, adding sodium chloride and potassium ferricyanide with stirring, and thinning out with methyl or ethyl alcohol. The presence of pinholes in the plating is revealed by brown or blue spots developing on the test-paper.

C. A. KING.

Alloy [for electrical heating units]. H. J. LORANG, Assr. to F. J. KUNA (U.S.P. 1,753,748, 8.4.30. Appl., 24.12.28).—The alloy contains 1.6% Cu, 1.6% Zn, 6.25% Sn, 21.75 Pb, and 68.75% Sb. The metals are melted in the presence of small quantities of manganese dioxide and borax.

C. A. KING.

Manufacture of porous metal articles [iron and nickel accumulator plates]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,052, 8.7.29).—A porous negative plate for alkaline accumulators is prepared by sintering a mixture of iron powder from iron carbonyl and 15% of colcothar at 600° in an atmosphere of nitrogen and hydrogen, then for 2 hrs. at 1100–1200° in the same atmosphere; the plate has a pore

volume of 81% and a high resistance to breakage. A porous positive plate is obtained by sintering a mixture of 90% of nickel powder and 10% of iron oxide in the same way; the plate has a pore volume of over 76% and is a good carrier for the positive mass in alkaline accumulators.

A. R. POWELL.

Production of printing plates. A. R. TRIST (B.P. 331,220, 25.3. and 12.4.29).—A sheet of copper is electroplated with a thin film of chromium and the film is activated by making it the cathode in a 10% acid solution containing 10% of ammonium sulphate. The chromium is then covered with a thin film of electro-deposited nickel and a thin coating of silver bromide emulsion is applied to the nickel surface. The plate is exposed in the usual way and the image developed with a pyrogallol developer. The nickel thus exposed is removed by treatment with alcoholic ferric chloride and the exposed chromium by electrolysis in a mixture of 1 pt. of hydrochloric acid (d_1 1.19) and 7 pts. of glycerin. The clean copper areas are then amalgamated by dipping the plate in mercuric cyanide solution. The plate is then cleaned, the nickel removed from the remainder of the plate by electrolysis in 2% hydrochloric acid, and the amalgamated copper part is plated with silver at 0.5 volt, at which *E.M.F.* silver is not deposited on the chromium.

A. R. POWELL.

Manufacturing chemically pure aluminium by electrolysis. K. L. MEISSNER (U.S.P. 1,770,940, 22.7.29. Appl., 1.8.27. Ger., 2.12.26).—See B.P. 299,869; B., 1928, 933.

Protecting surfaces of aluminium or aluminium alloys. G. D. BENGOUGH and J. M. STUART (U.S.P. 1,771,910, 29.7.30. Appl., 28.7.24. U.K., 2.8.23).—See B.P. 223,994; B., 1925, 14.

Coating bodies with tungsten. J. A. M. VAN LIEMPT, Assr. to N.V. PHILIPS' GLOBILAMPENFABR. (U.S.P. 1,771,974, 29.7.30. Appl., 2.12.24. Holl., 28.6.24).—See B.P. 236,153; B., 1925, 726.

Radiators (U.S.P. 1,753,318).—See I. **Treating ores etc.** (B.P. 332,366).—See II. **Catalyst for ammonia oxidation** (B.P. 331,728). **Kilns** (B.P. 332,037).—See VII.

XI.—ELECTROTECHNICS.

Moisture determination of wood. STAMM.—See IX. **Zinc.** HANLEY and others.—See X. **Manganese compounds and the quinhydrone electrode.** OSUGI and KASHIHARA.—See XVI. **Analysis of air.** HAMILTON.—See XXIII.

PATENTS.

[High-temperature] electric furnace. BRITISH THOMSON-HOUSTON Co., LTD., Assees. of E. HERZOG (B.P. 316,562, 30.7.29. U.S., 30.7.28).—The heating chamber comprises a hollow ceramic resistor, *e.g.*, of zirconium silicate, having a negative temperature coefficient of resistance, in which are embedded ceramic rods of material which is electrically non-conducting when cold but becomes conducting at a lower temperature than that of the furnace walls. Current is supplied to carborundum rods or a resistance wound on a refrac-

tory core within the resistor in order to raise the resistor to the temperature at which the ceramic rods become conducting.

J. S. G. THOMAS.

Electron-discharge device and getter therefor. E. A. LEDERER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,752,747, 1.4.30. Appl., 10.3.26).—A getter for an electron-discharge device consists of a phosphorus compound, *e.g.*, calcium metaphosphate, copper phosphide, or, more especially, phosphorus dinitride, a reducing agent, *e.g.*, powdered aluminium and magnesium, and a binder, *e.g.*, a solution of nitrocellulose in amyl acetate.

J. S. G. THOMAS.

[Preventing liberation of spray in] electrolysis. U. C. TANTON (B.P. 332,244, 17.4.29).—Substances, more especially a mixture of cresylic and silicic acids, together with gum arabic if desired, are added to the electrolyte so that a durable froth is formed.

J. S. G. THOMAS.

Electrolytic cell. F. LAWACZECK, Assr. to LAWACZECK GES.M.B.H. (U.S.P. 1,771,091, 22.7.30. Appl., 24.8.25. Ger., 1.9.24).—See B.P. 266,803; B., 1927, 338.

Thermostatic controlling means for electric ovens etc. N. DENNES, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 332,703, 19.6.29).

Methods of making selenium cells. C. ZEISS (B.P. 332,877, 27.12.29. Ger., 7.1.29).

[Laminated] electrically insulating materials. H. WADE, From BAKELITE CORP. (B.P. 332,594, 23.4.29).

Emulsions etc. (B.P. 332,533). **Degasifying liquids** (B.P. 332,542). **Controlling gas mixtures** (U.S.P. 1,753,319). **Hygrometers** (B.P. 332,135).—See I. **Acetylene and hydrogen** (B.P. 332,057).—See II. **Reducing metals** (U.S.P. 1,753,822). **Metallised insulating substances** (B.P. 306,902). **Coated articles** (B.P. 331,203). **Heating units** (U.S.P. 1,753,748). **Accumulator plates** (B.P. 332,052). **Printing plates** (B.P. 331,220).—See X. **Rubber articles from dispersions** (B.P. 331,563).—See XIV.

XII.—FATS; OILS; WAXES.

Selective dissolution of soap. S. KAWAI (J. Soc. Chem. Ind., Japan, 1930, 33, 244—245B).—Commercial toilet soap was immersed in distilled water for different periods of time and the dissolved soap decomposed with hydrochloric acid. The neutralisation and iodine values and m.p. of the fatty acids obtained were compared with those of the original soap content. The selective effect of dissolution was very marked and is considered to be of importance in the cracking of soap surfaces.

C. IRWIN.

Fluorescence of olive oil under ultra-violet light. A. LE R. GLANTZ (Ind. Eng. Chem. [Anal.], 1930, 2, 256—258).—All pure virgin oils exhibit a yellow fluorescence under ultra-violet light, and all refined oils show a characteristic blue fluorescence. By examination of the fluorescence, adulterations of as little as 5% of refined oil in a virgin oil can be detected. The fluorescence of California oils is due to the application of

heat during the pressing and also to the extent to which the olives are pressed. The spectrum analysis of virgin olive oils reveals a characteristic red band at 669μ , which is lacking in refined oils, but reappears on the addition of chlorophyll to the oil. The blue fluorescence of an oil is independent of its chlorophyll content. The result of adding carotene and annatto showed that the yellow fluorescence of virgin oils can be regained, and it is believed that this property of virgin oils is due to the content of carotene, which is destroyed during the refining. The addition of annatto, but not of carotene, can readily be detected by chemical analysts.

E. S. HEDGES.

Detection of carnauba wax in beeswax. L. K. WATSON (Amer. Bee J., 1930, 70, 118—120, 146).—The temperature of crystallisation of beeswax from butyl alcohol ($36-40^\circ$) is raised by carnauba wax (but not by tallow or paraffin) proportionally to the amount present. The microscopical appearance of crystals of beeswax (radiating shafts, or flakes) is altered (to minute stars or spherulites) in the presence of carnauba wax.

CHEMICAL ABSTRACTS.

PATENTS.

Treating materials (B.P. 332,577 and 332,593). **Making emulsions** (B.P. 331,928).—See I. **Sulphonated hydroxy-fatty acids** (B.P. 330,904).—See III. **Extracting fat from carcasses etc.** (B.P. 332,637).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Testing wood primers. H. A. GARDNER and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Circ. 368, July, 1930, 343—353).—An accelerated test is described in which the untreated backs of exposed painted panels were subjected to a humidity of 88% at 46° for 6 days. Moisture percolating through the wood causes rapid blistering etc. of incorrectly primed systems. The panels were subsequently removed to a test frame for further exposure, when paint affected by moisture in the early stages of drying usually disintegrate quickly by washing and chalking. The panels were braced to prevent warping. Details of a comprehensive series of tests recently undertaken on these lines on paint systems over various primers on different types of wood are given.

S. S. WOOLF.

Modification of the spinning-film hiding-power method [for paints]. G. S. HASLAM (Ind. Eng. Chem. [Anal.], 1930, 2, 319—322).—A method for measuring the hiding power of paints, regardless of their pigment or vehicle composition, has been developed. The test consists in applying a uniform film to a contrasty panel by spinning on an eccentric spinning device. Eye-grading in north light is the final criterion of hiding. Dry hiding power does not always agree with wet measurements as made on the cryptometer, and some of the exceptions are discussed.

E. S. HEDGES.

The Pfund glossmeter applied to paints and lacquers. G. S. HASLAM and L. D. GRADY, JUN., (Ind. Eng. Chem. [Anal.], 1930, 2, 346—351).—An apparatus for measuring objective gloss is described.

Data are given to illustrate the adaptability of the apparatus to paint and lacquer problems.

E. S. HEDGES.

Analysis of red lead. G. A. AMPT (J. Soc. Chem. Ind. Victoria, 1930, 30, 353—360).—Current methods of analysis are critically examined and the following procedure is advocated: 1 G. of sample is decomposed with 15 c.c. of 5*N*-nitric acid, 115 c.c. of water are added, and the liquid is heated to 100° . 20 C.c. of 0.2*N*-oxalic acid are then added and, after 10 min., the solution is titrated with 0.1*N*-permanganate. It is shown that the concentration of the nitric acid affects the titration and should be kept within specified limits.

H. I. DOWNES.

Fast pigments for façades. H. RASQUIN (Farben-Ztg., 1930, 35, 2279—2281).—Exposure tests on aged cement of various pigments in proprietary aqueous-emulsion vehicles are detailed. The suitability of the pigments for exterior painting work is discussed, with comments on their lime-fastness.

S. S. WOOLF.

Constitution of artificial resins. M. KOEBNER (Chem.-Ztg., 1930, 54, 619).—Published work explaining the constitution of phenol-formaldehyde resins by the formation of chains of phenol groups linked by methylene groups is briefly summarised. Crystalline compounds containing 2 and 3 cresol groups have been discussed previously (cf. Morgan, also Megson and Drummond, B., 1930, 600), and the preparation of chains containing up to 7 members is described. Whilst the less complex compounds are soluble in caustic soda solution, the material containing 7 phenol groups was apparently unaffected thereby, but was subsequently shown to form sodium salts insoluble in water. This loss of solubility has been erroneously attributed to loss of phenolic character by esterification etc., but the elimination of water to an extent made necessary by this assumption is considered unlikely.

S. S. WOOLF.

Rubber micro-sections. ALLEN.—See XIV. **Foreign materials in resins etc.** GEORGI.—See XVII. **Hop resins.** BAZILEVICH.—See XVIII.

PATENTS.

Mills for grinding paints, enamels, inks, and other viscous substances. DISC BAR MILLS, LTD., and E. A. WHITE (B.P. 331,762, 17.9.29).—The mill comprises a disc rotating against or near to a stationary block. The surfaces may or may not be roughened.

B. M. VENABLES.

Drying kilns (B.P. 332,491).—See I. **Cellulose acetate compositions** (B.P. 306,911).—See V. **Titanium pigments** (B.P. 308,725).—See VII. **Laminated glass** (B.P. 332,234).—See VIII. **Coated articles** (B.P. 331,203).—See X. **Adhesive** (B.P. 331,428).—See XV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Autographic stress-strain curves of rubber at low elongations. A. A. SOMERVILLE, J. M. BALL, and L. A. EDLAND (Ind. Eng. Chem. [Anal.], 1930, 2, 289—293).—A new test-piece adapted to the autographic registration of stress-strain curves is described, and the effects of variations in curves, speed of testing machine,

temperature, humidity, and loading of carbon black are discussed. E. S. HEDGES.

Making micro-sections of rubber stocks. R. P. ALLEN (Ind. Eng. Chem. [Anal.], 1930, 2, 311—313).—A simple method for making thin sections of rubber for microscopical examination is described. For clear observation of highly-loaded gas-black stocks the sections must be less than 1 μ thick, though somewhat greater thicknesses may be used with lighter-coloured pigments, such as litharge and zinc oxide. The method has been developed for the study of pigments in rubber stocks, but it is further pointed out that rubber is an excellent medium in which to examine finely-dispersed pigments, for it is rigid enough to prevent Brownian movement and sufficiently plastic to be easily deformed, and its refractive index is considerably different from that of most pigments. E. S. HEDGES.

Viscosity of rubber solutions under the influence of benzoyl peroxide. G. FROMANDT (Kautschuk, 1930, 6, 177—180).—In the determination of mineral ingredients such as antimony sulphide in commercial rubber articles by heating with a rubber solvent such as dekaline, the addition of about 3% of benzoyl peroxide to the solution after disintegration of the sample followed by further boiling for 1—2 hrs. greatly facilitates sedimentation of the mineral matter. The addition of benzoyl peroxide (1 g.) to a solution of purified rubber (1 g.) in benzene (100 c.c.) causes a greater rate of decrease in the viscosity of the solution than heating at 80°. Exposure of the solution, with or without benzoyl peroxide, to sunlight or ultra-violet radiation accelerates the change, the effect of sunlight for one day being roughly equivalent to that of heating at 80° for 8 hrs.

D. F. TWISS.

Application of the falling cylinder to the measurement of the viscosity of thick rubber cements. W. F. BUSSE and W. B. DOGGETT (Ind. Eng. Chem. [Anal.], 1930, 2, 314—319).—With many thick cements the time of fall, t , of cylinders can be expressed by the formula $dt = k \cdot dx^2 (W - \pi r^2 \rho x)^n$, where ρ is the density of the cement, n a constant of the cement, and k a constant which is independent both of the distance, x , through which the cylinder falls, and of the weight, W , but varies with the cement under test and the radius of the cylinder used. A 15% suspension of clay in mineral oil, in contrast with rubber solutions, showed a definite yield value and also anomalous turbulence. Falling-cylinder viscosimeters using steel cylinders have been designed for use in control testing, and simplified equations for their use have been developed. They were found to be satisfactory over a wide range. The thicker cements made from only slightly milled rubber do not follow the law on which the equations given are based, but even in these cases the viscosimeters can be used for control testing, provided that the shearing force at the surface of the cylinder is not greater than the adhesion of the cement to the cylinder surface. The steel viscosimeters have been used in testing asphalts having viscosities up to about 900,000 poises and should be useful in testing other materials. E. S. HEDGES.

PATENTS.

Treatment of rubber and products thereof. NAUGATUCK CHEM. CO., ASSCS. OF S. I. STRICKHOUSER

(B.P. 332,489, 30.10.29. U.S., 26.11.28).—Surface-cracking of soft vulcanised rubber materials, especially those of high rubber content, is retarded by incorporating an α -diketone (e.g., 0.5% on the rubber content), such as benzil, phenanthraquinone, or furil.

D. F. TWISS.

Manufacture of articles of rubber and similar substances from aqueous dispersions containing such substances. DUNLOP RUBBER CO., LTD., D. F. TWISS, and R. G. JAMES (B.P. 331,563, 7.2.29. Addn. to B.P. 301,100; B., 1929, 105).—Aqueous rubber dispersions in which the particles are negatively charged are treated with formic or acetic acid in the presence of a suitable protective colloid to make the charge on the particles positive, and are then subjected to electrophoretic deposition upon cathodes coated with a paste of litharge, red lead, and glycerin to prevent liberation of hydrogen.

A. R. POWELL.

Manufacture of rubber goods. ANODE RUBBER CO., LTD. (B.P. 317,435, 4.4.29. U.S., 16.8.28).—The material to be coated is dipped first into rubber latex, then into a solution of a coagulant, and, after wiping, into an aqueous dispersion of rubber. Any desired thickness of rubber may be built up by repeating the second and third operations; the whole is then vulcanised *in situ* or after removing the rubber layer from the base.

A. R. POWELL.

Manufacture of artificial sponges of rubber. Y. CORNIC (B.P. 314,539, 27.6.29. Fr., 29.6.28).—Rubber latex containing 70—80% of rubber is treated with 1% of an accelerator base (a guanidine derivative), 10—12% of a saturated solution of sulphur in carbon disulphide, 4% of zinc oxide, a suitable quantity of inert filler and/or colouring matter, and 12% of ammonium carbonate. The whole is thoroughly mixed and heated at 140° for 2—3 hrs., whereby the ammonium carbonate decomposes as the rubber vulcanises and a porous spongy mass is obtained.

A. R. POWELL.

Manufacture of rubberised fabric etc. FIRESTONE TYRE & RUBBER CO., LTD. From FIRESTONE TYRE & RUBBER CO. (B.P. 331,594, 10.1.29).—In the manufacture of rubberised fabric the material is passed through a drying chamber to which inert gas is supplied, and the composition of the gas mixture in the chamber is controlled by passing a portion of it over an electrical resistance coil the resistance of which varies according to the thermal conductivity of the gas mixture.

A. R. POWELL.

Manufacture of sea caoutchouc. C. A. HOUQUES-FOURCADE (U.S.P. 1,772,218, 5.8.30. Appl., 8.10.27. Fr., 9.10.26).—See F.P. 622,896; B., 1928, 762.

Rubber-like substance from cashew-nut-shell oil. M. T. HARVEY, ASSR. to HARVEL CORP. (U.S.P. 1,771,785, 29.7.30. Appl., 28.11.25).—See B.P. 262,134; B., 1928, 420.

XV.—LEATHER; GLUE.

Effect of neutral salts on the alkaline swelling of hide powder. I. Sodium chloride. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1930, 14, 355—358).—The addition of sodium chloride to 0.1N-sodium hydroxide causes a diminution in its swelling power on hide powder. As the amount of added sodium chloride

is increased, the swelling power diminishes to a minimum, after which further additions of salt cause a rapid rise in swelling power.

D. WOODROFFE.

Stripping effect of fat liquors on dyed leathers. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1930, 14, 353—355).—Samples of vegetable-tanned sheepskin dyed with different acid dyes were fat-liquored with different commercial fat liquors and soap-oil fat liquors. It was found that all except sulphonated cod oil stripped some of the colour of the dyed leathers, but the amount of stripping depended on the particular fat liquor used, the p_H values of which were found to differ. Samples of leather dyed with 32 different acid dyes were treated with the above-mentioned fat liquors, one half of each sample being dried out and the other treated with acid before drying. No stripping of the colour was observed from those pieces which had been pretreated with acid. The stripping effect of fat liquors is due to their effect on the p_H of the dyed leather.

D. WOODROFFE.

Analysis of bating materials. A. BOIDIN (J. Soc. Leather Trades' Chem., 1930, 14, 358—363).—Milk concentrated to one half its volume and to which 4% of calcium chloride has been added is used as the substrate. One part of the bating material is extracted with 10 pts. of a solution (p_H 5.3) containing 44.1 g. of potassium dihydrogen phosphate and 2.97 g. of disodium hydrogen phosphate in 5 litres of water. Then 1 c.c. of the milk, 0.2 c.c. of a solution (p_H 5.8) containing 95.5 c.c. of 0.67*M*-potassium dihydrogen phosphate and 2.5 c.c. of 0.6*M*-disodium hydrogen phosphate, 1.0—0 c.c. of boiled enzyme infusion, and 0—1.0 c.c. of untreated enzyme infusion are mixed and kept in a water-bath at 35°; the time taken for an image on a kinematograph film to be seen through the mixture is then compared with that required by a standard product under similar conditions.

D. WOODROFFE.

Use of calcium hydroxide prepared from marble in the leather industry. J. OETTINGER (Chem.-Ztg., 1930, 54, 586).—The product obtained by slaking marble lime sufficiently to yield a dry powder contains 96% $\text{Ca}(\text{OH})_2$. By its use in liming, a large excess is unnecessary, stains and lime burns are avoided, and the lime pits do not require to be emptied so frequently. This lime does not require to be slaked. It can be used for making lime paints or in liming glue pieces.

D. WOODROFFE.

Japanese dyeing tannins. VII. Analysis of the dyeing tannins by the cinchonine method. Y. UYEDA (J. Soc. Chem. Ind., Japan, 1930, 33, 228—231b).—The effect of varying conditions on the determination of tannin by the cinchonine method is examined. To ensure accuracy the solution to be analysed should contain about 0.4% of soluble tannin to which are added 25 c.c. of cinchonine sulphate solution (1.3 g./100 c.c.) for each 0.1 g. of tannin in solution. F. R. ENNOS.

Problem of tanning and its generalisation. L. MEUNIER and K. LE VIET (Rev. Gén. Colloid., 1930, 8, 49—61).—A method for measuring swelling particularly applicable to fibrous structures has been developed; it entails centrifuging the substance so as to determine

the bound liquid and not the capillary liquid. The method has been applied in an examination of the swelling of hide and the effect of some organic phenolic compounds thereon. The amount of swelling is considered to be determined by the presence in the protein molecules of certain lyophilic groups, particularly amino-groups, which exert a polarity comparable with that of the water molecules in the external liquid. When tanning occurs, these groups are transformed irreversibly into less polar groups and the resulting substance is greatly reduced in swelling capacity. The measurement of swelling gives a value for the astringence of the tanning solution. The action of certain tanning agents, such as quinol, depends on the external conditions of oxidation. The theory of tanning suggested is extended so as to apply to general colloid phenomena of a similar nature. The swelling of cellulose and of agar is considered to be due to the presence of hydroxyl groups, and the action of tanning agents is similar to the effect on collagen.

E. S. HEDGES.

Standard methods (revised) for determining viscosity and jelly strength of glue. (Ind. Eng. Chem. [Anal.], 1930, 2, 348—351; cf. B., 1924, 394).—Standard methods adopted by the National Association of Glue Manufacturers (U.S.A.), for sampling and preparation of sample, testing of sample, viscosity determination, jelly strength determination, and chilling are given. Some general precautions are emphasised.

E. S. HEDGES.

Foreign materials in glue etc. GEORGI.—See XVII.

PATENTS.

Pretreatment of hides for promoting tanning thereof and preparations therefor. B. QUENDT (B.P. 332,262, 10.1.29).—To accelerate the subsequent tannage, hides ready for tanning are pretreated with a solution of a water-soluble, non-sulphonated condensation product of an arylamine (e.g., aniline, dimethylaniline) with formaldehyde, alone or with a dilute tanning solution, or with a mixture of alum and one or more neutral salts (e.g., Glauber's salt).

D. WOODROFFE.

Treatment of casein. P. SCHIDROWITZ, Assr. to J. B. CROCKETT (U.S.P. 1,754,651, 15.4.30. Appl., 8.12.28).—A preparation of casein which will remain fluid when cold is made by heating it with alkali solution and then adding piperidine.

E. B. HUGHES.

Adhesives. J. TAYLOR and A. V. KELLER (B.P. 331,428, 29.8.29).—Urea, thiourea, or their derivatives are heated at 90° with paraformaldehyde and water to produce a thin paste; a violent reaction occurs with effervescence and a transparent liquid is obtained which is evaporated at 90° to a syrupy consistency before use as an adhesive.

A. R. POWELL.

Unhairing and preparing hides for tanning. C. K. M. M. LE PETIT, Assr. to ROHM & HAAS Co. (U.S.P. 1,772,258, 5.8.30. Appl., 7.7.25. Fr., 18.4.25).—See B.P. 250,907; B., 1926, 1022.

Adhesive or cement. R. ARNOT (U.S.P. 1,771,553, 29.7.30. Appl., 3.9.24. U.K., 15.9.23).—See B.P. 225,953; B., 1925, 108.

Cellulose acetate compositions (B.P. 306,911).—See V. Dyeing leather (B.P. 309,166).—See VI. Dissolving glue etc. (B.P. 307,079).—See XVII.

XVI.—AGRICULTURE.

Lime additions to friable clay. W. BRENNER (Bull. Agrogeol. Inst. Finland, 1929, No. 29, 13 pp.; Proc. Internat. Soc. Soil Sci., 1930, 5, 129—130).—Addition to soil of lime in quantities predetermined from titration curves to produce p_H changes up to 5.6 effected the calculated changes. To bring about p_H 6 in the field, 1.6 times the quantity of lime calculated from laboratory tests was required. It was impossible in the field to bring the p_H value to 7.0. The most heavily limed areas of this soil rapidly developed further acidity.

A. G. POLLARD.

Agrogeological studies in the tropics. I. High altitudes of the Oriental tropics. M. W. SENSTIUS (Soil Res., 1930, 2, 10—56).—Modern conceptions of soil formation are summarised and discussed. Field examination and laboratory analyses of tropical soils from high altitudes are recorded. Although podsoils were not found at high altitudes, evidence is obtained that in these soils the podsolisation process has occurred with minor modifications, which are examined and explained.

A. G. POLLARD.

Adsorbed bases and unsaturation of Polish sandy soils. W. BUTOWSKI (Rep. Polish Agric. Exp. Sta., 1928, 2, 195; Bied. Zentr., 1930, 59, 348).—The proportion of adsorbed bases in the soils examined varied with their mechanical composition and increased with the clay content. The plant covering of forest soils increased the adsorbed bases in the upper horizons. The amount of adsorbed bases was less in the B-horizon than in the surface, but increased at still greater depths approaching the parent rock. Variations in the degree of unsaturation were parallel with those of the base content and were greatest in the loams. The genesis of the soils is discussed. Podsolisation is considered to take place in these soils.

A. G. POLLARD.

Drainage and leaching trials at Peradeniya, 1927-30. A. W. R. JOACHIM (Trop. Agriculturalist, 1930, 74, 323—330).—Analyses of drainage waters from cropped and uncropped soils in cylinder experiments are recorded. In the leachings nitrates, calcium, magnesium chloride, and potassium occurred in decreasing order of magnitude.

A. G. POLLARD.

Soil humus. S. OSUGI and Y. SANO (J. Sci. Soil and Manure; Proc. Internat. Soc. Soil Sci., 1930, 5, 112—113).—Soil humus was fractionated by Woksman's method. The soluble portion is too small to be significant. The carbon, nitrogen, and ash contents of the α - and β -fractions were examined. No particular relationships between the α -fractions of rich humus soils or the β -fractions in mineral soils were observed. Schollenberger's chromic acid method for determining humus (B., 1927, 663) is satisfactory if samples of suitable size are used, e.g., 0.01 g. sample of soil with 30% humus, 0.02 g. with 15-20%, and 0.1 g. with 5%.

A. G. POLLARD.

Reactions of electro dialysed humus and benton-

ite. A. O. ALBEN (J. Amer. Soc. Agron., 1930, 22, 311—326).—Observations on Tama and Clinton silt loams are recorded; the final p_H was 2.04. The p_H of an electro dialysed soil returned in 60 days to 4.0. The application of electro dialysis to basic soils is discussed.

CHEMICAL ABSTRACTS.

Mechanical analysis of soils without acid pretreatment. L. B. OLMSTEAD and L. T. ALEXANDER (Soil Res., 1930, 2, 68—72).—Modifications of the International method of mechanical analysis are described. In the pretreatment of the sample, the acid decomposition is omitted and, where manganese dioxide is present in sufficient amount to prevent the satisfactory action of hydrogen peroxide, glacial acetic acid is added in quantity slightly in excess of the equivalent of manganese dioxide. Subsequent filtration and washing of the sample is carried out with a Pasteur-Chamberland candle from which the soil is removed by filling the cone of the candle with distilled water and applying pressure. The washed sample is evaporated to dryness on a steam-bath and baked in an oven at 105°. This permits of determinations of "loss by solution" without interfering with the subsequent dispersion of the sample. The sample is shaken with water containing 10 c.c. of 0.5*N*-sodium oxalate and, after separation of the coarser fractions by sieving (up to 300-mesh), the suspension is diluted to 1 litre for analysis by the pipette method for clay (<5 μ) and colloid (<2 μ). Variations in the amount of sodium oxalate necessary for complete dispersion may be necessary.

A. G. POLLARD.

Dispersion and mechanical analysis of certain soils high in sesquioxides. L. T. ALEXANDER (Soil Res., 1930, 2, 72—76).—A sample of bauxite could not be dispersed for mechanical analysis either with sodium oxalate (cf. previous abstract) or with ammonium hydroxide, but was completely dispersed with sodium hydroxide (5 c.c. of 0.6*N*-solution per litre). Lateritic soils of low silica : sesquioxide ratio and small base-exchange capacity may tend to flocculate in sodium oxalate suspensions and are preferably dispersed in sodium hydroxide. This difficulty arises only when the colloid fraction (<2 μ) is required separately, the sodium oxalate method being generally suitable when only the clay fraction (<5 μ) is needed.

A. G. POLLARD.

Nitrification in uncultivated soils. I. Various analyses. II. Nitrification as affected by hydrogen-ion concentration. T. GAARDER and O. HAGEM (Medd. Vestlandets Forstl. Forsöksstat., 1921, 2, No. 4, 172 pp.; 1928, 4, No. 11, 194 pp.).—I. Nitrate production in numerous uncultivated soils is examined. Poor nitrification in many forest and peat soils is traced either to the presence of denitrifying organisms or to the cessation of the oxidation process at the nitrite stage. The moisture content of soil affects nitrification mainly by its influence on soil aeration. The nitrifying power of soil is influenced directly by the soil reaction (but not by its buffer capacity) and by the presence of suitable mineral nutrients for the organisms concerned, and, indirectly, by the numerous factors controlling ammonification. The presence of nitrites in soil interferes with the accuracy of the phenolsulphonic acid method for

determining nitrates. Preliminary decomposition with urea is recommended.

II. The effect of the soil reaction on the nitrification of ammonium sulphate is examined. Nitrifying organisms are active within the p_H limits 6.0–7.8 (approx.). Exact values are difficult to specify owing to the progressive change in soil reaction occurring during the process. Probably four types of organisms, each active over a specific range of reaction, are present in soils. Nitrification of nitrites occurs in soils with p_H 6.8 to above 9.0; here again several types of organisms are concerned.

A. G. POLLARD.

Agreement between "Azotobacter values" and results of the seedling method. H. NIKLAS, H. POSCHENRIEDER, and F. CZIBULKA (Superphosphat, 1930, 6, 111–115).—Comparative investigations of numerous soils by the Neubauer and *Azotobacter* methods for determining available phosphate contents are recorded, and from the values obtained co-ordination tables for the two methods are prepared. In general, agreement between the methods is good, the least satisfactory being the intermediate range of phosphate contents corresponding with Neubauer values of 4–8 mg.

A. G. POLLARD.

Potassium thiocyanate method for determining soil acidity. Y. KAMOSITA (J. Imp. Agric. Exp. Sta., Tokyo; Proc. Internat. Soc. Soil Sci., 1930, 5, 103–104).—The colour intensity in the Comber test for soil acidity was standardised by titrating the alcoholic extract with potassium hydroxide solution. Values so obtained for a number of soils were compared with p_H values, exchange acidity, and exchangeable base content. Soils exhibiting exchange acidity have $p_H < 5.5$. Soils with $p_H < 6.88$ give, with Comber's test, red colorations which increase in intensity with increasing exchange acidity. In soils showing exchange acidity the proportions of exchangeable calcium, and magnesium are smaller than those of iron and aluminium. The reverse is the case where no exchange acidity exists. It is suggested that the red coloration in Comber's test is due to the exchange of ferric ions for potassium ions in the thiocyanate and that exchange acidity is partly the result of the presence of exchangeable iron.

A. G. POLLARD.

Influence of adsorbed ions on soil reaction. B. AARNIO (Bull. Agrogeol. Inst. Finland, 1927, No. 22; Proc. Internat. Soc. Soil Sci., 1930, 5, 105–106).—Clays were saturated with various cations by treatment with solutions of the appropriate chlorides and, after washing, the p_H values of the suspensions were determined. Normal soils saturated with naturally occurring cations had neutral or weakly alkaline reactions. The effect of the uni- and bi-valent cations on the p_H value was in the order of their hydration values. The hydrogen- and aluminium-clays were much more acidic. Treatment of acid soils with chlorides reduced their p_H values in every case, the adsorbed hydrogen and aluminium being exchanged and the reaction altered accordingly. In this respect the effect of univalent ions is greater than that of bivalent ions. Extreme alkalinity and acidity in soils is caused by the presence of carbonates (alkali soils) and of hydrolysable iron and aluminium salts,

respectively. Adsorbed hydrogen ions can be removed from clays only by base exchange and not by leaching with water.

A. G. POLLARD.

Effect of various manganese compounds on the quinhydrone electrode. S. OSUGI and H. KASHIMURA (J. Sci. Soil and Manure, 1929, 3, 4; Proc. Internat. Soc. Soil Sci., 1930, 5, 104).—The presence of oxides of manganese (MnO_2 and Mn_2O_4) in small proportions (0.001% and 0.005%, respectively) caused inaccuracies in p_H measurements with the quinhydrone electrode. In soils known to contain manganese, however, no significant differences in p_H values as determined by hydrogen and quinhydrone electrodes were observed.

A. G. POLLARD.

Use of barium sulphate for clarifying soil suspensions, with particular reference to colorimetric p_H determinations. L. D. BAYER and C. J. REHLING (Ind. Eng. Chem. [Anal.], 1930, 2, 338).—Experiments on 19 different types of soils showed that pure barium sulphate containing no soluble barium salts can be used for clarifying soil suspensions without materially affecting their p_H values. The amount of barium sulphate required depends on the texture and the degree of dispersion of the soil. Clays and silt loams require more than sandy soils.

E. S. HEDGES.

Report of Committee on soil reaction measurements. I. D. J. HISSINK (Soil Res., 1930, 2, 77–139).—Detailed results of comparative trials of the quinhydrone electrode method are recorded.

A. G. POLLARD.

Nitrite production in soils. G. S. FRAPS and A. J. STERGES (Ind. Eng. Chem., 1930, 22, 863–864).—Considerable proportions of nitrite-nitrogen (up to 360 p.p.m.) were found in certain soils and subsoils. Ammonium sulphate is oxidised to nitrites in these soils, but no nitrate is formed. Addition of calcium carbonate increased nitrite production. Nitrates and nitrites can be extracted from soils with water containing calcium hydroxide and the extract remains stable for at least 7 days.

A. G. POLLARD.

Adsorption of nitrates by Kuban chernozem. P. A. KURCHATOV and E. S. KRAMAROVA (Nauch. Agron. Zhur., 1925, 6, 329–325).—The capacity for adsorption of nitrate was higher when the soil originally contained nitrates. Lateritic soils adsorbed large amounts. The humus-free parent material of chernozem adsorbed less than the horizons with the humus.

CHEMICAL ABSTRACTS.

Fertilising effects of sodium nitrate in paddy fields. Y. KIDA (J. Sci. Agric. Soc. [Nogaku Kwai Ho], No. 245 and 246; Proc. Internat. Soc. Soil Sci., 1930, 5, 130–134).—Comparison is made of the efficiencies of ammonium sulphate and sodium nitrate used alone and mixed with varying proportions of oil cake.

A. G. POLLARD.

[Fertiliser trials at Rothamsted.] (Rothamsted Expt. Sta. Rept. for 1929, 125 pp.).—Efficiency of phosphate fertilisers on grass land is in the order superphosphate > high-solubility basic slag > low-solubility basic slag > mineral phosphate. The high-solubility fertilisers increased the proportion of phosphate in the herbage. Superphosphate increased the potato crop

provided no nitrogen was given, but decreased it in the presence of nitrogen and potash. In cooking tests the colour of potatoes which had received potash fertilisers was superior. In this respect the chloride gave best results, followed, in order, by the sulphate and potash manure salts. In flavour only the best of the potatoes fertilised with potash equalled that of the unfertilised, all others were inferior. Complete fertilisers gave best results with sugar beet, and these were still further improved by dressings of salt. With barley, cyanamide and ammonium chloride again gave as good results as ammonium sulphate, though still inferior to sodium nitrate. Potash reduced the attack of *Fusarium culmorum*. In pot culture chlorides delayed the ripening of barley straw and gave a lower dry weight yield than sulphates. The highest-quality barley was obtained with ammonium chloride and urea. No harm resulted from sowing up to 2 cwt. of cyanamide per acre with the seed, but 4 cwt. per acre was distinctly injurious.

A. G. POLLARD.

Influence of liming on the dynamics of soil processes. N. P. REMEZOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 65, 3—58).—Liming increases the citrate-soluble phosphorus and the nitrates; it is more effective in combination with barnyard manure.

CHEMICAL ABSTRACTS.

Influence of varying amounts of phosphates on the dynamics of soil processes. P. N. KOSHELOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 65, 59—89).—The yield and phosphorus content of plants, and the soil nitrate, were increased by application of raw phosphate.

CHEMICAL ABSTRACTS.

Tomato, a sensitive and rapid indicator of phosphate deficiency in soils. L. MAYER (Forts. Landw., 1929, 4, 684; Bied. Zentr., 1930, 59, 348—353).—Tomato seedlings grown in test soils artificially supplied with sufficient nitrogen and potash indicate phosphate deficiency after 8—14 days, *i.e.*, when the first pair of true leaves have developed, by (1) the upward inclination of the seed leaves forming an acute angle, (2) the first pair of true leaves remaining unparted at the tips, (3) the dark green colourings of the upper side and the violet-red colour of the undersides of both true and seed leaves. By allowing plants to continue growth for 2—3 months, the relative phosphate contents of soils may be compared on the basis of the weights of the plants. Exhaustion of phosphate is marked by cessation of growth and the colouring of the undersides of the leaves. Results so obtained agree well with those of Neubauer's and of Wrangell's extraction methods.

A. G. POLLARD.

Effect of soil type on the availability of finely-ground rock phosphate. P. O. RIPLEY (Sci. Agric., 1930, 10, 678—689).—Pot trials with barley grown in several soil types are recorded. In all cases finely-ground rock phosphate proved decidedly inferior to superphosphate. Plants can absorb phosphate from soils in which water-soluble phosphorus cannot be detected by customary methods. The growth of plants is not directly proportional to the accumulation of phosphorus in the sap.

A. G. POLLARD.

[Conversion of crude phosphates into concen-

trated fertilisers]. S. I. VOLFKOVICH and V. P. KAMZOLKIN [with others] (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 67, 8—134).—Optimal conditions of fineness, temperature, time of reaction, and concentration of sulphuric acid were determined for phosphates from various deposits. Factory tests of the manufacture of precipitated calcium phosphate are also described.

CHEMICAL ABSTRACTS.

Utilisation of natural aluminium phosphate. Y. KIDA (Proc. Pan-Pacific Sci. Congr.; Proc. Internat. Soc. Soil Sci., 1930, 25, 137—138).—Methods for converting natural aluminium phosphate into valuable fertilisers are described. The mineral may be decomposed by sulphuric acid and neutralised with ammonia, lime, or tricalcium phosphate. Alternatively, sodium phosphate may be obtained by treating the mineral with hot sodium hydroxide solution. Products of fusion processes, although sometimes suitably soluble, were usually hygroscopic.

A. G. POLLARD.

Effect of peat and phosphorite on the yield and composition of summer wheat and vetch. O. K. KEDROW-SICHMAN (Prazy Gory-Garezkaga Nauk. Tavar., 1928, 5, 86—104; Chem. Zentr., 1930, i, 1200).—The combined fertilisers increase the yield more than does either alone.

A. A. ELDRIDGE.

Nitrogen content and "quality" of barley. L. R. BISHOP (J. Inst. Brew., 1930, 36, 352—364).—From the nitrogen content and 1000-corn weight of the barley, the extract which will be obtained with given malting conditions and varieties can be predicted. In addition, the calculation can be made of the amount of permanently soluble nitrogen which will be found in the resulting wort. The nitrogen content is affected chiefly by the soil and season, whilst the influence of variety and artificial manuring is less important. High yields and correspondingly low nitrogen contents are determined on clay soils by drought in March–April and cold weather in May–June, whilst rain and cool weather in July are beneficial. With light soils the rainfall effects are slightly earlier. The presence of organic nitrogenous matter produces excessive nitrification later in the season, which leads to a high nitrogen content and a low yield. Artificial nitrogenous manures applied in moderation in spring produce an approximately proportional increase of yield to the nitrogen absorbed, so that the nitrogen percentage is not markedly affected.

C. RANKEN.

Phosphoric acid of barley grain. E. M. CROWTHER (J. Inst. Brew., 1930, 36, 349—351).—The content of phosphoric acid in barley was reduced by the addition of a nitrogenous manure, but was not related to the yield, barley valuation, malt valuation, nitrogen content, 1000-corn weight, diastatic power, cold-water extract, and extract calculated on barley. The different varieties of barley showed a considerable variation in the content of phosphoric acid, which was higher in the winter than in the spring barleys.

C. RANKEN.

Effectiveness of iodine in the control of smut on oats. G. M. KARNS (Ind. Eng. Chem., 1930, 22, 864).—Seed infested with smut was treated with a concentrated solution of iodine in carbon disulphide, thorough contact being established in an end-over-end shaker. With

0.5 oz. of iodine per bushel of seed, infestation was reduced to 0.3% (similar to the standard ethyl mercuric chloride treatment), and with 1 oz. of iodine per bushel only 0.04% of infested seed remained. (Cf. Sayre, Ohio Agric. Exp. Sta. Bimonthly Bull., 1928, 13, 19.)

A. G. POLLARD.

Influence of fallow methods of culture on some physico-chemical properties of plant juices from winter grain. V. V. KVASNIKOV (Nauch. Agron. Zhur., 1929, 6, 368—379).—The changes in osmotic pressure and hydrogen-ion concentration of the juices from winter rye and wheat under conditions of various methods of fallow have been followed.

CHEMICAL ABSTRACTS.

Influence of ammonium phosphate on yield and chemical composition of meadow hay. H. O. ASKEW (New Zealand J. Sci. Tech., 1930, 12, 21—23).—Ammonium phosphate was applied to two soils, a light gravelly loam, well supplied with organic matter, which did not normally respond to phosphates, and to a loam poor in lime and phosphate. Significant increases of yield of hay resulted in both cases, but analysis of the hay indicated that the utilisation of the nitrogen was much higher than that of the phosphoric acid of the fertiliser.

E. HOLMES.

Influence of phosphoric acid on the cropping power of seed potatoes. DENSCH (Superphosphat, 1930, 6, 120—123).—Phosphate deficiency in soil not only reduces the crop yield of potatoes but lowers the seed value of the tubers, which in the subsequent season produce smaller crops of lower starch content. Moreover, the smaller crops in the second season are not improved by phosphate manuring. A. G. POLLARD.

Action of ammonium sulphate and of sodium nitrate on the yield and starch content of potatoes. O. ENGELS (Forts. Landw., 1930, 5, 97; Bied. Zentr., 1930 59, 358—359).—Ammonium sulphate produced higher crop increases and starch content of potatoes than did sodium nitrate. With each fertiliser there was an economic return.

A. G. POLLARD.

Composition of a tobacco crop that burns poorly compared with one that burns well. E. M. BAILEY and P. J. ANDERSON (Conn. Agric. Exp. Sta. Bull., 1930, No. 311, 228—233).—The latter (associated with seasons of high rainfall) contained more potassium and chlorine and less magnesium, nitrogen, phosphorus, and sulphur than the former. Of substances inert as to burning quality, the latter contained more silicon, aluminium, and iron than the former; the reverse holds for manganese.

CHEMICAL ABSTRACTS.

Manurial effects of pyro- and meta-phosphates. Y. KIDA (J. Sci. Agric. Soc. [Nogaku Kwai Ho], No. 245 and 246; Proc. Internat. Soc. Soil Sci., 1930, 5, 134—137).—Pyro- and meta-phosphates can be utilised by plants in the same way as orthophosphates. The soluble meta- and pyro-phosphates of the alkalis are the most efficient. Others examined, in order of efficiency, were calcium pyro- > magnesium meta- > magnesium pyro- > calcium meta-phosphates. Pyro- and meta-phosphates of iron and aluminium are inferior to the orthophosphates. The efficiency of these phos-

phates is closely related to their solubility in 2% citric acid solution. On heating to 210—220° monocalcium orthophosphate is converted into soluble monocalcium pyrophosphate, and at higher temperatures to the insoluble metaphosphate. Field trials with heated orthophosphate show a correspondingly decreased efficiency as the temperature of heating exceeds 220°. The fertilising efficiency of superphosphate diminishes when heated above 170—180°. This corresponds to a decrease in water solubility and an increase in ammonium citrate solubility of the phosphate at this temperature. Above 210° the proportion of insoluble phosphate increases steadily.

A. G. POLLARD.

Ammonia fixation and nitrogen losses in manuring with liquid manure. S. T. JENSEN (Wiss. Archiv f. Landw., 1930, 3, 161; Bied. Zentr., 1930, 59, 359—360).—To minimise nitrogen losses, liquid manure should be applied to soil when in an open and absorbent condition, in cool quiet weather. Addition of calcium chloride or nitrate to the liquor reduced nitrogen losses by 90%. Gypsum, if used, must be well stirred with the liquor to ensure complete decomposition. Analyses of, and field trials with, the treated liquor confirm the value of the conservation process. A. G. POLLARD.

Influence of surface configuration, climate, and vegetation on the range of soil types in Chile. A. MATTHEI (Soil Res., 1930, 2, 57—67).

Critical considerations of fertiliser practice and crop yields in German agriculture. ALLEMEYER (Superphosphat, 1930, 6, 115—120).

Superphosphate. SHOJI and others.—See VII.

PATENTS.

Production of chemical fertiliser. A. HOLZ and T. VAN D. BERDELL (B.P. 332,750, 30.7.29).—Crude phosphates are treated with sulphuric acid to yield calcium sulphate and phosphoric acid; the calcium sulphate is converted by treatment with ammonium (or potassium) carbonate into the corresponding sulphate and calcium carbonate; and the phosphoric acid is neutralised with part of the calcium carbonate and some calcium hydroxide to yield dicalcium phosphate which, after removal from the mother-liquor, is mixed with concentrated ammonium (or potassium) sulphate solution, and the mixture is crushed and dried.

L. A. COLES.

Chemical fertilisers. SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 332,864, 22.11.29. Fr., 23.11.28).—Solutions containing sodium phosphate or the mixture obtained by the double decomposition of sodium phosphate and potassium chloride, with or without the addition of, e.g., sodium nitrate, are treated with ammonia and carbon dioxide to replace the sodium by ammonium with the simultaneous precipitation of sodium bicarbonate; after filtration, the solution is evaporated to yield a mixed fertiliser. The sodium bicarbonate, or the carbonate obtained by calcining it, is used to prepare sodium phosphate from monocalcium phosphate prepared from crude phosphates by the usual method. (Cf. B.P. 331,451; following).

L. A. COLES.

Manufacture of compound fertilisers and of sodium bicarbonate. SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 331,451, 7.10.29. Fr., 20.11.28).—A mixture of potassium chloride and sodium nitrate in any desired proportion is treated with ammonia and carbon dioxide as in the Solvay process, the precipitated sodium hydrogen carbonate is removed, and the solution evaporated to obtain a mixed fertiliser containing potassium and ammonium nitrates and chlorides. A. R. POWELL.

Manufacture of a concentrated fertiliser material. W. W. TRIGGS. From ARMOUR FERTILIZER WORKS (B.P. 332,639, 25.4.29).—Phosphatic material which, to increase the proportion of available phosphates, has been calcined in the presence of alkali salts and, if desired, silica and carbonaceous material, is treated with ammonia, water, and carbon dioxide to yield ammonium phosphate, alkali phosphates, and calcium carbonate; after filtration of the solution, the phosphates are recovered together by evaporation for use as a fertiliser or ammonium phosphate may be separated, *e.g.*, by fractional crystallisation. The process may be accelerated by adding finely-divided clay, feldspar, etc. to the calcined phosphates, and the constituents may be so selected that the insoluble residues are suitable for the manufacture of cement. L. A. COLES.

Simultaneous preparation of fertilisers containing magnesium phosphate and of nitrogenous fertilisers. F. UHDE (B.P. 332,803, 5.9.29. Ger., 25.10.28).—Solutions of crude phosphates in nitric acid are treated with an alkali or ammonium sulphate, which may be added prior to dissolution, and with sufficient magnesium chloride solution (*e.g.*, residual potash lyes) to convert the phosphoric acid into di- or tri-magnesium phosphate, or, alternatively, the alkali or ammonium sulphate may be partly or completely replaced by magnesium sulphate; after removal of the calcium sulphate, the solution is treated with ammonium or an alkali hydroxide or carbonate to precipitate the magnesium phosphate and to yield a solution which, on evaporation, yields a nitrogenous fertiliser. The calcium sulphate is converted into ammonium sulphate for re-use in the process and into calcium carbonate which, if desired, can be mixed with the fertiliser. L. A. COLES.

Manufacture of phospho-nitrogenous manures. SOC. D'ETUDES SCIENTIFIQUES ET D'ENTREPRISES INDUSTRIELLES, Assees. of E. VOITURON (B.P. 313,612, 14.6.29. Ger., 15.6.28).—The acid product obtained by treatment of phosphate rock with nitric acid or with the nitrogen oxides formed by the catalytic oxidation of ammonia is mixed with an alkali or alkaline-earth silicate, *e.g.*, a metallurgical slag, sufficient to neutralise the free acidity. The product is non-hygroscopic and non-corrosive to the usual containers and contains silica in an active form which improves the assimilation of phosphorus and nitrogen by the plant. A. R. POWELL.

Insecticides and fungicides. J. STODDART (B.P. 332,638, 25.4.29).—A mixture of finely-divided kaolin with 10% or less of sulphur is heated to vaporise the sulphur and thus to deposit it as a film on the kaolin. Alternatively, a mixture of kaolin and a solution of

sulphur in, *e.g.*, carbon disulphide is heated to expel the solvent. L. A. COLES.

Disinfecting seeds and the like. (SIR) G. C. MARKS From E. I. DU PONT DE NEMOURS & Co (B.P. 330,548, 29.11.28).—Dusting powders for seed disinfection comprise an organic mercury compound, with or without sodium thiosulphate, potassium thiocyanate, etc., and 90–98% of diluent (preferably active carbon). Examples of mercury compounds are: β -ethylmercurithiol-naphthalene ($C_{10}H_7 \cdot S \cdot HgEt$), *p*-ethylmercurithiol-benzenesulphonic acid, *o*-ethylmercurithiolbenzoic acid, ethyl mercurichloride, methyl mercuri-iodide, methyl mercurisulphate, ethyl mercuriacetate. Solid alkalis, *e.g.*, sodium carbonate, magnesia, lime, may be added. C. HOLLINS.

Seed disinfectant. M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,752,424, 1.4.30. Appl., 15.4.27).—Seeds may be disinfected by dusting with a dry mixture of disinfectants (*e.g.*, mercury compounds) and a wetting-out agent (*e.g.*, soap, saponin, casein). In moist soil the disinfectant becomes evenly distributed over the seed. E. B. HUGHES.

Keeping cut flowers and the like fresh. J. Y. JOHNSON. From F. G. FARBERIND. A.-G. (B.P. 332,278, 17.4.29).—Small quantities of a silver or copper salt together with a sugar or polyhydric alcohol, and, if desired, another plant nutrient, are dissolved in the water in which the flowers are kept. The total amount of sugar and other nutrients is 10–500 times that of the metallic salt. C. RANKEN.

Manufacture of concentrated fertiliser. H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,760,990, 3.6.30. Appl., 13.3.28).—See B.P. 332,639; preceding.

XVII.—SUGARS; STARCHES; GUMS.

Steam consumption in [beet-sugar] diffusion. J. HAMOUS (Z. Zuckerind. Czechoslov., 1930, 44, 409–410).—The heat theoretically required for the diffusion process can be calculated from the heat contents of the materials entering and leaving the battery. For fresh cossettes entering at 5°, and water entering and juice leaving at 25°, the heat required is about 1760 kg.-cal. per 100 kg. of beets, which is increased to 2640–2990 kg.-cal. by radiation and other losses. This heat could be obtained by injection of 4.2–4.7 kg. of steam at 6 atm. or 158°, or 4.3–4.9 kg. at 113° after suitable compression. By insulation the radiation losses can be reduced by one half, thus lowering the consumption of steam at 158° to 3.3–3.8 kg. per 100 kg. of beets. The temperature of the juice leaving the battery should be as low as possible, consistent with efficient extraction, for it can be heated in the juice heaters more cheaply than in the battery and without dilution by injected steam. J. H. LANE.

Determination of the true sugar content of beets in factory control work. V. MARES (Z. Zuckerind. Czechoslov., 1930, 54, 442–447).—Attention is called to the error that may arise in sampling beet by means of Staněk's segment rasp, whereby too high a sucrose content may be indicated. Errors arising from the dry-

ing of the pulped material previous to analysis, or from the absorption by the interior of the root of water from the flumes, are of much less importance.

J. P. OGILVIE.

Determination of sugar in the beet. J. VONDRÁK (Z. Zuckerind. Czechoslov., 1930, 54, 499–504).—Replying to Kopecky (B., 1930, 634) the author says that error due to the drying of the pulped sample can be reduced to 0.1% or less by preparing a large sample of pulp (at least 1 kg.), and at once weighing it out for analysis.

J. P. OGILVIE.

Adsorption by insoluble calcium salts in the carbonatation of beet juice. J. DEDEK and F. DOLAK (Z. Zuckerind. Czechoslov., 1930, 54, 507–513).—Experiments on the elimination of colouring matters from solutions in which different precipitates were produced showed little effect in the case of the formation of calcium oxalate. Calcium carbonate was more effective, but the best result was obtained with calcium phosphate, which adsorbed 7–10 times as much as either of the other two precipitates.

J. P. OGILVIE.

Influence of carbonatation on the rate of filtration. V. STANĚK and K. ŠANDERA (Z. Zuckerind. Czechoslov., 1930, 54, 373–382).—The addition of about 1% of lime in the carbonatation of normal beet juice increases the rate of filtration of the scums; with 2.5–3.0% a notable improvement is effected. If juice from which the organic particles have been eliminated be carbonatated after the addition of 0.25% of lime, a cake is formed which in its filtering qualities is similar to that otherwise obtained with 1–3% of lime. Centrifugal pumps have a distinctly unfavourable effect on the rate of filtration of the carbonatated juice transported by them.

J. P. OGILVIE.

Crystallisation of grape concentrates and syrups. D. S. GLENN and W. V. CRUESS (Fruit Prod. J., 1930, 9, 248–249).—Grape syrups did not crystallise in the absence of crystals or other solid matter; addition of dextrose caused the crystallisation of dextrose and tartrates. In white syrup crystallisation appears to occur above 70° Balling; in red syrups the crystallisation point is higher.

CHEMICAL ABSTRACTS.

Charring of sugar in centrifuging. W. C. BEDDING (Arch. Suikerind. Nederl.-Indië, 1930, 38, 256).—Sugar which was grey was found under the microscope to be permeated with particles of carbon, due to the fact that it had been washed with superheated steam which had been produced from boiler-water containing sugar.

J. P. OGILVIE.

Variations in the quality of raw sugars. E. W. RICE (Facts about Sugar, 1930, 25, 290–291).—Raw cane sugars differing greatly in colour showed widely differing characteristics when examined by a number of methods. It is considered that the variations in the nature of the organic impurities cannot be due to fundamental characteristics of the original juices.

J. P. OGILVIE.

Analyses of Trinidad molasses. J. A. MACDONALD (Mem. Imperial Coll., Trinidad; Sugar Tech. Ser., No. 2).—Analyses of 20 samples of Trinidad final molasses led to the following observations. The electrometric ash calculated from the conductivity of the 5% solution

agreed within 4% of the value obtained by incineration. The ash figure is of value in judging the exhaustion of a molasses, since certain samples, though having high purities, were nevertheless exhausted, the high purity values being correlated with high ash contents and low reducing sugar contents. The apparent purity value may be quite misleading. The viscosity increases as the crop proceeds. The refractometric solids are closer to the true solids than the degree Brix. J. P. OGILVIE.

Determination of the fine grain of molasses and syrups. K. ŠANDERA and C. SAMAL (Z. Zuckerind. Czechoslov., 1930, 54, 389–394).—Using a method based on the polarisation of a thin layer of the product, the fine grain content was determined in molasses of the 1925–26 and 1929–30 campaigns, and found in every case to be less than 2%. Even heating for $\frac{1}{2}$ hr. at 110° does not always suffice for dissolving the fine grain of a molasses.

J. P. OGILVIE.

Application of polarography to sugar-factory analysis. B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1930, 54, 394–397).—Heyrovsky's polarographic method (B., 1922, 986) is especially applicable for determining the conditions under which sucrose may be decomposed in the factory or refinery, e.g., in evaporation and boiling. It also gives indications capable of differentiating between the several grades of white sugars.

J. P. OGILVIE.

Unified conductometric method for determination of ash in refinery syrups. F. W. ZERBAN and L. SATTLER (Ind. Eng. Chem. [Anal.], 1930, 2, 322–325).—The relation between electrical conductivity and chemical ash of crude sugar solutions is discussed. It is shown that though there is a fairly universal ratio for beet sugars the ratio is very variable for cane sugars. Attempts are made to find a universal formula applicable to any cane product.

E. S. HEDGES.

Analysis of refined sugars. B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1930, 44, 405–409).—Five types of Czechoslovakian refined sugars, viz., cubes, centrifugal pilé, Raffinade gries (coarse castor) Raffinademehl (fine castor), and inland crystals (large grain), representing actual market standards, were analysed by the most recent chemical and physical methods. With a few insignificant exceptions, the arrangement of the five sugars in order of purity was as given above, whether judged by polarisation (99.84–99.75), invert sugar content (0.002–0.041%), ash content (0.009–0.028%), luminescence in solution, tendency to caramelisation on heating, absorption of light, or oxygen-maximum of polarographic curves (cf. following abstract). The moisture contents ranged from 0.029 to 0.046%. Results of screening tests on the last three types are recorded.

J. H. LANE.

Influence of sugars and non-sugars on the oxygen-maximum of polarographic curves. B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1930, 54, 425–432).—The polarographic method of Heyrovsky (A., 1924, 598; 1925, 674) based on electrolysis with the dropping mercury electrode will detect not only incipient caramelisation of sugar (cf. B., 1929, 695), but also the presence of extremely minute traces of molasses. Even at a concentration of 0.00002% in 5% sucrose solutions

the depressing effect of molasses on the oxygen-maximum of the polarographic curves is detectable. The constituents of molasses mainly responsible for this effect are the colouring matters and colloids, which have a measurable action at 0.000002% concentration. Much less active are potassium hexoate, vernine, and adenine, whilst invert sugar, betaine, glutimines, leucine, and potassium formate are without action. Blueing agents in the amounts used with white sugars are also inactive. The polarographic method provides a very delicate means of comparing the qualities of refined sugars.

J. H. LANE.

Photographic estimation of foreign materials in gums and resins. E. A. GEORGI (Ind. Eng. Chem. [Anal.], 1930, 2, 331—334).—A photomicrographic method for determining dirt in gums and resins is outlined and the results are shown to be as accurate for practical purposes as those obtained by the usual gravimetric procedure, whilst the cost is less. The method is applicable to glue, gelatin, etc.

E. S. HEDGES.

Sources of error in quinhedrone p_H determination of sugar-house products. B. G. SAVINOV and E. P. KOSIEVEROVA (Nauch. Zapiski Sakh. Prom., 1930, 8, 525—543).—The quinhedrone should be recrystallised twice from alcohol, and 0.01 g. per 15 c.c. of solution is the minimal quantity which should be employed.

CHEMICAL ABSTRACTS.

PATENTS.

Dissolution of organic colloids [starch, glue, and gums]. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 307,079, 2.3.29. Ger., 2.3.28).—Carbohydrates and proteins are dissolved in an organic solvent insoluble in water with the addition of a soap dissolved in an organic solvent wholly or partly soluble in water, e.g., an alcohol, ketone, phenol, chlorohydrin, an ester, or dioxan. E.g., a potash-olein soap is dissolved in tetrahydronaphthalene, the solution is clarified with ammonia solution, and powdered glue is stirred into this solvent; the clear viscous product does not solidify.

A. R. POWELL.

Recovery of nitrogen and acetone from vinasses. J. GUILLISSEN, ASST. to UNION CHIM. BELGE (U.S.P. 1,772,078, 5.8.30. Appl., 27.8.27. Belg., 21.9.26).—See B.P. 277,932; B., 1928, 345.

XVIII.—FERMENTATION INDUSTRIES.

Composition and determination of barley proteins. III. L. R. BISHOP (J. Inst. Brew., 1930, 36, 336—349; cf. B., 1929, 696).—For each separate variety of barley the weights of the individual proteins are simple regular functions of the total nitrogen content per 1000 corns. The percentage of salt-soluble nitrogen on total nitrogen decreases with increase of the total nitrogen. The percentage of hordein nitrogen shows a corresponding increase so that the glutenin percentage remains constant throughout. The curves for the different varieties are similar in form, but they differ in actual magnitude. Accordingly, the relationship between the quantities of the individual proteins and the total nitrogen is characteristic of the variety. If they are allowed time to reach the natural equilibrium, the same proportions of the proteins are found in samples of developing grain as in mature grain of the same total

nitrogen content per 1000 corns. Development of the proteins is essentially a synthesis of the simple compounds which enter the grain, and the synthesis progresses to a definite equilibrium point which is controlled only by the total nitrogen and the variety.

C. RANKEN.

Barley proteins. Influence of method of grinding on their determination. G. HOFMAN-BANG (J. Inst. Brew., 1930, 36, 381—388).—When the barley is ground in a ball-mill with the production of 95% of flour, as measured by a Pfungstädter sieve, the percentage amounts of salt-soluble protein, hordein, and glutenin reach constant values which are unaffected by the heating effect in the ball-mill.

C. RANKEN.

Numerical expression of the analytical results as basis for the calculation of the brewing value [of barley and malt]. F. KUTTER (Woch. Brau., 1930, 47, 237—241, 245—249, 261—265, 273—279, 283—285, 297—300).—Suggestions are made for assigning such definite numerical values to the results of the analytical examination of barley and malt as may be combined into an equation to give a figure for the brewing value which will vary continuously and not by stages. The optimum amount of protein is considered to be 10%, and the brewing value to fall off with increasing steepness on either side of this. It is claimed that high screening values in malts are associated with high fermentability of the resulting worts. The original must be consulted for details.

F. E. DAY.

Separation and determination of the bitter resins of hops. A. S. BAZILEVICH (Nauch. Agron. Zhur., 1929, 6, 415—428).—Resins precipitable with lead acetate are termed "humulids," and others "lupulids." The former are grouped in four, and the latter in three, classes. The hops (10 g.) are shaken for 1.5 hrs. with ether (200 c.c.), and the residue from 100 c.c. of filtrate is dried for 2—3 hrs. at 80° and weighed. The residue after repeated extraction with light petroleum consists of hard resins. For the determination of waxes methyl alcohol is employed as solvent. The amounts of various substances present are influenced by the soil and the method of fertilisation, and they vary in different parts of the plant.

CHEMICAL ABSTRACTS.

Copper as applied to brewing. D. M. STEWARDSON (J. Inst. Brew., 1930, 36, 369—381).—A description is given of the application of copper to brewing plant, including mash tuns, backs, fermentation vessels, and racking machines, and it is suggested that copper and gun-metal can be used with advantage to replace iron or galvanised iron for the construction of pulp-washing machines.

C. RANKEN.

Dry, unpressed wine and natural wine. J. WERDER and C. ZÄCH (Wein u. Rebe, 1929, 11, 272—276; Chem. Zentr., 1930, i, 1394).—Dry, unpressed wine, but not natural wine, after decolorisation by animal charcoal, shows a blue luminescence in filtered ultra-violet light.

L. S. THEOBALD.

Detection of fruit wine in grape wine. C. VON DER HEIDE (Wein u. Rebe, 1929, 11, 251—271; Chem. Zentr., 1930, i, 1394).—Methods are discussed; Brauer's (phosphomolybdic acid) and Seyda's (sodium gold

chloride) methods differentiate, whilst with the sorbitol method 5% of fruit wine in grape wine can be detected.

A. A. ELDRIDGE.

Detection of fruit wine in grape wine by Werder's sorbitol method. G. FIESSELMANN (Wein u. Rebe, 1929, 11, 317—326; Chem. Zentr., 1930, i, 1394).

Barley. BISHOP; CROWTHER.—See XVI.

PATENTS.

Manufacture of yeast. G. S. BRATTON, ASSR. to ANHEUSER-BUSCH, INC. (U.S.P. 1,732,922, 22.10.29. Appl., 15.4.27).—Yeast is propagated in a solution of sacchariferous materials to which is added a ureide of a reducing sugar.

C. RANKEN.

Cultivation of micro-organisms. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,235, 11.3.29).—Peat is completely hydrolised by acids, and prior to the inoculation of the resulting solution the humous substances are removed by treating it with non-alkaline adsorbent substances of large superficial area, the pH being kept within the range 5—8. (Cf. B.P. 318,649; B., 1929, 994.)

C. RANKEN.

XIX.—FOODS.

Measuring the relative plasticity of pastes and doughs. C. H. BAILEY (J. Rheology, 1930, 1, 429—432). The work-input required to move the blades of a small mixing machine through the material is measured by inserting a watt-hour meter in the motor circuit. The values recorded are particularly useful in the bread-making industry for determining the effect of unit additions of water on the consistency of the dough.

E. S. HEDGES.

Composition and food value of carob-bean meal and "patay." C. T. RIETTI (Rev. farm. Buenos Aires, 1929, 71, 633—635; 1930, 72, 3—16).—The meal of *Prosopis alba*, Griesebach, and cake contain, respectively, water 6.67, 9.67; ash 2.70, 6.71; reducing carbohydrates 3.20, 5.70; carbohydrates hydrolysable and soluble in the cold 41.20, 43.98; starch 8.01, 10.40; fibre 10.0, 5.93; pentosans 4.41, 3.49; protein 6.56, 4.37%.

CHEMICAL ABSTRACTS.

Composition of cottonseed hull bran. K. S. MARKLEY (J. Amer. Soc. Agron., 1928, 20, 1102—1107).—Comprehensive analytical values are recorded.

CHEMICAL ABSTRACTS.

Determination of age of eggs by the aid of hydrogen-ion concentration measurements. C. SCHWEIZER (Mitt. Lebensm. Hyg., 1929, 20, 312—313; Chem. Zentr., 1930, i, 1240).—The method is untrustworthy.

A. A. ELDRIDGE.

Determination of crude fibre in cocoa and chocolate. J. RUFFY (Mitt. Lebensm. Hyg., 1929, 20, 355; Chem. Zentr., 1930, i, 1239).—Matthes and Müller's modification of König's method is preferred to Fellenberg's method but the use of smaller quantities is recommended.

A. A. ELDRIDGE.

Vitamin content of ethylene-treated and untreated tomatoes. D. D. JONES and E. M. NELSON (Amer. J. Publ. Health, 1930, 20, 387—394).—Naturally ripened tomatoes are a better source of vitamins-A, -B, and -C than is fruit picked while green and treated with ethylene to develop the red colour, but treatment with

ethylene did not appear to affect vitamins already formed.

CHEMICAL ABSTRACTS.

PATENTS.

Processing cereal grains. J. W. BECKMAN (U.S.P. 1,753,643, 8.4.30. Appl., 20.3.29).—Cereal grains are treated with alkali and bleaching agent to remove the fat from the outer layer and bleach and sterilise the grain, which is afterwards dried at below 60°.

E. B. HUGHES.

Preservation of milk and cream. T. HOFIUS and A. REPPMAN (B.P. 332,641, 26.4.29).—The milk etc. is de-aerated by displacing the dissolved air by carbon dioxide, the latter gas is then displaced by oxygen, and the product kept in that condition until required. Subsequently, the oxygen is displaced by nitrogen immediately before use. Suitable apparatus is described.

E. B. HUGHES.

Production of stable, homogenised, [milk- or cream-like] emulsions. C. W. A. KLEINE (B.P. 309,836, 15.4.29. Swed., 13.4.28).—The disperse-phase constituent is introduced into an emulsification machine which is already charged with the constituent which is to constitute the continuous phase. The gradual introduction of the two constituents is continued in the same order and the resulting basic emulsion is well agitated and then immediately homogenised.

C. RANKEN.

Compositions comprising carbohydrates and acid-reacting substances for incorporation with milk. G. B. KILGOUR. From F. H. FAULDING & Co., LTD. (B.P. 332,595 and 332,597, 24.4.29).—By intimately mixing small quantities of edible organic acids with relatively large quantities of dextrose and then adding the mixture (in tablet etc. form, if desired) to milk, a digestible humanised milk may be produced.

E. B. HUGHES.

Dry milk products. W. R. B. S. GATES, J. TAVROGES, and Cow & Gate, LTD. (B.P. 332,047, 3.7.29).—A laxative milk product is prepared by drying milk in which phenolphthalein has been dispersed or added in alkaline solution.

E. B. HUGHES.

Preventing rancidity in dried or powdered milk and milk foods or preparations. W. R. B. S. GATES, J. TAVROGES, and Cow & Gate, LTD. (B.P. 332,669, 14.5.29).—By the addition of a polyhydric alcohol, e.g., glycerin or various glycols, to milk before drying, a milk powder is produced in which the production of rancidity with time is largely inhibited.

E. B. HUGHES.

Preservation of artificial and natural butter. J. LONGMAN. From A.-G. F. MEDIZ. PRODUKTE (B.P. 332,806, 9.9.29).—To the butter etc. an unorganised albumin-digesting ferment, preferably in acid solution, is added. E.g., a margarine containing pepsin 0.05%, glycerin 0.06%, and 25% hydrochloric acid (0.1%) is claimed.

E. B. HUGHES.

Lard product. J. R. SHIPNER, ASSR. to CUDAHY PACKING Co. (U.S.P. 1,755,135, 15.4.30. Appl., 1.6.29).—Lard is refined to remove fatty acids, hydrogenated to an iodine value of 55—60, deodorised, and cooled to a lard-like consistency. It is claimed that the product is neutral in flavour, and is superior to ordinary lard in cooking qualities.

E. B. HUGHES.

Apparatus for extracting fat and other substances from animal carcasses, slaughterhouse refuse, fish, fish waste, etc. A. SOMMERMEYER (B.P. 332,637, 24.4.29).—In the continuously operating apparatus described, the separation vessel is arranged by the side of the digestion vessel, a feed pipe connecting the two. A steam and vapour separator is provided in this pipe, and the products condensed are conducted to the separation vessel. E. B. HUGHES.

Removing lead arsenate from fruit. A. R. MAAS (U.S.P. 1,754,173, 8.4.30. Appl., 21.12.26).—The fruit is washed or sprayed with a solution of an alkali thio-sulphate, with soap or alkali as a wetting agent. E. B. HUGHES.

Preservation of food products. E. MILANI (B.P. 332,209, 16.2.29).—Fresh fruit or vegetables are hermetically sealed in the presence of an inert gas in a container which is minutely vented before, or very soon after, the product generates its own gas. C. RANKEN.

Manufacture of a [pectin-sugar] jelly preparation. A. LEO (B.P. 331,295, 15.4.29).—Sugar crystals maintained in suspension by an upward current of warm air are sprayed with a 3–5% pectin solution, whereby they become coated with a thin film of pectin. Powdered citric or tartaric acid is coated with a thin film of paraffin wax or palmitic or stearic acid by moistening it with an ethereal solution of the wax or fatty acid. The sugar-pectin powder is then mixed in desired proportion with the acid powder. To prepare a jelly the powder is added to cold fruit juice, whereby the pectin dissolves followed by the sugar; the solution is then heated to melt the film on the acid particles and to cause them to dissolve. On cooling, the mass sets to a jelly. A. R. POWELL.

Heat-exchanger (U.S.P. 1,754,857). **Treating materials** (B.P. 332,577 and 332,593).—See I.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Acid in ether. HALL.—See III. **Tobacco crops.** BAILEY and ANDERSON.—See XVI.

PATENTS.

Manufacture of substituted ureas and thioureas. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,583, 10.1.29).—Substances of the "Bayer 205" type (cf. B.P. 9472 of 1914; B., 1914, 825), but containing one or more heterocyclic residues, have similar destructive action on blood parasites. The condensation product from 1 : 4 : 6 : 8-naphthylaminetrisulphonic acid and 4-chloro-6-nitroquinazoline is reduced; the amine so obtained is treated with 2-nitro-*p*-toluyl chloride, and the product is reduced and phosgenated; or the amine may be condensed with a second mol. of 4-chloro-6-nitroquinazoline, reduced, and phosgenated; or the amine may be phosgenated directly. Similarly 2 : 4 : 6 : 8-naphthylaminetrisulphonic acid is *m*-nitrobenzoylated, the reduced product is condensed with 10(*ms*)-chloro-1-nitroacridine, reduced, and phosgenated. C. HOLLINS.

Manufacture of salts of higher homologues of polyhydroxybenzenes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,519, 6.3.29).—The causticity or unpleasant taste of octyl- and higher alkyl-

resorcinols etc. is removed by their conversion into salts with alkalis or organic bases. The hexamethylene-tetramine salts of 4-octylresorcinol, decomp. 118°, 4-decylresorcinol, decomp. 120°, 3-decylpyrocatechol, and decylpyrogallol, decomp. 140°, and the piperazine, anhydrosarcosine, and betaine salts of decylresorcinol (the last two having m.p. 76° and 80°, respectively), are described. Sodium decylresorcinol is salted out by calcium chloride solution. C. HOLLINS.

Manufacture of 3 : 3'-dichloro-5 : 5'-diacetamido-4 : 4'-dihydroxyarsenobenzene. I. G. FARBENIND. A.-G. (B.P. 330,862, 25.10.29. Ger., 25.10.28. Addn. to B.P. 296,327; B., 1928, 837).—3 : 3'-Dichloro-5 : 5'-diamino-4 : 4'-dihydroxyarsenobenzene is acetylated by addition of acetic acid to an aqueous solution of the sodium salt at 15–35°. C. HOLLINS.

Antiseptic composition. V. LEONARD, ASSR. to SHARPE & DOHNE, INC. (U.S.P. 1,771,895, 29.7.30. Appl., 19.1.28).—See B.P. 299,522; B., 1928, 944.

Manufacture of aromatic arsenic compounds containing an isoxazine ring. G. NEWBERRY, ASSR. to MAY & BAKER, LTD. (U.S.P. 1,771,307, 22.7.30. Appl., 27.6.27. U.K., 8.7.26).—See B.P. 280,613; B., 1928, 211.

Chlorine (U.S.P. 1,729,043).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Optical and photographic properties of sensitising and desensitising dyes of the cyanine and related types. O. BLOCH and F. M. HAMER (Phot. J., 1930, 70, 374–390).—The absorption and sensitising curves of dyes of the ψ - and *iso*-cyanine type derived from a number of heterocyclic ammonium iodides, and of corresponding *p*-dimethylaminostyryl and *p*-dimethylaminocinnamylidene derivatives, and *p*-dimethylaminoanils are determined and compared (cf. A., 1930, 930). In general, the absorption bands of the styryl compounds are nearer the blue end of the spectrum than are those of the corresponding anils; in both cases they are usually single and broad in contrast to the narrow double bands of the cyanines. All the cyanines are photographic sensitisers; the styryl compounds with one exception are much weaker, and the two cinnamylidene derivatives have but little photographic activity. The anils are powerful desensitisers, comparable with, and in most cases superior to, safranin, but are not recommended for technical application as they give decomposition products which attack the latent image (cf. Hamer, B., 1929, 1032). The following are prepared by interaction of the appropriate quaternary salt with *p*-dimethylaminobenzaldehyde, *p*-dimethylaminocinnamaldehyde, or *p*-nitrosodimethylaniline in alcoholic solution in the presence of piperidine: 4-*p*-dimethylaminostyrylquinoline ethiodide, m.p. about 124°; tetramethyl-*pp'*-diamino-2 : 4-distyrylquinoline ethiodide, m.p. 243° (decomp.); 4-methyl-2-*p*-dimethylaminostyrylthiazole ethiodide, m.p. 249° (decomp.); 1-*p*-dimethylaminostyrylbenzthiazole ethiodide, m.p. 255–257° (decomp.); 2-*p*-dimethylaminostyryl-3 : 3-dimethylindolenine ethiodide, m.p. 222° (decomp.); 1-*p*-dimethylaminostyrylbenzoxazole ethiodide, red and blue dichroic crystals, m.p. 248–249° (decomp.); *p*-dimethylaminocinnamylidene- α -picoline

ethiodide, m.p. 209°; 1-*p*-dimethylaminocinnamylidene-methylbenzthiazole *ethiodide*, m.p. 220°; the *p*-dimethylaminoanils of β -naphthaquinaldinealdehyde *ethiodide*, m.p. 222° (decomp.), quinoline-4-aldehyde *ethiodide*, m.p. 174°, 4-methylthiazole-2-aldehyde *ethiodide*, m.p. 227° (decomp.), and of benzthiazole-1-aldehyde *ethiodide*, m.p. 240°; and the di-*p*-dimethylaminoanil of quinoline-2:4-dialdehyde *ethiodide*, m.p. 217° (decomp.). H. A. PIGGOTT.

"Halogen value" of silver iodide emulsions, its determination and photographic interpretation. H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 23, 111—117).—Silver iodide emulsions were prepared by different methods in excess of potassium iodide, thoroughly washed, and then broken down with dilute nitric acid. In all cases, contrary to results obtained with the other silver halides, iodide ions, but no silver ions, were detected in the filtrate, the precipitate being free from silver ions. Emulsions prepared in presence of excess of silver ions gave, after breakdown with nitric acid, a filtrate containing appreciable amounts of silver ions. The amount of iodide in the filtrate depends on the type of emulsion and particularly on the grain size, and appears to be inversely proportional to the latter. Some of the known properties of silver iodide emulsions, *e.g.*, sensitisation with dyes, are discussed in the light of these results.

J. W. GLASSETT.

Nomenclature of "silver and halogen values" in photographic layers. H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 28, 118—120).—A system of nomenclature, based mainly on the authors' work, is suggested for the characterisation of photographic layers.

J. W. GLASSETT.

Solarisation. I. Effect of bromide ions on solarisation. II. Dependence of solarisation on method of preparation of emulsion. H. ARENS (Z. wiss. Phot., 1930, 28, 91—98, 98—110).—I. Experiments on a series of commercial plates and films showed that bromide ions were effective in increasing solarisation only when present during exposure. When the bromide ions were removed by washing in silver nitrate, followed by sodium chloride to destroy excess of silver ions, no solarisation was exhibited. Similarly treated layers of peptised silver bromide showed a very much reduced solarisation. The effect, however, reappeared when the treated plates were bathed in potassium bromide solution. The inhibition of solarisation exhibited by nitrate ions is destroyed in presence of bromide ions.

II. The solarisation properties of various silver bromide layers prepared from silver bromide precipitated in presence of excess of silver, equivalent proportions, and excess of bromide, and afterwards peptised in gelatin solutions with varying additions of potassium bromide, have been investigated. The silver body solarised less than the bromide body even if equal quantities of potassium bromide were present in the finished emulsion. Contrary to previous experiments, increased quantities of potassium bromide at peptisation decreased the solarisation, the minimum of the curve of the solarisation range being raised. Furthermore, an increase in bromide content caused a displacement of the solarisation range towards the shorter exposures,

whilst the sensitivity of the foot of the curve was decreased. The difference in solarisation of the silver and bromide bodies cannot be correlated with differences in grain-size distribution. The silver, equivalent, and bromide bodies peptised in presence of ammonia solarised strongly, the effect being increased with increase in ammonia content.

J. W. GLASSETT.

PATENTS.

Developing kinematograph films sensitised with dichromated gelatin. M. G. M. G. BIZOT (B.P. 332,195, 10.4.29. Fr., 11.10.28).—The impressed films are dipped for a short time first in cold water, and then in cold water containing sodium hypochlorite. They are then washed in hot water so as to remove all the unimpressed gelatin, the impressed gelatin being dyed with a dye such as methylene-blue, or so as to remove part of the unimpressed gelatin, in which case the impressed and the remainder of the unimpressed gelatin are dyed, either to a different extent or by means of different dyes.

W. J. WRIGHT.

Production of diazo-type prints. KALLE & Co. A.-G. (B.P. 316,563 and Addn. B.P. 318,108, [A] 30.7.29. Ger., 30.7.28; [B] 17.8.29. Ger., 27.8.28).—The resistance of diazo-types to water is improved, where the azo dye contains sulphonic or carboxylic groups, by adding to the layer at any convenient stage (A) an arylated diguanidine or (B) an arylated guanidine (diphenylguanidine). Examples are: diazotised 5-dimethylaminoanthranilic acid, phloroglucinol, phenyldiguanidine, tartaric and boric acids (black); diazotised *p*-aminodiethylaniline-*m*-sulphonic acid, phloroglucinol, resorcinol, 3-chloro-*o*-tolyl diguanidine or diphenyl- or di-*o*-tolyl-guanidine, tartaric and boric acids (black).

C. HOLLINS.

Films [with filter mask] for use in colour photographic processes. KODAK, LTD., Assees. of P. FAVOUR (B.P. 318,238, 15.7.29. U.S., 31.8.28).

Paper (B.P. 332,743).—See V. Photochemical dyeing etc. (B.P. 309,166).—See VI. Imitating wood-grain (B.P. 308,371 and 308,373—6).—See IX. Printing plates (B.P. 331,220).—See X.

XXII.—EXPLOSIVES; MATCHES.

Nitrostarch [explosives]. A. SCHRIMPF (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 273—278).—Two kinds of nitrostarch explosives are used in America. For one kind, nitrostarch is mixed with sodium nitrate and an oil, a typical composition consisting of nitrostarch 50%, sodium nitrate 47.5%, impregnating oil 1.5%, and sodium bicarbonate 1.0%. This explosive has a high brisance and is readily detonated; it is suitable for chamber blasts and quarrying limestone or granite. The other type contains a large amount of ammonium nitrate mixed with the nitrostarch, together with oxidisable materials, such as coal dust, trinitrotoluene, and aluminium, and a small amount of oil. A representative explosive consists of ammonium nitrate 73.0%, nitrostarch 15.0%, trinitrotoluene 3.0%, coal dust 2.0%, aluminium 6%, mineral oil 0.5%, and zinc oxide 0.5%. Explosives of this type have a lower brisance and are more difficult to detonate; they also give rise to larger amounts of gaseous products. Some

of these compositions are used in fiery mines. Investigations on the sensitising of ammonium nitrate by nitro-starch, made by the Pittsburgh experimental station, are described. W. J. WRIGHT.

Universal explosive. A. STETTbacher (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 273—279).—"Penthrinit," consisting of mixtures of pentaerythritol tetranitrate (penthrinit) and nitroglycerin or dinitroglycerol in the proportions of 10—70% and 90—30%, respectively, is superior as regards efficiency and detonation velocity to blasting gelatin. In order to obtain uniform distribution of the penthrinit, about 1% of collodion cotton is added. Both normal and gelatinised penthrinit can be moderated by means of camphor or centralite. Mixtures of 80:20 or 50:50 penthrinit with collodion cotton and camphor or centralite are less sensitive to shock, as tested by the falling weight, than is penthrinit alone, and the latter is less sensitive than penthrinit. Penthrinit suffers no loss of efficiency or reduction in detonation velocity on storage. "Ammonium penthrinit," consisting of penthrinit 40%, nitroglycerin 10%, vaseline 1.7%, and ammonium nitrate 48.3%, equally retains its original strength. W. J. WRIGHT.

Determination of centralite in double-base, smokeless powders. H. LEVENSON (Ind. Eng. Chem. [Anal.], 1930, 2, 246—247).—The centralite (diethyl-diphenylurea) in smokeless powders made from nitro-cellulose and nitroglycerin may be determined directly by the following method. 5 G. of the powder are extracted with ether for 24 hrs., and the extract is evaporated. The liquid residue is transferred to a 250-c.c. flask with 50 c.c. of alcohol, 25 c.c. of standard bromide-bromate solution are added, the mixture is cooled to 20°, and 5 c.c. of hydrochloric acid are added. The flask is stoppered and the contents are shaken, and exactly 30 sec. from the time of adding the acid 10 c.c. of 15% potassium iodide are added. The liberated iodine is titrated with sodium thiosulphate. The bromide-bromate solution should contain 5.568 g. of potassium bromate and 30 g. of potassium bromide in 1 litre and should be standardised in the presence of alcohol and 30 sec. after adding the acid as described above. E. S. HEDGES.

Action of volatile solvents on cellulose nitrate powders. J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 149—150).—Disintegration, due chiefly to surface denitration, is greater with powders prepared with volatile solvents. CHEMICAL ABSTRACTS.

PATENTS.

Manufacture of priming compositions. H. RATHSBURG and E. VON HERZ (B.P. 310,509, 18.3.29).—Priming compositions containing lead styphnate are sensitised by addition of guanynitrosoaminoguanyl-tetrazene, the amount of this latter compound being 0.5—15% of the total composition. W. J. WRIGHT.

Method of loading explosives. F. OLSEN and C. J. BAIN (U.S.P. 1,752,391, 1.4.30. Appl., 14.6.29).—In filling projectiles or moulds with mixtures of ammonium nitrate and an explosive such as trinitrotoluene, a predetermined amount of the latter, after melting, is first poured in, and pellets of ammonium nitrate larger than 10-mesh are added until the liquid just covers them. The

unheated pellets absorb heat from the molten explosive, and thereby accelerate the setting of the mixture.

W. J. WRIGHT.

Manufacture of smokeless powder. C. R. BORLAND, ASSR. to AMER. POWDER CO. (U.S.P. 1,752,881, 1.4.30. Appl., 1.3.24).—In the process of hardening grains of nitrocotton for smokeless powders by means of a liquid solvent, the latter is introduced as a spray of such fineness that the particles of the solvent have a smaller volume than the grains of the nitrocotton. A special type of atomiser is used. The granular mass of nitrocotton is caused to move in such a manner as to uncover all the grains successively and to remove those already treated on the surface. W. J. WRIGHT.

Manufacture of a tetrazene explosive. H. RATHSBURG (B.P. 308,179, 18.3.29. Ger., 16.3.28).—The preparation of tetrazene by treating an aqueous solution of an aminoguanidine salt, such as the nitrate, with sodium nitrite solution is accelerated, and the yield is increased if the reacting solutions are heated to 25—30° before being mixed. During mixing, the temperature is allowed to reach 40—50° by suitably insulating the reaction vessel, and the reaction is controlled by cooling the vessel. W. J. WRIGHT.

Explosive and solvent therefor. T. L. DAVIS (U.S.P. 1,754,417, 15.4.30. Appl., 8.11.27).—Aliphatic nitroamines are solvents for nitroguanidine, having the property of converting it into a non-crystalline mass. Being soluble in water and organic solvents, they may be used in the preparation of nitrocellulose-nitroguanidine explosives, and as they contain a nitro-group they increase the power of such explosives. W. J. WRIGHT.

Manufacture of [strip] matches. J. G. NEWMAN (B.P. 332,700, 17.6.29).

XXIII.—SANITATION; WATER PURIFICATION.

Industrial analysis and recording of carbon dioxide and oxygen in air. W. F. HAMILTON (Ind. Eng. Chem. [Anal.], 1930, 2, 233—237).—The quantitative analysis of gaseous mixtures by measurements of thermal conductivity of the gases is discussed, and the application of this method has led to the construction of an apparatus for the continuous recording of the percentages of carbon dioxide and oxygen in air. Quartz-protected platinum spirals are used both for heating elements and for the measurement of temperature. An improved thermal conductivity gas-analysis unit is described, and the effects of varying gas composition, temperature, current, and flow rate are indicated. The time lag is shown to be constant and independent of the magnitude of change of composition of the gas, providing a constant rate of flow is maintained. The successful operation of the apparatus depends on the maintenance of a Wheatstone bridge circuit which is only slightly unbalanced and the adjustment of the cells to a point where the rates of heat dissipation from the reference and analysis spirals are nearly equal. E. S. HEDGES.

PATENTS.

Purification of water. O. and R. ADLER (U.S.P. 1,771,518, 29.7.30. Appl., 9.7.26. Czech., 15.7.25).—See B.P. 286,338; B., 1928, 350.

Water softener (U.S.P. 1,752,789). Filtration apparatus (U.S.P. 1,754,667).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCT. 10 and 17, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Rapid countercurrent mixer. L. ZAKARIAS (Chem.-Ztg., 1930, 54, 607—608).—The advantages of the Eirich mixer (cf. Beton u. Eisen, 1928, No. 4) for the cement, concrete, and colour industries are confirmed; it can also be used for dough-like materials and in the manufacture of bread, chocolate, cosmetics, etc.

E. LEWKOWITSCH.

Continuous "unit" rotary filters in the chemical industry. C. ALEXI (Chem. Fabr., 1930, 321—322).—A rotary drum filter of the type in which the slime is sucked against the periphery at one quadrant, dried in the next two and removed, and the filter surface then washed before it re-enters the filtering zone is described and illustrated.

A. R. POWELL.

Use of revolving tubes for crystallisation and concentration. M. EVÉQUOZ (Giorn. Chim. Ind. Appl., 1930, 12, 348—352).—Descriptions and diagrams are given of: a rotating tubular crystalliser, in which saline solutions are crystallised, with continuous separation of the crystalline product; a tubular concentrator; and a tubular concentrator-crystalliser, which combines the two forms of apparatus in a single tube and permits the crystallisation of dilute saline solutions in a single phase.

T. H. POPE.

Degasification apparatus [for evaporators]. G. BRUNNS (Chem.-Ztg., 1930, 54, 629).—It would seem that, as air and carbon dioxide are heavier than steam and ammonia, connexions for the removal of the first two from steam chests should be at the bottom, and for the ammonia at the top. In practice, however, the effect of density is often neutralised by diffusion and turbulence. If "dead angles" exist, this is where the connexions should be placed. The question is of importance in relation to the corrosion of steam tubes, but the solution depends on the design and even on the method of operation of the evaporator. C. IRWIN.

Application of a modern turbine for the utilisation of steam used in the production of [ammonium] sulphate. F. REUTER (Gas- u. Wasserfach, 1930, 73, 361—364).—The possibility of using waste steam from the ammonia evaporators in a turbine for the generation of electrical current is discussed, and figures are given showing the economies thereby effected in the cost of production of ammonium sulphate from dilute ammoniacal liquors produced in a gasworks.

A. R. POWELL.

Determination of lustre. ANON. (Jahresber. VII., Chem.-Tech. Reichsanst., 1928, 220—222; Chem. Zentr., 1930, i, 1976—1977).—An apparatus and unit of measurement are described.

A. A. ELDRIDGE.

Checking flue-gas analyses. HERTZELL. Moisture loss in kilns. BLESSING. Compensator for pyrometer installations. ARNOLD.—See VIII. Fastness of colours. STERN.—See XIII. Treatment of boiler waters. BARTOW.—See XXIII.

See also A., Aug., 1014, Viscosimeter (KÄMPF and SCHRENK).

PATENTS.

Heat exchangers for abstracting heat from waste furnace gases. F. K. WOODROFFE (B.P. 333,397, 14.8.29).—The heat is transferred to both air and water simultaneously. One form of the apparatus comprises a casing set above a flue in which are dampers to divert the flue gases upwards into the exchanger and downwardly out into the same flue. The air is heated in a number of narrow casings dependant from the roof, and between the air passages are vertical rows of pipes for water. Zig-zag and generally countercurrent flow is arranged for.

B. M. VENABLES.

Heat-exchanging apparatus. HEENAN & FROUDE, LTD., and G. H. WALKER (B.P. 331,602, 10.1.30).—A tubular heat-exchanger is provided with cores consisting of metal ribbon slit and twisted, to produce turbulence of the fluid.

B. M. VENABLES.

Heat-exchange apparatus. [Radiator.] C. E. SAFFORD, Assr. to A. H. GREELEY (U.S.P. 1,753,318, 8.4.30. Appl., 12.5.25).—A deeply corrugated structure is formed by pressing and welding (or brazing) sheet metal, the form being such that the operation of pressing produces bending, but no stretching, of the metal.

B. M. VENABLES.

Generation of steam from waste industrial gases, particularly in water-gas plants. SEMET-SOLVAY ENG. CORP., Assees. of F. W. STEERE (B.P. 333,472, 13.11.29. U.S., 17.11.28).—The waste gases are passed through a heat-absorbing medium over which water is afterwards sprayed. The superheated steam produced is passed into a supply of water and produces a further volume of steam, the combined steam being saturated; the water thus heated is preferably used to supply the sprays. The apparatus forms a convenient means for generating the steam required for water-gas producers from the waste heat of the "blow" period alone.

B. M. VENABLES.

Washing waste or chimney gases, in connexion with steam-generating plants, blast furnaces, etc. T. CARTWRIGHT (B.P. 333,317, 21.5.29).—A number of inclined annular shelves and conical trays are arranged alternately and overlapping within a tower, the gas passing upwards round their inner and outer edges, respectively. Approximately horizontal water sprays

* The remainder of this set of Abstracts will appear in next week's issue.

are provided beneath the shelves, and the muddy water is exhausted through a bus pipe leading from the points of the cones. A moisture eliminator is provided at the top.

B. M. VENABLES.

Absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE GES.M.B.H., Assees. of SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 316,299, 9.7.29. Ger., 27.7.28).—In consequence of improved gas-tightness obtainable in the present-day machines, hydrogen may be used as the neutral carrier gas in the apparatus described in B.P. 229,332 (B., 1926, 519). [Stat. ref.]

B. M. VENABLES.

Vessels of the autoclave type. G. MONETA (B.P. 310,852, 1.5.29. It., 1.5.28).—A vessel, more particularly of domestic size, is formed with a mouth turned inwardly and then outwardly, presenting a channelled surface convex to the interior, against which the lid is held by the internal pressure.

B. M. VENABLES.

Comminuting mills. ALLIS-CHALMERS MANUF. Co., Assees. of R. C. NEWHOUSE (B.P. 333,061, 16.8.29. U.S., 20.12.28).—In a rotary drum mill divided transversely into at least two compartments, the material from an earlier stage of grinding is admitted to the interior of an annular screen rotating with the drum. The under-size passing the screen is delivered (*e.g.*, by scoops) to the next later compartment, and the oversize retained in the screen is moved by a conveying device to a hollow tyre, by which it is returned to the earlier compartment.

B. M. VENABLES.

Drying of granular materials. C. MOORE & Co., LTD., W. M. SHAW, and W. TRANTOM (B.P. 332,788, 5.9.29).—A number of trays or plates of stainless steel or monel metal are inclined alternately from opposite walls of a tower at an angle steeper than that of repose of the salt or other material being dried. Cams are rotated under the plates to shake them and their motion is limited by rods extending from them to outside the tower, where springs are provided.

B. M. VENABLES.

Apparatus for treating and mixing comminuted or finely-divided materials. A. B. and C. R. SMITH (B.P. 333,051, 2.8.29).—In an apparatus of the type in which rotating arms or sweeps act in conjunction with fixed or relatively moving surfaces, one of the elements is resiliently supported in such a way that it is normally rigid but can yield to exceptionally hard bodies.

B. M. VENABLES.

Centrifugal separator. E. VAN DER MOLEN (B.P. 333,452, 11.10.29).—A centrifugal separator of the straining type has the basket divided into a number of compartments into which the feed is admitted in sequence, and from which the collected solids are removed by a scraper which enters each one in turn without slowing the machine or affecting the operation of the other compartments.

B. M. VENABLES.

Centrifugal separation. H. S. COE (B.P. 332,993, 28.5.29).—In a centrifuge with continuous discharge of both products, an additional carrier or diluting liquid is delivered to the zone of discharge of the heavier product by means of a pump device comprising an inverted hollow cone rotating with the centrifuge and dipping into a tank of the carrier fluid. The amount of dilution

is regulated by permitting more or less of the carrier fluid to pass unused through adjustable ports provided in the conical wall of the pump.

B. M. VENABLES.

Centrifugal apparatus for treatment of gases, vapours, liquids, etc. M. AURIG and G. BRÜCKLMAYR (B.P. 333,399, 14.8.29).—A number of discs are rotated on a horizontal shaft; there are no alternate fixed discs, but the rotating discs have projections arranged in curved lines which are interrupted, the projections of each disc intercalating with those on its neighbours.

B. M. VENABLES.

[Stationary] centrifugal apparatus. J. M. SCHUTZ, ASSR. to CENTRIFIX CORP. (U.S.P. 1,753,972, 8.4.30. Appl., 3.3.25).—A multi-tuyèred device suitable for the production of rapid rotary motion in a fluid is constructed of a number of castings all alike. (Cf. U.S.P. 1,539,435; B., 1925, 578.)

B. M. VENABLES.

Centrifugal baskets. E. ROBERTS, ASSR. to WESTERN STATES MACHINERY Co. (U.S.P. 1,753,023, 1.4.30. Appl., 6.4.28).—The clogging of the liquor outlet holes of the basket and the openings of the grid supporting the filtering elements can be prevented by elongating the holes in a circumferential direction and bevelling their edges so that acute angles are formed at the inner surface, and so fixing the grid that the rear boundaries of the holes are slightly behind those of the grid openings.

D. K. MOORE.

Tubular filters for air and gases. F. AMME (B.P. 332,475, 12.10.29. Ger., 12.10.28).—A filter composed of a number of vertical tubular bags, preferably of comparatively small diameter, is kept clean by continuous horizontal vibration of the supporting platforms.

B. M. VENABLES.

Centrifugal apparatus for dust extraction. BRIT. "REMA" MANUF. Co., LTD., and P. HOWDEN (B.P. 332,405, 18.7.29).—The apparatus comprises a number of concentric cylindro-conical walls, the stream of gas and dust being admitted tangentially through the outer wall. The only communication to the inner spaces is through ports in the walls (also provided with tangential guides) so that the air or gas is continually directed to a central outlet.

B. M. VENABLES.

[Vapour-phase] catalytic apparatus. SELDEN Co., Assees. of A. O. JAEGER (B.P. 331,468, 4.11.29. U.S., 22.12.28).—In a plant comprising a series of (two) catalyst layers or converters, the first layer is cooled by the entrant gases, the second being cooled independently either by fresh entrant gases or by other gas, preferably re-circulated. Four such plants are figured.

C. HOLLINS.

[Boiler-]scale removing and preventing apparatus. G. S. NEELEY (U.S.P. 1,773,274—5, 19.8.30. Appl., [A] 21.11.25, [B] 19.4.26).—See B.P. 264,551 and 287,196; B., 1927, 240; 1928, 390.

Heat-treatment process and apparatus in which a hot liquid mass is employed. C. F. HAMMOND, ASSR. to W. SHACKLETON (U.S.P. 1,765,148, 17.6.30. Appl., 1.4.27. U.K., 15.4.26).—See B.P. 278,768; B., 1927, 928.

Centrifugal machines. SHARPLES SPECIALTY Co., Assees. of W. H. BATH (B.P. 330,158, 27.6.29. U.S., 5.12.28).—See U.S.P. 1,750,154; B., 1930, 746.

Mechanical preparation of pulverised solid fuels and like materials. G. S. LOY (U.S.P. 1,774,487, 26.8.30. Appl., 1.12.28. Fr., 9.12.27).—See B.P. 301,887; B., 1930, 403.

Pulveriser. W. A. WHITE (U.S.P. 1,772,974, 12.8.30. Appl., 11.7.29. U.K., 21.8.28).—See B.P. 318,375; B., 1929, 875.

Machine for crushing ore, stone, etc. J. W. ERICSSON (U.S.P. 1,773,616, 19.8.30. Appl., 13.10.27. Swed., 4.11.26).—See B.P. 293,651; B., 1928, 657.

Machinery for grinding, mixing, and like operations. J. H. J. WOOD (U.S.P. 1,774,464, 26.8.30. Appl., 24.5.28. U.K., 7.6.27).—See B.P. 291,952; B., 1928, 627.

Atomiser for liquids. R. ELSÄSSER, Assr. to SIEMENS-SCHUCKERTWERKE A.-G. (U.S.P. 1,764,437, 17.6.30. Appl., 8.3.28. Ger., 15.3.27).—See B.P. 287,105; B., 1928, 658.

Apparatus for separating liquids of different sp. gr. W. LINNMANN, JUN. (Re-issue 17,776, 26.8.30. of U.S.P. 1,671,115, 29.5.28).—See B., 1928, 552.

Separation, by distillation, of miscible liquids. T. E. PERKS (U.S.P. 1,774,210, 26.8.30. Appl., 3.11.27. N.Z., 26.4.27).—See B.P. 289,394; B., 1928, 773.

Separating out and removing matter suspended in a fluid. H. S. HELE-SHAW (U.S.P. 1,773,797, 26.8.30. Appl., 13.3.23. U.K., 19.7.22).—See B.P. 210,101; B., 1924, 239.

Separation of gas mixtures. P. SCHUFTAN, Assr. to GES. F. LINDE'S EISMASCHINEN A.-G. (U.S.P. 1,773,012, 12.8.30. Appl., 9.7.28. Ger., 27.7.27).—See B.P. 294,611; B., 1930, 3.

Preparation of condensed gases. C. W. P. HEYLANDT (U.S.P. 1,773,140, 19.8.30. Appl., 24.8.28. Ger., 20.9.27).—See B.P. 297,384; B., 1930, 41.

Handling and conveying furnace residues and other solids by water flushing. ASH Co. (LONDON), LTD. (B.P. 333,628, 18.5.29).

[Ductile metal ring for] hydraulic piston packing. H. J. B. SCHARNBERG (B.P. 333,376, 30.7.29).

Tightening and bearing surfaces of apparatus or machines [containing dust]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,939, 26.3.29).

Rotary kilns (B.P. 332,761).—See IX. **Lead alloys for packing joints** (U.S.P. 1,743,303).—See X. **Preventing scums etc. in water** (U.S.P. 1,745,141).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Action of chlorine on coal. A. ECCLES and A. McCULLOCH (J.S.C.I., 1930, 49, 377—386 r).—When chlorine is brought into contact with coal 1 volume of hydrogen chloride is produced for 1 vol. of chlorine taken up by the coal through substitution; a further quantity of chlorine reacts with the coal through addition. The coal, however, absorbs chlorine in excess of that accounted for above, owing to its adsorptive capacity. Hydrogen chloride produced by reaction is also adsorbed by the coal. An apparatus in which the

absorption of chlorine by coal, and the evolution of hydrochloric acid, have been studied under standard experimental conditions is described. Absorption of chlorine by the coal, and the evolution of hydrochloric acid, are rapid in the initial stages of chlorination, but later become very slow until finally equilibrium is attained between the free chlorine in the atmosphere, the hydrogen chloride produced by chlorination, and the chlorinated coal. Temperature, pressure, and fineness of division of the coal influence the amount of hydrogen chloride evolved by the coal under any given set of conditions. An increase in the pressure and temperature, or in the fineness of division of the coal, results in an increased evolution of hydrogen chloride. The initial rate of absorption of chlorine is influenced by all these factors, but the rate of absorption in the later stages is not so strongly affected. If the chlorinated coal is placed under reduced pressure it loses further quantities of hydrogen chloride. At pressures from atmospheric to about 100 mm. of mercury the rate of evolution is comparatively slow, but below the latter pressure evolution is very rapid. Chlorinated coal partly freed from hydrogen chloride by evacuation will re-absorb a large amount of chlorine very rapidly for a short initial period, after which absorption ceases. Re-chlorinated coal, after evacuation, again absorbs chlorine in the same manner as and practically to an equal extent to the chlorinated coal after evacuation. Re-chlorinated coal rapidly loses hydrogen chloride at a reduced pressure approximately equal to that at which hydrogen chloride is evolved freely by the chlorinated coal.

Chemical composition and methods of analysis of peat-forming plants and varieties of peat. S. A. WAKSMAN (Brennstoff-Chem., 1930, 11, 277—281).—Various methods of analysis which have been proposed are briefly criticised and some results obtained by the author's method are discussed (cf. B., 1928, 880). The marked differences in the cellulose and nitrogen contents of high-moor and low-moor peats are attributed to differences in the activity of the micro-organisms (cf. B., 1929, 965).
A. B. MANNING.

Determination of moisture in coal. E. E. CASIMIR and A. POPESCU (Inst. Geol. Roman. Stud. tech. econ., 1929, 13, No. 2; Chem. Zentr., 1930, i, 1563).—Distillation with xylene is preferred; the methods are critically discussed.
A. A. ELDRIDGE.

Action of organic solvents on Wattenbach bright pitch coal, with especial reference to the tar yield therefrom. C. STAEMMLER (Brennstoff-Chem., 1930, 11, 281—282).—On refluxing with benzene, aniline, and tetralin, respectively, the coal yielded 8.7, 53.3, and 36.2% of extract. With aniline it was found difficult to free either the extract or the residue completely from the solvent; with tetralin some decomposition occurred, as was shown by the formation of water and the evolution of hydrogen sulphide. The yield of tar on carbonising the partially extracted coal in the Fischer assay retort was lower (10—16%) than that from the original coal (22.5%). Addition of the solvent to the coal before carbonisation was without influence on the tar yield (cf. Kreulen, B., 1929, 966). With the addition of a

machine oil (b.p. 200–400°) to the coal the carbonisation results were irregular, but no increase in the tar yield was observed. The residues from the aniline and tetralin extractions contained 5.03 and 9.72% of humic acids, respectively. Carbonisation of the humic acids yielded only traces of tar (cf. Erdmann, B., 1921, 570 A).

A. B. MANNING.

Cenospheres and the structure of coke. F. S. SINNATT (J.S.C.I., 1930, 49, 335–338 T).—Cenospheres are produced by allowing particles of coal to enter an inert atmosphere maintained at temperatures above about 380°. The bodies are hollow spheres, of which the main features consist of a lattice with the intermediate spaces covered by a film which, by transmitted light, is translucent or transparent. The walls of certain cenospheres contain minute particles which have been termed the tertiary structure. Up to the present the source and nature of the tertiary structure have not been determined. The structures found in cenospheres are shown to be present in coke produced by the carbonisation of coal at low and high temperatures. The results confirm the work of Beilby, who showed that in the formation of coke from a caking coal the product passed through a stage in which it was a foam.

Formation of cenospheres as a means of studying the swelling capacity of coal. J. H. CARLILE and F. S. SINNATT (J.S.C.I., 1930, 49, 355–359 T).—Various methods which have been used to obtain a measure of the swelling capacities of coals are reviewed. A new method is proposed based on the carbonisation of coal in the form of fine particles—cenosphere formation. Figures are given for six coal seams showing the swelling resulting from this method of treatment. Strongly caking coals give a value of 32, which corresponds to an actual swelling of the coal of about 20 times. The method is readily adapted for examination of any coal seams and for studying various effects such as oxidation, weathering, and blending.

Speed and temperature of combustion after preheating gas and air. H. PASSAUER (Gas- u. Wasser-fach, 1930, 73, 313–319, 343–348, 369–372, 392–397).—The rate of combustion of hydrogen, carbon monoxide, ethylene, methane, acetylene, and various technical mixture of these gases has been determined and the maximum flame temperature measured with and without preheating of the gases. In all cases maximum flame temperature is obtained when the theoretical amount of air for complete combustion is present. This maximum approaches more closely to the theoretical value the higher is the speed of combustion, the difference between the theoretical and actual values being dependent on the rate of heat exchange per cm.² of combustion surface per sec. This difference is a hyperbolic function of the intensity of combustion in all cases, so it is possible to calculate the actual temperature of the flame from a knowledge of the theoretical maximum temperature and the conditions of combustion. The maximum speed of combustion occurs in mixtures containing a greater deficiency of oxygen as the temperature to which the gases are preheated is increased; the extent of this displacement is greatest at flame temperatures of 1200–1600° and then decreases. The

rate of combustion in g./cm.²/sec. is directly proportional to the increase in the preheating temperature; extrapolation of the curve indicates that this rate approaches zero in the neighbourhood of the absolute zero.

A. R. POWELL.

Composition of benzol from gas manufactured in vertical retorts. W. A. VOSS (J.S.C.I., 1930, 49, 343–348 T).—Benzol extracted from gas derived from continuously operated "steamed" vertical retorts carbonising Yorkshire run-of-mine coal is characterised by high contents of unsaturated hydrocarbons and paraffins. As a result of the latter the sp. gr. is low and approximates to 0.845. Though there is no reason to suppose that this low gravity would adversely affect its use as a motor fuel, it makes compliance with the existing benzol specification difficult. The investigation was carried out on two different types of vertical-retort settings and the benzols, which represented the make over a continuous period of some weeks, showed close agreement on analysis.

Recovery of phenol from coke-oven gas liquor in relation to the best known processes of by-product recovery. H. WIEGMANN (Brennstoff-Chem., 1930, 11, 285–288, 304–306; cf. Hoening, B., 1929, 383; Prüss, B., 1929, 1038).—The most efficient arrangement of the phenol recovery plant with various types of by-product recovery process (direct, semi-direct and indirect) is discussed and illustrated by reference to the operation of plants in the Ems district.

A. B. MANNING.

Removal of coke from Boryslaw asphalt. R. FUSSTEIG (Petroleum, 1930, 26, 867–868).—During treatment of Boryslaw asphalt for decomposition of the paraffin wax which it contains, considerable quantities of coke are produced. This makes the material unsuitable for road construction, as its softening and wetting properties are modified, and it is necessary to extract the asphaltic substances with benzene in order to separate the coke.

R. H. GRIFFITH.

Determination of tar and bitumen in mixtures. J. MARCUSSE and P. LEDERER (Petroleum, 1930, 26, 866–867).—Sulphonation is used to differentiate coal tar, brown coal, and bituminous substances in the presence of each other. The mixture under examination (3 g.) is boiled for 15 min. with chloroform, and the solvent is driven off. The residue is mixed with 6 c.c. of sulphuric acid, heated for $\frac{3}{4}$ hr. on a water-bath, and then poured into 500 c.c. of water. Coal-tar products give substances which are thus rendered completely soluble, but in the case of brown-coal derivatives only partial solubility in warm water is found. If the residue is completely insoluble, only bituminous material was present. Further information can be obtained by repeating sulphonation on the same sample; the products from bituminous substances are insoluble in dilute aqueous ammonia.

R. H. GRIFFITH.

Volatility of motor fuels. G. G. BROWN (Dept. Eng. Res., Univ. Michigan, Bull. 14, 1930, 299 pp.).—The ease with which a motor will start, the length of time required to warm it up, and the character of its general performance have been investigated with regard to the volatility of the fuel. It is shown that equi-

librium volatility as determined by the methods of air distillation, computation by Raoult's law, and the dew point is far from reliable and that the ordinary A.S.T.M. method of distillation compares very favourably with the results obtained by continuous equilibrium vaporisation. Tests on a small, water-cooled, 4-cylinder engine appear to show that the ability of a fuel to start a motor at 18° depends very largely on those constituents making up the A.S.T.M. distillation curve below 94°, and, at 2°, on the presence of those constituents making up the curve below 65°. The 10% point is related to the lowest engine temperature at which satisfactory starting may be obtained and the lowest mixture temperature at which the motor may be operated. The 35% point is related to the lowest mixture temperature at which satisfactory performance may be obtained during the warming-up period, and therefore determines the length of time necessary to warm up the motor. The 65% point is related to the lowest mixture temperature at which perfect performance can be obtained. For these reasons the 10%, 35%, and 65% points should be low to ensure satisfactory starting, warming-up, and general performance. The 90% point, however, should not be so low as to indicate a dry mixture, as this means loss of power or acceleration with modern cars equipped with heated manifolds and accelerating devices. The vapour pressure of the fuel or the 10% point should not be so low as to cause trouble through vapour-lock. The relations developed make it possible to determine the volatility characteristic of a fuel for any desired engine performance.

H. S. GARLICK.

Removal of sulphur from petroleum products. M. NAPHTALI (Brennstoff-Chem., 1930, 11, 282—285).—Recent developments in the methods of refining petroleum products are summarised.

A. B. MANNING.

Polymerisation reactions of ethylene. H. M. STANLEY (J.S.C.I., 1930, 49, 349—354 r).—A summary of previous work on the polymerisation of ethylene by ultra-violet light, silent electric discharge, heat, pressure, and catalysts such as anhydrous metallic halides is given. The polymerising action of aluminium chloride is considered to be a reaction of the Friedel-Crafts type and numerous examples of the Friedel-Crafts reactions in the olefine series are adduced. The author's own experiments on the polymerising action of various catalysts on ethylene at pressures up to 60 atm. in a steel autoclave are briefly summarised. In the absence of catalyst, condensation of ethylene to liquid hydrocarbons took place under pressure at 325° and the reaction was rapid at 350°. The reaction between aluminium chloride and ethylene under pressure at room temperature resulted in the formation of a free oil and a pasty double compound of aluminium chloride and liquid hydrocarbons. The free oil consisted of liquid hydrocarbons of the formula C_nH_{2n} with about 10—45 carbon atoms per mol. and mostly of a saturated nature, presumably cycloparaffins. The "combined" oil, liberated from the aluminium chloride complex by iced water, also consisted of a mixture of liquid hydrocarbons containing from 10 to over 50 carbon atoms per mol. The latter appeared to be unsaturated and contained rather less hydrogen than C_nH_{2n} . At higher temperatures (up

to 180°) the condensation between ethylene and aluminium chloride was much more rapid and resulted, as before, in the production of a free oil and an aluminium chloride complex compound. This complex tended to become more and more carbonaceous at higher temperatures. The free oil produced at higher temperatures contained large proportions of low-boiling paraffin hydrocarbons, whilst the higher-boiling fractions were deficient in hydrogen and corresponded to the formulae C_nH_{2n-6} , C_nH_{2n-8} , and C_nH_{2n-10} . Lower paraffin hydrocarbons also appeared in the residual gases from the experiments, their amount increasing with rise in temperature. A theory of the mechanism of these reactions is outlined.

Fatty acids from oxidation of mineral oils. COLLIN.—See III. **Determination of traces of carbon monoxide.** ANON.—See VII. **"Penetrol" as insecticide.** HOERNER.—See XVI.

See also A., Aug., 994, **Water-gas equilibrium and the electric discharge** (PETERS and KÜSTER). 1002, **Oxidation processes in motor fuels** (BERL and WINNACKER).

PATENTS.

Apparatus for the dry distillation and method for the preliminary drying of bituminous materials. EESTI PATENDI AKTSIASELTS (B.P. 307,795, 8.3.29. Estonia, 13.3.28).—A tunnel oven suitable for carrying out the distillation process described in B.P. 278,740 (B., 1929, 44) comprises a drying and preheating chamber, a distillation chamber, and a cooling chamber, through which the material is conveyed in layers of over 50 cm. thickness on cars having gas-permeable bottoms. Lock chambers are provided at both ends of the distillation chamber to prevent access of air thereto. Distillation is effected by the repeated circulation of hot gaseous and vaporous products of distillation, to which additional steam may be added if desired, through suitably arranged superheaters and through the material. The circulation is produced by means of fans in the distillation chamber, each car therein having its corresponding fan and superheater, which are so arranged that a different régime may be maintained in different parts of the distillation chamber. The superheaters are heated by the gases from a combustion chamber, and the exhaust gases from the superheaters are utilised in drying and preheating the material.

A. B. MANNING.

Combustion apparatus and method. A. J. WHEELER (B.P. 331,183, 19.2.29).—The combustion of a stream of pulverised coal, oil, or gas in a furnace is accelerated by applying heat to the stream at a point near its entry into the furnace by means of a supplementary flame. Provision may be made for supplying air for combustion at successive points in the length of the main flame.

A. B. MANNING.

Preparation of pulverised fuel for combustion. PEABODY ENG. CORP., Assees. of H. T. DYER (B.P. 309,572, 9.3.29. U.S., 14.4.28).—The finely-divided fuel suspended in carrier air is conveyed to a distributor into which a current of compressed air is injected in such a manner as to effect uniform distribution of the fuel in the air. The stream is then split up into a

number of separate streams for delivery to the individual points of consumption.

A. B. MANNING.

Preheating of carbonaceous materials prior to their destructive hydrogenation. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,498, 8. and 31.12.28). The material is preheated in stages at successively higher pressures. The first stages are carried out in the absence of hydrogen, but under pressure if desired, and without substantial decomposition of the material, and the succeeding stages are carried out in the presence of hydrogen.

A. B. MANNING.

Distillation of coal or other carbonaceous material. F. PUENING (B.P. 312,238, 22.5.29. U.S., 22.5.28).—A series of concentric circular walls are suspended from a water-cooled framework in such a manner as to form between them a series of annular coking chambers. The walls are hollow and are heated by the circulation therein of hot gases supplied from superimposed, annular, hot gas chambers connected with a furnace. The charging and discharging mechanism operates from the underside of the chambers, and consists of a rotating platform on the front of which is a number of rakes, each passing into one of the chambers to remove the coke, and at the rear of which is a coal charger for supplying each individual chamber from a central bunker. The base of the chambers is formed by drop-doors, the operation of which is dependent on the rotating platform. An annular chamber below the system of coking chambers permits the movement of the drop-doors and accommodates the coke-removing mechanism.

A. B. MANNING.

Distillation of solids carrying carbonaceous materials. H. O. SCHUNDLER (B.P. 330,531, 8.3.29).—Oil shale, coal, etc. is distilled in a rotary, horizontal retort, preferably of hexagonal cross-section and in the shape of a frustum of a pyramid, which is slowly rotated and is devoid of internal projections likely to cause gas disturbance, so that the vapours evolved at different points as the material passes down the retort and is heated to successively higher temperatures form stratified layers and pass through the outlet in such formation. A current of nitrogen may be introduced into the retort at the charging end at a rate which does not disturb the stratification.

A. B. MANNING.

Low-temperature distillation or carbonisation of bituminous coal. A. V. ABBOTT (B.P. 330,902, 18.3.29).—A horizontal rotary retort is constructed with outer and inner walls forming an annular distillation chamber, the outer wall being provided with lifting ribs or baffles, and the inner wall with retarding baffles. The coal is thereby given an alternately advancing and retarding movement as it passes through the chamber. The temperatures of the walls are so adjusted that the coal is subjected to different distillation temperatures while advancing and retarding, respectively. The temperatures are controlled by the provision of vessels filled with molten lead which are interposed between the combustion chamber and the walls of the retort.

A. B. MANNING.

Low-temperature carbonisation of coal and other suitable fuels. J. E. HACKFORD (B.P. 331,193,

21.12.28).—The fuel is charged into cylindrical containers which are then carried through a horizontal retort on an endless-chain conveyor. The containers enter and leave at both ends of the retort, one set being carried through in one direction on the upper part of the conveyor, and another set in the opposite direction on the lower part. Automatic means are provided outside the retort for lining the containers with a suitable medium (cf. B.P. 326,813; B., 1930, 545), charging them with coal, and discharging the coke therefrom. The retort, the walls of which are built up of hollow cast-iron sections, comprises a central carbonising zone, and two end zones wherein the coal is preheated or the coke produced is cooled. The central zone is heated by means of tubes which traverse the retort from side to side, and are fired at one end by gas or oil burners. After traversing the heating tubes the hot gases are circulated within the hollow walls of the retort.

A. B. MANNING.

Carbonisation of coal, peat, wood, etc. C. B. WINZER (B.P. 330,980, 22.3.29).—A series of stationary retorts are mounted within a circular-travelling, closed tunnel of inverted U-shape in cross-section. Part of the tunnel is provided with double walls and is heated by the circulation of hot gases in the intervening space, thereby bringing about the carbonisation of the material in the retorts as the heated section of the tunnel passes over them. After circulating between the double walls of the tunnel, the hot gases may be passed round the retorts within the tunnel and thence to a chimney. Provision is made for charging and discharging the retorts while they are in the cooler part of the tunnel, and for isolating these retorts from the others by gas-tight doors during this operation. Each retort is provided with offtake pipes for leading off the volatile products of distillation.

A. B. MANNING.

Production of free carbon from hydrocarbons. G. C. LEWIS (B.P. 309,488, 14.3.29. U.S., 11.4.28).—Natural gas, stripped of its heavier constituents, is passed through a tube heated at a temperature (600—900°) at which cyclic hydrocarbons are produced, but below that at which any free carbon is formed, the condensable products are removed, and the residual gas, containing 15—70% H₂, is subjected to incomplete combustion for the production of free carbon. The heat evolved in the last stage of the process is used to preheat the gas. (Cf. B.P. 309,992, following.)

A. B. MANNING.

Production of carbon [black]. G. C. LEWIS (B.P. 309,992, 14.3.29. U.S., 19.4.28. Cf. B.P. 309,488, preceding).—The process depends on the incomplete combustion of mixtures of gaseous hydrocarbons with hydrogen, containing 15—70% of the latter. The proportion of hydrogen used varies with the hydrocarbons and with the desired yield and quality of the carbon produced. The hydrogen may be added to the hydrocarbons or may be produced from the latter by a suitable cracking process. Any aromatic hydrocarbons formed in the cracking process may be separated from the gas before it is supplied to the burners.

A. B. MANNING.

Reactivation of decolorising carbon. F. W.

MEYER (B.P. 317,017, 8.8.29. Ger., 8.9.28).—The spent carbon, preferably in a moist condition, is subjected to the action of steam at temperatures between 180° and 300°, in a closed vessel. A. B. MANNING.

Method of generating gas. C. W. ANDREWS and W. B. CHAPMAN (B.P. 330,509, 9.2.29).—An ash-liquefying zone is maintained at an intermediate level in the fuel bed of a gas producer, and the liquefied ash runs down into a slightly cooler zone wherein it is subjected to the action of a water-cooled agitating member which breaks up the clinkers and prevents the formation of large masses. The steam is passed both up and down through the fuel bed, the ratio of downward steam to total steam being at least 60%. A relatively shallow fuel bed is maintained so that the blast gases contain not more than 14% CO. The ash is removed continuously from the bottom of the fuel bed. A. B. MANNING.

Gas producers. J. U. MARGUET (B.P. 330,815, 30.7.29).—A gas producer with reversed draft is provided with an annular metallic evaporator embedded in the refractory wall of the shaft. The air for combustion passes through the evaporator, the mixture of air and steam thereby formed passing into the shaft through a circular slot. A. B. MANNING.

Manufacture of gas. HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (B.P. 319,769, 10.9.29. U.S., 29.9.28).—A mixed oil gas and blue water-gas is produced by cracking oil gas with deposition of carbon in an incandescent fuel bed, and generating blue water-gas by air- and steam-blasting the fuel bed, the deposited carbon being partly burned to heat the fuel bed and partly converted into water-gas by interaction with steam. The oil gas is preferably passed through the fuel bed alternately in one direction and then in the other between air-blasting periods, and may be derived from an outside source or may be generated in the carburettor or superheater. A. B. MANNING.

Producing a mixture of oil gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (B.P. 331,138, 11.9.29. U.S., 8.11.28).—Blue water-gas is produced by air- and steam-blasting an ignited fuel bed, and oil is then introduced into the system in such a manner that the oil gas or oil vapour passes through the carburettor and the fuel bed in parallel, thereby producing lightly cracked and heavily cracked oil gas. A. B. MANNING.

Production of gaseous mixtures suitable for the synthesis of hydrocarbons, methyl alcohol, and other oxy-organic compounds. G. NATTA (B.P. 330,918, 11.3.29).—A mixture of carbon monoxide, carbon dioxide, and hydrogen, containing at least 2 vols. of hydrogen for each vol. of carbon monoxide, is produced by passing a mixture of oxygen and steam over carbon, substantially free from volatile impurities, at not above 750°. The reaction temperature is regulated either by varying the preheating of the gaseous mixture, or by introducing suitable quantities of water into the reaction zone in place of the corresponding quantities of steam. The reaction may, with advantage, be carried out under pressure. A. B. MANNING.

Preparing [explosive] gases [e.g., acetylene] by

interaction of solids and liquids. Production of acetylene from carbide and water. AUTOGENWERK SIRIUS GES.M.B.H. (B.P. 330,821—2, 8.8.29. Ger., [A] 22.2.29, [B] 15.5.29).—Forms of acetylene generators are described. A. B. MANNING.

Treatment of natural gas. G. C. LEWIS (B.P. 316,999, 14.3.29. U.S., 8.8.28).—A portion (90%) of the gas is passed through tubes heated by the combustion of the remainder, and the gases produced by the heat treatment of the former are mixed with the hot products of combustion of the latter; the mixed gases are expanded and cooled, and are then passed through an absorber for the removal of benzene. The residual gas, if desired, may be burned for the production of carbon black, the waste heat from this process being utilised in preheating the original gas. A. B. MANNING.

Removal of sulphur impurities from combustible gases. W. J. HUFF, L. LOGAN, and O. W. LUSBY (B.P. 330,933, 19.1.29).—The gas is passed over a purifying mass consisting of a mixture of metals or metallic oxides, including at least one from group V, e.g., mixtures of copper with chromium, uranium, or vanadium, at raised temperatures, e.g., 250°. The mass is revived by treatment with an oxygen-containing gas at 250° or above, the apparatus being purged with a non-combustible gas before and after passing the revivifying gas. The purifying mass may be formed into granules resistant to disintegration, by fusing, or by admixture with clay etc., or may be deposited on a suitable carrier. A. B. MANNING.

Distillation of tar. BARRETT Co., Assees. of S. P. MILLER (B.P. 308,577, 9.2.29. U.S., 23.3.28. Cf. B.P. 286,278; B., 1929, 970).—Tar, pitch, or tar oils are distilled in an externally heated still into which the hot coal-carbonisation gases from coke ovens, horizontal retorts, etc. are introduced in such a manner as to be brought into intimate contact with the material being distilled. If desired, the hot gases may be cleaned, e.g., by scrubbing with pitch or by electrical precipitation, before entering the still. A. B. MANNING.

Manufacture of coal-tar products [e.g., electric insulating material, artificial ebonite, etc.]. A. GERLEY and W. A. MACKINLAY (B.P. 330,712, 3.5.29).—A mixture of the middle oils from the distillation of coal tar with copper sulphate, methyl alcohol, an aqueous solution of glue, rosin, and hydrochloric acid or an alkali is submitted to suitable heat and pressure. Finely-ground inert materials, e.g., wood-pulp, worn-out tyres, etc. may be incorporated as fillers and aniline dyes added. A. B. MANNING.

Cracking of hydrocarbons. G. F. FORWOOD, J. G. TAPLAY, and UNITED KINGDOM OIL Co., LTD. (B.P. 332,285, 18.4.29).—Kerosene or crude oil residue is atomised with superheated steam into an inclined cylindrical vessel containing a catalyst obtained by treating cellulose charcoal with 5% of China clay dispersed in sodium silicate. The catalyst is maintained at 400–650°, and additions are made as the catalytic material is consumed. Vapours from the catalysing chamber are conducted to a large dephlegmating column, where they are cooled and heavy fractions separated.

From 1 to 1.5 lb. of steam is used per 1 lb. of oil, the steam being largely decomposed. T. A. SMITH.

Production of light hydrocarbons by destructive hydrogenation of carbonaceous materials. J. L. FOHLEN (B.P. 313,963, 18.6.29. Appl., 20.6.28).—Carbonaceous material, *e.g.*, shale oil, is heated in an autoclave with a metallic halide, iron filings, and copper or brass shavings. The temperature is maintained at 300–400° for 4 hrs. and the pressure rises to 120–180 atm. After treatment the shale oil yields 50% of a light oil (d 0.850) boiling below 200° and 42% of an oil (d 0.870) boiling at 200–300°. [Stat. ref.]

T. A. SMITH.

Working-up of distillable carbonaceous liquids containing substances of high mol. wt. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,336, 15.5.29).—In the hydrogenation of carbonaceous liquids containing substances (*e.g.*, asphalt or pitch) of high mol. wt., the latter, which causes trouble owing to the poisoning and clogging effect they have on the catalyst, may be removed by absorbing them on a prepared absorbent, such as silica, calcined after impregnation with molybdenum and zinc nitrates. The oil is freed from the asphalt and absorbent by means of a centrifuge, and each portion is hydrogenated separately under suitable conditions of temperature and pressure. The absorbent may then be regenerated. T. A. SMITH.

Working-up of the oil-bearing residues from destructive hydrogenation or pressure-extraction processes. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 330,723, 10.5.29. Addn. to B.P. 304,404; B., 1929, 232).—The residues are mixed with solid carbonaceous materials, *e.g.*, poorly coking coals, and are carbonised at temperatures above 600°. Recovery of 90–95% of the oil in the residues is possible and a highly reactive coke is obtained, particularly suitable for the production of water-gas. A. B. MANNING.

Illuminating oil and its manufacture. F. W. HALL, Assr. to TEXAS CO. (U.S.P. 1,752,229, 1.4.30. Appl., 9.1.26).—An oil fraction of viscosity 100–150 sec. (Saybolt) at 100°, water-white in colour, and of approx. zero iodine value is prepared by acid-treating a suitable fraction followed by upward filtration through fuller's earth. From 5 to 40% of this oil is mixed with refined heavy kerosene to give a suitable railway-signal lamp oil. T. A. SMITH.

Conversion of heavy hydrocarbon oils into light hydrocarbon oils or spirits. F. LAMPLUGH (U.S.P. 1,765,167, 17.6.30. Appl., 27.5.26. U.K., 23.6.25).—See B.P. 258,656; B., 1926, 972.

Purification of hydrocarbon oils. J. TAUSZ (U.S.P. 1,772,921, 12.8.30. Appl., 8.7.24. Ger., 16.7.23).—See G.P. 447,557; B., 1928, 807.

Apparatus for use in discharge of coke ovens. WELLMAN SMITH OWEN ENG. CORP., LTD., and S. STOCKDALE (B.P. 333,294, 11.5.29).

[Poking device for] gas producers. A. JADOUL (B.P. 315,327, 11.7.29. Belg., 11.7.28).

[Electrically controlled] apparatus for automatic manufacture of [carburetted] water-gas.

HUMPHREYS & GLASGOW, LTD., Assees. of M. PARSONS (B.P. 311,279, 6.5.29. U.S., 8.5.28).

Coal-dust burners. S. LÖFFLER (B.P. 312,059, 3.5.29. Ger., 19.5.28).

Apparatus for burning pulverised fuel. G. H. ROBINSON (B.P. 333,017, 29.6.29).

Gas burners. W. E. N. CLARK, and ELECTROLUX, LTD. (B.P. 333,269, 9.4.29).

Gas and/or oil heating appliances. GAS LIGHT & COKE CO., J. C. CLARK, and C. A. MASTERMAN (B.P. 333,338, 11.6.29).

Steam for water-gas plants (B.P. 333,472).—See I. **Reduction of carbon monoxide** (U.S.P. 1,741,306–8). **Higher alcohols** (U.S.P. 1,738,785). **Polymerisation of diolefines** (B.P. 331,265). **Catalytic oxidation of toluene etc.** (B.P. 331,525 and 331,535). **Purification of aromatic hydrocarbons** (U.S.P. 1,741,305). **Conversion of phenols etc. into hydrocarbons** (B.P. 331,199).—See III. **Filtering material** (U.S.P. 1,734,197).—See VII. **Pavement materials** (B.P. 333,433).—See IX. **Tanning agents** (B.P. 332,204).—See XV.

III.—ORGANIC INTERMEDIATES.

Fatty acids from oxidation of mineral oils. G. COLLIN (J.S.C.I., 1930, 49, 333–334 r).—An attempt has been made to apply the ester fractionation method to the analysis of a sample of fatty acids stated to have been made commercially by the oxidation of paraffin wax; the fatty acids in question consisted of a cream-coloured solid material with a faint characteristic odour, equivalent (saponification) 268.4, (by direct titration) 348.7, iodine value 7.5, non-acidic material about 17%. Fractionation of the esters of the fatty acid mixture indicated that the 80% or more of acidic material present had mol. wt. ranging from about 145 to about 300; the acids were almost entirely saturated, there was no sign of the presence of outstanding amounts of any one acid, the mixture of acids present was much more complex than in the case of those of a natural fat, and attempts to isolate individual acids by crystallisation of redistilled ester fractions did not lead to the identification of any known fatty acids. From the observed equivalents it seems probable that acids containing an odd number of carbon atoms in the molecule are present in the mixture, and the m.p. obtained after several recrystallisations suggest that mixtures of acids were still present or that certain of the products, if individual, consisted of branched-chain acids.

Phenol from gas liquor. WIEGMAN.—See II. **Hydrogenation of unsaturated fatty acids.** SUZUKI and INOUE.—See XII. **p-Dichlorobenzene as insecticide.** SNAPP.—See XVI. **Ethylene oxide as fumigant.** BACK and others.—See XIX.

See also A., Aug., 1027, **Preparation of diazomethane** (ARNDT and AMENDE). 1039, **Isomeric p-benzoquinonediithioglycolic acids** (GEBAUER-FÜLNEGG and JARSCH). 1042, **Benzanthrone series** (MAYER and others). 2:7-Dimethylantraquinone (MAYER and GÜNTHER). 1043, **Halogenoquinizarins** (WALDMANN). **Phenanthraquinone**, 1:4-naphtha

quinone, and 1:4-anthraquinone (SKITA and ROHRMANN).

PATENTS.

Reducing the products of carbon monoxide. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,741,306—8, 31.12.29. Appl., [A—C] 8., 9., and 10.11.27).—The activity of mild reduction catalysts, such as copper, manganese, cadmium, zinc, lead, tin, magnesium, silver, gold, and platinum, or their compounds, is moderated further by the addition of oxidation catalysts, such as compounds of chromium, vanadium, manganese, titanium, molybdenum, tungsten, cerium, thorium, uranium, zirconium, etc. (A) The reduction to the methyl alcohol stage takes place in presence of the mixed catalysts on porous carriers, and the further stage to methane in presence of strong reduction catalysts, such as iron, nickel, cobalt, or palladium. (B) In the first stage the mild reduction catalyst is supported on the oxidation catalyst as carrier, or (c) the two catalysts are mixed together.

C. HOLLINS.

Synthesis of methyl alcohol and catalyst therefor. H. H. STORCH, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,738,971, 10.12.29. Appl., 2.8.27).—Carbon monoxide and hydrogen are passed under pressure at 300–400° over a copper–magnesia–silica catalyst formed by reducing the precipitate obtained by adding sodium hydroxide to a solution of sodium silicate, magnesium nitrate, and copper nitrate.

C. HOLLINS.

Manufacture of higher alcohols. R. H. MCKEE and S. P. BURKE, Assrs. to R. L. BROWN and W. W. ODELL (U.S.P. 1,738,785, 10.12.29. Appl., 21.2.23).—Unsaturated hydrocarbons, *e.g.*, cracked gases, are passed with steam at 350–450° over alumina or other hydrating catalyst.

C. HOLLINS.

Polymerisation [of diolefines] and apparatus therefor. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,265, 23.3.29).—A second chamber, containing no polymerising agent, is connected with the reaction vessel so that on the occurrence of undesirable violent reactions the starting material distils into the second chamber, whence it may be returned when convenient, *e.g.*, by distillation or by gravity, to the reaction vessel.

C. HOLLINS.

Preparation of diolefines directly from alcohols. S. V. LEBEDEV (B.P. 331,482, 30.1.29).—Vapours of methyl, ethyl, or propyl alcohols, or mixtures of these, are led at 400°, preferably at reduced pressure (*e.g.*, 0.25 atm.), over a mixture of a dehydrating and a dehydrogenating catalyst, *e.g.*, alumina and zinc oxide.

C. HOLLINS.

Manufacture of acetic anhydride and acetaldehyde. SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 318,960, 2.7.29. Fr., 13.9.28).—The vapours from the dissociation of ethylidene diacetate are led through a suspension or solution of organic salts (*e.g.*, potassium acetate) in a mixture of ethylidene diacetate and acetic anhydride so as to remove hydrochloric acid or other polymerising agent.

C. HOLLINS.

Manufacture of aldehydes from α -oxides of hydrocarbons [olefine oxides]. J. Y. JOHNSON.

From I. G. FARBENIND. A.-G. (B.P. 331,185, 18.3. and 14.10.29).—An olefine oxide is passed at 180–300° over a catalyst comprising an element of group V or VI in the form of a non-volatile oxygenated acid, acid anhydride, or salt, *e.g.*, magnesium pyrophosphate, copper sulphate, cerous sulphate, with or without addition of metal oxides, hydroxides, or carbonates, on a carrier (pumice, kieselguhr) if desired. Propylene $\alpha\beta$ -oxide gives propaldehyde, with allyl alcohol and acetone; butylene $\alpha\beta$ - or $\beta\gamma$ -oxide yields *n*- and *iso*-butaldehydes, with methyl ethyl ketone and butenols; ethylene oxide gives acetaldehyde.

C. HOLLINS.

Manufacture of aldehydes from alcohols. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,883, 11.4.29).—An alcohol vapour is passed at 200–380° and at pressure not exceeding atmospheric over an alkali-free catalyst comprising copper, which has been reduced below red heat, and a metal oxide or phosphate, *e.g.*, zinc oxide, magnesia, or disodium hydrogen phosphate; such a catalyst retains its activity longer than in the absence of a promoter.

C. HOLLINS.

Concentration or extraction of organic acids occurring in aqueous solution. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 331,637, 10.5.29. Fr., 20.3.29. Addn. to B.P. 312,046; B., 1930, 232).—The process of the prior patent is extended to aliphatic acids other than acetic; *e.g.*, for propionic acid the solvent is butyl propionate and the entraining liquid a petroleum fraction, b.p. 138–140°; for formic acid, amyl formate and heptane are used.

C. HOLLINS.

Ether–alcohol esters of fatty acids. J. M. KESSLER and O. B. HELFRICH (U.S.P. 1,739,315, 10.12.29. Appl., 25.4.28).—A glycol monoalkyl ether is esterified with an aliphatic acid above C₅ (stearic acid) to give plasticisers. The stearate, m.p. 55–60°, of diethylene glycol monoethyl ether (β -hydroxy- β' -ethoxydiethyl ether) is described.

C. HOLLINS.

Production of amines from organic acids and their anhydrides. KNOLL A.-G. CHEM. FABR., and K. F. SCHMIDT (B.P. 307,798, 11.3.29. Ger., 13.3.28).—Carboxylic acids or anhydrides in sulphuric acid react with azoimide in chloroform at 40° to yield primary amines: $R \cdot CO_2H + N_3H \rightarrow R \cdot CO \cdot N_3 + H_2O \rightarrow R \cdot NH_2 + N_2 + CO_2$, or $N_3H \rightarrow NH_2 + N_2$; $R \cdot CO_2H + NH_2 \rightarrow R \cdot NH_2 + CO_2$. The preparation of aniline from benzoic acid, benzylamine from phenylacetic acid, tetramethylenediamine from adipic acid, pentamethylenediamine from *d*-leucine, and methylamine from acetic anhydride is described. The yields are 70–92% of theoretical.

C. HOLLINS.

Production of imino-ethers. K. F. SCHMIDT and P. ZUTAVERN (B.P. 331,947, 19.4.29).—Ketones react with azoimide in benzene in presence of sulphuric acid, hydrogen chloride, thionyl chloride, zinc chloride, phosphoryl chloride, phosphorus pentoxide, etc. in an alcohol, with production of imino-ethers: $R \cdot CO \cdot R' + N_3H \rightarrow RR'(OH) \cdot N < + N_2$; $RR'(OH) \cdot N < + R''OH \rightarrow R \cdot C(OR'') \cdot NR'$, a Beckmann transformation taking place. *cyclo*Pentanone, azoimide, and ethyl alcohol yield the cyclic imino-ether, 2-ethoxy-3:4:5:6-tetrahydropyridine, b.p. 161–165°. The

preparation of *N*-methylacetiminoethyl ether, b.p. 99–100°, and a cyclic imino-ether, b.p. 214–216°, from cyclohexanone, azoimide, and butyl alcohol is also described.

C. HOLLINS.

Manufacture of glyoxal and glycollic acid. E. E. AYRES, JUN., Assr. to B.A.S. Co. (U.S.P. 1,741,394, 31.12.29, Appl., 28.11.27).—A relatively slow stream of oxygen is introduced into a tube carrying acetylene at high velocity. The exit gases, after condensation of the glyoxal formed, are recirculated. The glyoxal is converted into glycollic acid, if desired, by caustic alkali in a scrubbing tower which replaces the condenser.

C. HOLLINS.

Preparation of thymol. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 309,031, 28.3.29. Ger., 3.4.28. Addn. to B.P. 326,215; B., 1930, 532).—In place of the propylated and isopropylated *m*-cresols of the prior patent there are used derivatives of these made by sulphonation or treatment with phosphorus pentoxide etc. Conversion into thymol occurs at lower temperatures.

C. HOLLINS.

Production of the potassium salts of the acid sulphuric esters of α - and β -diacetonefructoses. H. OHLE (B.P. 331,922, 12.4.29).—Diacetonefructose potassium sulphates are obtained by adding diacetonefructose to a cooled mixture of pyridine and chlorosulphonic acid, and after some hours distilling off the pyridine and neutralising with potassium hydroxide.

C. HOLLINS.

Manufacture of β -naphthylaminoaryloxy-fatty acids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,597, 10.4.29).—The Bucherer reaction is applied to the condensation of β -naphthols or -naphthylamines with *m*- or *p*-aminophenoxy-derivatives of aliphatic acids. Sodium *p*-aminophenoxyacetate, e.g., is boiled with sodium bisulphite and sodium 2:3-hydroxynaphthoate to give 4- β -naphthylaminophenoxyacetic acid, m.p. 156°, which yields with diazotised *p*-nitroaniline-*o*-sulphonic acid a bluish-violet dye. The following *N*-substituted *p*-aminophenoxyacetic acids are also described; 6-carboxy- and 7-sulpho- β -naphthyl-, 8-hydroxy-6-sulpho- β -naphthyl-, 5-amino- and 5-hydroxy-7-sulpho- β -naphthyl-, and 6-amino-8-sulpho- β -naphthyl-. *o*-Chloro-*p*-aminophenoxy-, 5-amino-*o*-tolyl-, and *m*-aminophenoxy-acetic acids behave similarly.

C. HOLLINS.

Manufacture of γ -alkyl- and γ -aryl-quinolines and their homologues. I. G. FARBENIND. A.-G. (B.P. 308,365, 22.3.29. Ger., 22.3.28).—A primary arylamine is condensed with a β -hydroxyketone or the corresponding unsaturated ketone in presence of sulphuric acid (or other dehydrating agent) and an oxidant, and the resulting 4-alkyl- or -aryl-quinoline is separated by steam-distillation or preferably by precipitation with ferrocyanic acid. The preparation of lepidine from aniline and methyl β -hydroxyethyl ketone or vinyl methyl ketone, of 4:6-dimethylquinoline from *p*-toluidine and methyl β -hydroxyethyl ketone, and of 3:4-dimethylquinoline from aniline and methyl β -hydroxyisopropyl ketone is described. The yields are 10–34% of theoretical.

C. HOLLINS.

Catalytic oxidation of (A) toluene, (B) organic compounds, particularly toluene and like hydrocarbons [anthracene, naphthalene, benzene]. GAS LIGHT & COKE Co., W. G. ADAM, W. V. SHANNAN, and M. CUCKNEY (B.P. 331,525 and 331,535, 29.1.29).—(A) In the air-oxidation of toluene to benzaldehyde and benzoic acid the exit gases are mixed with air and, after being successively saturated with toluene vapour and water vapour, are led at high velocity over a suitable catalyst, closely packed, where only a portion of the toluene is oxidised, and thence to a condenser. The toluene solution of benzaldehyde etc. is circulated to the toluene saturator and the exit gases return to the circuit. (B) A suitable catalyst for air-oxidation of toluene etc. is made by precipitating iron vanadate and iron oxide on short asbestos fibre, extruding the paste into rod form, and drying at 100°. The iron oxide should form 50–60% of the mixed vanadate and oxide.

C. HOLLINS.

Purification of aromatic hydrocarbons. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,741,305, 31.12.29. Appl., 4.2.26).—Crude light benzene, before or after removal of acids and bases, is treated with a limited amount of chlorine to convert aliphatic compounds, thiophenes, carbon disulphide, etc. into high-boiling products, from which the pure aromatic hydrocarbons are separated by distillation.

C. HOLLINS.

Manufacture of intermediates for dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and M. WYLER (B.P. 331,687, 27.6.29).—Acetyl-*m*-2-xylydine is nitrated at 5–20° with mixed acid, the nitro-compound is reduced, and the resulting amine is converted by diazotisation etc. into 2-acetamido-*m*-4-xylenol, m.p. 191°. Hydrolysis with 50% sulphuric acid yields 2-amino-*m*-4-xylenol, m.p. 170°.

C. HOLLINS.

Conversion of oxygen-containing organic substances, such as phenols, into substances free from oxygen. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 331,199, 8.2.29).—Hydrocarbons are obtained by passing vapours of phenols etc. (e.g., tars) with hydrogen etc. under pressure over a molybdenum-carbon catalyst, preferably colloidal molybdenum oxide on finely-divided charcoal or brown coal, heated gradually (40–50 min.) to 450°.

C. HOLLINS.

Manufacture of [nuclear] alkylated phenols. SCHERING-KAHLBAUM A.-G. (B.P. 308,662, 25.3.29. Ger., 23.3.28).—A phenol is heated at 140–200° with an olefine under pressure in presence of a surface catalyst (tensil), and heating is continued until the first-formed phenol ether is converted into *C*-alkylated phenol. The preparation of *p*-propylphenol, *p*-cyclohexylphenol, and 4-cyclohexyl-*m*-cresol is described. Alcohols generating olefines under the reaction conditions may be used. The reaction of cresols with propylene is excluded.

C. HOLLINS.

Manufacture of aminophenol derivatives. H. T. CLARKE, Assr. to EASTMAN KODAK Co. (U.S.P. 1,745,843, 4.2.30. Appl., 3.4.24).—*o*-Aminophenol is converted by means of acetic acid at 175–190° into 2-methylbenzoxazole, which is then treated with methyl sulphate at 80–100°. The resulting additive

compound is treated with cold alkali to give acetyl-methylaminophenol which is hydrolysed with dilute sulphuric acid to give methyl-*o*-aminophenol sulphate.

C. HOLLINS.

Manufacture of water-soluble preparations of phenolic condensation products useful for tanning and other purposes. I. G. FARBERIND. A.-G. (B.P. 302,666 and Addn. B.P. 302,938, [A] 19.12.28. Ger., 19.12.27. [B] 22.12.28. Ger., 23.12.27).—(A) Phenolic condensation products having tanning properties are dispersed with the aid of sulphonated aromatic or hydroaromatic hydrocarbons in high concentration; or the condensation is performed entirely or in its later stages in presence of these sulphonic acids, or in the sulphonation mix. *E.g.*, the resin from phenol and chloroacetone, introduced into tetrahydronaphthalene and 98% sulphuric acid, gives a product which dissolves in water to an acid solution remaining clear on addition of alkali. (B) In addition to the phenolic condensation product an inert or slowly reacting high-molecular, non-phenolic substance is introduced, *e.g.*, resins, oils, waxes, fats (colophony, coumarone resin, castor oil, crude tar fraction of b.p. 200—300°).

C. HOLLINS.

Manufacture of β -sulphophthalic acid. H. MILDNER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,745,025, 28.1.30. Appl., 4.9.28. Ger., 14.6.27).—Phthalic anhydride is sulphonated in the 4-position by oleum at 110—150° in presence of mercuric sulphate.

C. HOLLINS.

Metal salts of phthalic esters. [Driers for paints etc.] C. I. B. HENNING, C. E. BURKE, and E. E. REID, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,742,506, 7.1.30. Appl., 24.9.24).—The heavy-metal salts of monoalkyl phthalates are soluble in acetone and are useful as drying agents in paints and varnishes. The ferrous, ferric, zinc, mercurous, mercuric, lead, stannous, manganous, nickel, cobalt, and copper salts of *n*-butyl hydrogen phthalate are described.

C. HOLLINS.

Manufacture of aromatic amino-sulphochlorides [arylaminesulphonyl chlorides]. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 331,596, 10.4.29).—Primary arylaminesulphonic acids are heated at 80—90° with chlorosulphonic acid, whereby sulphonic groups are converted into sulphonyl chloride groups; new sulphonyl groups or chlorine may also be introduced. The following compounds are described: 4-chloroaniline-3-sulphonyl chloride, m.p. 80—90°; 2:5-dichloroaniline-4-sulphonyl chloride, m.p. 137—138°; 4:5-dichloroaniline-2-sulphonyl chloride, m.p. 125—126°; *p*-toluidine-3-sulphonyl chloride, m.p. 85—86°; 2-chloro-*p*-toluidine-5-sulphonyl chloride, m.p. 127—128°; 3-chloro-*o*-toluidine-5-sulphonyl chloride, m.p. 151—152°; *m*-4-xylylidine-5-sulphonyl chloride, m.p. 85—86°; *m*-phenylenediamine-4:6-disulphonyl chloride, m.p. 185° (decomp.); *m*-aminophenol-4:6-disulphonyl chloride, m.p. 181°; α -naphthylamine-7:1-disulphonyl chloride, m.p. 216—218°; aniline-*o*-sulphonyl chloride, m.p. 72—74°; 6-chloroaniline-3-sulphonyl chloride, m.p. 77—80°; 3-chloro-*o*-toluidine-6-sulphonyl chloride, m.p. 154—155°; *o*-nitroaniline-*p*-sulphonyl chloride, m.p. 149—150°; *m*-nitro compound, m.p. 136°; 1-chloro- β -naphthylamine-6-sulphonyl chloride, m.p.

152—153°; 2:8-aminonaphthol-3:6-disulphonyl chloride, darkens above 250°; dichloro-4:4'-diaminodiphenylamine-2:2'- and -3:3'-disulphonyl chlorides, m.p. 132—133° and 76°, respectively; 2:7-dichloro- α -naphthylamine-4-sulphonyl chloride, m.p. 204—205°.

C. HOLLINS.

Manufacture of 4:6-dihydroxy-2-arylpyrimidines. I. G. FARBERIND. A.-G. (B.P. 309,033, 2.4.29. Ger., 3.4.28).—A malonic ester is heated with an amidine base. 4-Nitrobenzamidine, m.p. 215°, gives 4:6-dihydroxy-2-*p*-nitrophenylpyrimidine, m.p. 320° (decomp.), convertible into the 4:6-dichloro-compound, m.p. 266°. 4:6-Dihydroxy-2-phenylpyrimidine is similarly obtained in quantitative yield.

C. HOLLINS.

Manufacture of naphthalene-2:3-dicarboxylic acid. W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 332,122, 10.9.29).—2:3-Aminonaphthoic acid is converted by way of the diazo compound into the cyanonaphthoic acid, which is hydrolysed by dilute alkali to naphthalene-2:3-dicarboxylic acid, m.p. 236°.

C. HOLLINS.

Purification of 2-aminoanthraquinone. W. M. MURCH and W. J. CAUWENBERG, Assrs. to NAT. ANILINE & CHEM. CO. (U.S.P. 1,744,055, 21.1.30. Appl., 19.10.25).—Crude 2-aminoanthraquinone prepared from 2-chloroanthraquinone is treated with water and an oxidant (chromic acid) to oxidise impurities.

C. HOLLINS.

Production of anthraquinone derivatives [o-halogenated aminoanthraquinones]. R. J. LOVELUCK, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 331,537, 28.12.28).—1:2- and 2:3-Aminoanthraquinonesulphonic acids are converted by potassium persulphate in sulphuric acid into mixtures of the corresponding nitroso- and nitro-compounds. The sulphonic group is then exchanged for chlorine, and reduction, preferably with sodium hydrogen sulphide, yields 2:1- and 2:3-chloroaminoanthraquinones, respectively.

C. HOLLINS.

Manufacture of monoalkyl ethers of ethylene glycol. W. GIBSON and J. B. PAYMAN, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,774,089, 26.8.30. Appl., 7.9.29. U.K., 27.9.28).—See B.P. 322,037; B., 1930, 315.

Production of keten. H. DREYFUS (U.S.P. 1,773,970, 26.8.30. Appl., 27.5.26. U.K., 13.6.25).—See B.P. 262,364; B., 1927, 125.

Manufacture of sulphonic acids of 6-chloro-2-amino-1-methylbenzene [6-chloro-*o*-toluidine]. F. HENLE and B. VOSSEN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,773,706, 19.8.30. Appl., 30.4.28. Ger., 10.5.27).—See B.P. 290,229; B., 1929, 123.

Catalytic apparatus (B.P. 331,468).—See I. Methyl alcohol etc. (B.P. 330,918).—See II. Carbon disulphide (B.P. 333,090).—See VII. Tanning agents (B.P. 331,216).—See XV.

IV.—DYESTUFFS.

Mixer. ZAKARIAS.—See I. Fastness of colours. STERN.—See XIII.

See also A., Aug., 1044, *Physalis* dyes (KUHN and others). Colouring matter of boxthorn berries

ZECHMEISTER and VON CHOLNOKY). 1051, Mercured azo dyes derived from benzidine and *o*-tolidine (McMAHON and MARVEL).

PATENTS.

Dye composition [for domestic use]. W. KRITCHEVSKY, H. C. PRUTSMAN, and E. MORRILL (B.P. 331,491, 31.12.28).—To the usual ingredients of domestic dye preparations is added an alkylated naphthalene-sulphonic acid, *e.g.*, the *isopropyl* or *n*-butyl compound.

C. HOLLINS.

Manufacture of acid wool dyes. I. G. FARBENIND. A.-G. (B.P. 312,175, 21.5.29. Ger., 18.5.28. Addn. to B.P. 299,721; B., 1930, 455).—A sulphonated 4-halogeno-1:8-naphthalic anhydride is heated in water, if desired under pressure, with ammonia or an amine. The resulting 4-aminonaphthalimidesulphonic acids are acid wool dyes; ammonia gives a yellow, *p*-toluidine a reddish-orange.

C. HOLLINS.

Manufacture of acid wool dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,217, 25.3.29).—A diazotised 1-aminoanthraquinone-2-sulphonic acid, carrying in the 4-position a halogen atom or an arylamino-group, is treated at 15–30° with a primary or secondary amine. The diazo group and the halogen, if present, are replaced by the new amino-residue, giving acid wool dyes. Diazotised 4-bromo-1-aminoanthraquinone-2-sulphonic acid is treated with aniline (grey to black), cyclohexylamine (reddish-black), ethylamine (reddish-black), ethylaniline (reddish-grey); diazotised 1-amino-4-anilinoanthraquinone-2-sulphonic acid gives with aniline a grey to black dye.

C. HOLLINS.

Manufacture of blue vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,697, 10.7.29. Addn. to B.P. 320,397; B., 1930, 277).—Halogenated indanthrones are improved in purity and fastness to chlorine by a treatment with manganese dioxide and sulphuric acid. The treatment may follow chlorination without isolation.

C. HOLLINS.

Manufacture of anthraquinone derivatives [vat dyes]. I. G. FARBENIND. A.-G. (B.P. 309,193 and Addn. B.P. 309,454, [A] 6.4.29. Ger., 7.4.28, [B] 10.4.29. Ger., 10.4.28).—Yellow to brown vat dyes are obtained (A) by heating a 2-amino-3-cyanoanthraquinone in a solvent, *e.g.*, quinoline or pyridine, preferably in presence of copper, cuprous cyanide, cuprous bromide, etc., or (B) by heating a 2-halogeno-3-cyanoanthraquinone with cuprous cyanide in a solvent. Examples are 2-amino-3-cyano(or bromo)-anthraquinone (orange-yellow); brominated 2-amino-3-cyanoanthraquinone (yellow); 3-bromo-1:2-diaminoanthraquinone (chocolate-brown); 1:3-dibromo-2-aminoanthraquinone (yellow).

C. HOLLINS.

Manufacture of [vat] dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 309,192, 6.4.29. Ger., 7.4.28).—A halogenated indanthrone (*e.g.*, 3:3'-dibromoindanthrone) is heated with cuprous cyanide in a solvent (quinoline). The product gives blue shades greener than those from the starting material.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,620, 30.4.29).—2-Thiol- or 2:6-dithiol-anthracene is condensed with 1-chloro-, 1-nitro-, or 1-diazoanthraquinone-2-carboxylic acid, and the resulting mono- or di-2-carboxyanthraquinonylthiolanthracene is heated with acid condensing agents (phosphorus pentachloride in trichlorobenzene) to give yellowish-brown vat dyes.

C. HOLLINS.

Manufacture of vat dyes of the anthanthrone series. I. G. FARBENIND. A.-G. (B.P. 311,347, 9.5.29. Ger., 9.5.28. Addn. to B.P. 286,669; B., 1929, 674).—The products of the prior patent containing free amino-groups are acylated. *E.g.*, the compounds from mono- or di-bromoanthanthrone and 1:5-diaminoanthraquinone, and from monobromoanthanthrone and 1:4-diaminoanthraquinone, are benzooylated to give bluish-grey to greenish-blue vat dyes.

C. HOLLINS.

Manufacture of [azo] dyes and application thereof. SOC. CHEM. IND. IN BASLE (B.P. 310,011, 19.4.29. Sw., 19.4.28).—Dyes obtained by coupling diazotised *o*-aminophenol-4:6-disulphonic acid with an unsulphonated arylpyrazolone are prechromed to give orange dyes for wool.

C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 331,247, 21.3.29).—2:3-Hydroxynaphthoic 4-halogeno-2:5-dialkoxyanilide is coupled in substance or on the fibre with a non-sulphonated, non-carboxylated, diazo, tetrazo, or diazoazo compound. The dyes are especially fast to light. The 4-chloro-2:5-dimethoxyanilide is coupled, *e.g.*, with diazotised 4-chloro-*o*-toluidine (red), 2:5-dichloroaniline (orange-brown), *m*-chloroaniline (brown-orange); the 4-bromo-compound gives similar shades.

C. HOLLINS.

Preparation of triarylmethane dyes. F. W. LINCH and E. H. RODD, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,772,516, 12.8.30. Appl., 25.10.28. U.K., 17.11.27).—See B.P. 301,193; B., 1929, 165.

Preparation of triarylmethane dyes. E. H. RODD and F. W. LINCH, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,772,522—3, 12.8.30. Appl., [A] 15.12.26, [B] 2.2.28. U.K., [A, B] 17.4.26. Renewed [A] 30.12.29).—See B.P. 272,321; B., 1927, 598.

Vat dyes of the anthraquinone [dibenzanthrone] series. K. KRAUER and G. HESS, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,774,443, 26.8.30. Appl., 10.10.27. Switz., 20.10.26).—See B.P. 279,479; B., 1929, 165.

β -Naphthylaminoaryloxy-fatty acids (B.P. 331,597).—See III. Diazo-types (B.P. 331,459).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Absorption of iron by various technical celluloses. K. CZAPLA (Faserforsch., 1930, 8, 55–59; Chem. Zentr., 1930, i, 1721).—The differing ability of cellulosic paper materials to absorb iron from solutions is of diagnostic value. Nearly all old papers give a positive reaction (due chiefly to the filling, dye, and

size). Unbleached Finnish spruce cellulose, bleached aspen cellulose, and bleached strawstuff did not absorb iron.

A. A. ELDRIDGE.

Testing of acetylcellulose in the film, lacquer, and artificial silk industries. W. FERMAZIN (Chem.-Ztg., 1930, 54, 605—606).—The water-absorptive power of acetylcellulose decreases with increasing acetic acid content. The absorptive power for 75% alcohol is greater, but decreases when the acetic acid content falls below 52%, thus giving a false impression of the moisture condition of the gel. The viscosity of acetone solutions depends on the acetic acid content, but the viscosity of a 2% solution in formic or acetic acid is proportional to the tensile strength of films of the material and is therefore preferable as a test of quality. The viscosity of solutions decreases rapidly on warming, and steeper curves are obtained with inferior products; similarly, great variation of viscosity with pressure is indicative of poor quality. The solubility (measured by titration of a 1% acetone solution with a non-solvent, *e.g.*, benzene, water) may be correlated with the absorbability and the tensile strength of the dry material, but is independent of the acetic acid content.

E. LEWKOWITSCH.

Reaction variables of the alkaline pulping processes. C. E. CURRAN and M. W. BRAY (Ind. Eng. Chem., 1930, 22, 830—836).—The effect of varying the conditions in the soda-, sulphate-, and semi-chemical pulp processes has been investigated. Temperature affects only the rate of reaction. Increased concentration of reagent increases the rate of reaction, lowers the yield, and gives a weaker pulp. Accelerated rate of reaction combined with good pulp strength is attained by increasing the ratio of reagent to wood, the concentration of the reacting liquor being kept low. The use of sulphide to replace part of the caustic soda gives more easily bleached and stronger pulps than those cooked with caustic soda alone. Small-scale cooks have been made with southern yellow pines, yielding strong kraft pulps of good colour.

T. T. POTTS.

Effect of alkalis on rosin-sizing of paper. CHINTSCHIN (Papier-Fabr., 1930, 28, 517—520).—The effect is to increase the adsorptive power of the cellulose, resulting in increased sizing. The use of Delthirna and other fully saponified sizes is equivalent to the addition of alkali to a normal sizing operation. The effect is particularly of value when sizing takes place in a hard water. A method for the determination of adsorptive power of fibres is outlined.

T. T. POTTS.

Pine rosin in mixtures. DONATH.—See XIII.

See also A., Aug., 1014, Viscosimeter (KÄMPF and SCHRENK).

PATENTS.

Manufacture of cellulose esters. E. F. EHRHARDT and G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 331,260, 31.12.28. Addn. to B.P. 328,588; B., 1930, 655).—Cellulose acetate crotonates are obtained by esterifying cellulose, preferably pretreated with acetic acid with or without catalysts, with crotonic anhydride and acetic acid at 15—75° in presence of sulphuric acid, a sulphonic acid, zinc chloride, or other catalyst.

C. HOLLINS.

Manufacture of unsaturated arylcellulose esters. H. T. CLARKE and C. J. MALM, ASSTS. to EASTMAN KODAK Co. (U.S.P. 1,739,210, 10.12.29. Appl., 4.5.27).—Mercerised and dried cellulose fibre, or viscose rayon, is heated with crotonic acid in chlorobenzene at 155—157°. After 48 hrs. about 5% of crotonyl group has been introduced, and the product may then be acetylated etc. in the usual manner.

C. HOLLINS.

Compositions comprising cellulose derivatives [plasticisers]. BRIT. CELANESE, LTD. (B.P. 312,606, 10.5.29. U.S., 28.5.28).—Alkyl ethers of acylated aminophenols, *e.g.*, acet-*o*-anisidide, acetophenetidides, butyr-*o*-anisidide, or mixtures of these, are used as plasticisers for cellulose derivatives (acetate, propionate, ethyl ether).

C. HOLLINS.

Cellulose derivative compositions. CELLULOID CORP. (B.P. 308,657, 25.3.29. U.S., 23.3.28).—The compositions comprise cellulose esters or ethers (cellulose acetate), unpolymerised compounds containing a vinyl group, *e.g.*, organic or inorganic vinyl esters, styrene, or the compounds obtained by dehalogenating vinyl halides, and auxiliary organic compounds having a solvent or plasticising action on the cellulose derivative, *e.g.*, alcohols, esters, benzene, toluene. Fillers, pigments, gums, resins, etc. may also be added to form lacquers or plastics.

L. A. COLES.

Manufacture of films or sheets. CELLULOID CORP. (B.P. 310,540, 26.4.29. U.S., 27.4.28).—The films etc. are prepared by the extrusion of solutions of cellulose acetate etc., at a temperature somewhat below the b.p. of the solvent employed, into an atmosphere maintained at a raised pressure, the quantity of solvent or mixture of solvents being such that the solution does not flow readily at the ordinary temperature.

L. A. COLES.

Manufacture of artificial filaments, films, and the like. BRIT. CELANESE, LTD. (B.P. 310,046, 18.4.29. U.S., 21.4.28).—A 25% solution of cellulose acetate of acetyl value 52.5—56%, preferably 54.5%, in a mixture of 2—5% or more of methyl alcohol and at least 70% of acetone, which has a low viscosity and an improved capability of being drawn out, is dry-spun at 50—60°.

F. R. ENNOS.

Mixed [wool-acetate silk] fibre. ACETA GES.M.B.H. (B.P. 319,354, 4.9.29. Ger., 22.9.28).—Wool is mixed with artificial fibres of nitroacetylcellulose containing about 1—2% N and acid equivalent to about 54% of acetic acid. Such a mixture may be subjected to the ordinary hot vat treatments, such as are used in wool dyeing, and to carbonisation, as with aluminium chloride, without damage.

F. R. ENNOS.

Manufacture of artificial threads from cellulose esters or cellulose ethers. ACETA GES.M.B.H. (B.P. 310,842, 17.4.29. Ger., 1.5.28).—The tensile strength of the filaments of cellulose esters and ethers produced by the wet-spinning process is increased by the addition to the precipitating bath, containing a salt having a swelling action (thiocyanates, zinc chloride), of a colloid substance such as molasses, sulphite-cellulose lye, a tanning agent, soluble starch, albumin, a soluble synthetic resin, or a solution of degraded keratin.

F. R. ENNOS.

Treatment of textile filaments, yarns, threads, or the like. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,263, 11.1.29).—A saponifying agent (aqueous or alcoholic caustic soda or potash, alkali carbonate, silicate, or phosphate) is intermittently applied by suitable mechanical means to the cellulose ester filaments in the course of their production, so that multi-coloured or cross-dyed effects are obtainable with dyes having a different affinity for the saponified and unsaponified parts of the filaments. F. R. ENNOS.

Manufacture or treatment of (A) materials made of or containing cellulose esters or ethers, (B) textile or other material. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 332,187 and 332,231, 5.1.29).—Structureless cellulose ester products are rendered resistant to the delustering action of hot aqueous liquids (A) by incorporating a solvent or gelling agent (e.g., resorcinol, quinol, cresols, diacetone alcohol, *p*-toluenesulphonamides, glycol ethers, etc.) with the spinning solution, or the precipitating bath, or with the fibre after its production, and then subjecting to the action of dry steam under pressure for upwards of $\frac{1}{2}$ hr.; (B) by treating with dry steam for upwards of $\frac{1}{2}$ hr. at 20–40 lb./in.² above atmospheric pressure; in the latter case the treatment also serves to restore the lustre to products which have been delustred by hot aqueous liquids. F. R. ENNOS.

Manufacture of [crimped] artificial filaments or fibres. S. G. BARKER, and BRIT. RES. ASSOC. FOR WOOLLEN & WORSTED INDUSTRIES (B.P. 332,353, 10.6.29).—Artificial filaments having the crimp or waviness of wool are produced by maintaining the spinning orifice in vibration. F. R. ENNOS.

Improvement of cellulose raw material. ZELLSTOFFFABR. WALDHOF, O. FAUST, and V. HOTTENROTH (B.P. 317,046, 23.7.29. Ger., 9.8.28. Addn. to B.P. 301,088; B., 1930, 456).—The concentration of the alkali lye used in the process is reduced when the cellulose is treated at lower temperatures. F. R. ENNOS.

[Production of] textile fabrics [having crêpe-like or pebbled effects.] H. DREYFUS (B.P. 332,903, 29.4.29).

Imitation fur. G. ROMANE (B.P. 310,467, 3.4.29. Fr., 26.4.28).

Drying of seamless tubular bodies made of cellulose. KALLE & Co. A.-G. (B.P. 319,771, 12.9.29. Ger., 28.9.28).

[Tension device for] manufacture of artificial threads. COMPTOIR DES TEXTILES ARTIFICIELS SOC. ANON., Assees. of W. H. BRADSHAW and G. P. HOFF (B.P. 318,258, 29.8.29. U.S., 1.9.28).

Manufacture of paper, pulpboard, etc. R. MARX (B.P. 333,167, 1.5.29).

Coating of paper. A. E. WHITE. From MEAD PUMP & PAPER CO. (B.P. 333,034, 15.7.29).

High-gloss drying apparatus for papers. I. G. FARBENIND. A.-G., and S. KAMINSKI (B.P. 333,662, 31.5.29).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fastness of colours. STERN.—See XIII.

See also A., Aug., 991, Adsorption of mineral colours by wool fibres (ILJINSKI and KOZLOV).

PATENTS.

Manufacture of azo dyes on the fibre. I. G. FARBENIND. A.-G. (B.P. 308,660, 25.3.29. Addn. to B.P. 306,844; B., 1930, 902).—Textile materials are printed with a paste containing a coupling component and a diazoamino-compound prepared as in the prior patent, and the colour is developed, after drying or steaming if desired, in an acid bath. Examples are pastes containing: 5-nitro-*o*-anisidine \rightarrow dimethylamine with 2:3-hydroxynaphthoic *p*-anisidide (bordeaux); α -aminoanthraquinone \rightarrow dimethylamine with the α -naphthylamide (red); dianisidine \rightarrow piperidine with the anilide (blue); 5-nitro-*o*-anisidine \rightarrow dimethylamine with acetoacet-tolidide (reddish-yellow).

C. HOLLINS.

Manufacture of water-insoluble azo dyes on the fibre [ice colours]. I. G. FARBENIND. A.-G. (B.P. 304,326, 18.1.29. Ger., 20.1.28. Addn. to B.P. 284,247; B., 1929, 637).—2:3-Hydroxynaphthoic β -naphthylamide is coupled on the fibre with diazotised monoaroylated *m*-phenylenediamines carrying in 4- and 6-positions either two halogen atoms or a halogen atom and a methyl group, e.g., 5-chloro-2-benzamido-*p*-toluidine, m.p. 158°, prepared by reduction of benzoic 5-chloro-4-nitro-*o*-toluidide, m.p. 172°, the nitration product of benzoic 5-chloro-*o*-toluidide, m.p. 176°. The dyeings are fast to bucking with 0.35% sodium hydroxide at 1.5 atm. pressure.

C. HOLLINS.

Dyeing of cellulose acetate silk. R. METZGER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,738,660, 10.12.29. Appl., 27.9.27. Ger., 11.7.24).—Acetate silk is dyed with suitable insoluble dyes in conjunction with a sulphonated mineral oil.

C. HOLLINS.

Compositions for decolorising dyed fabrics. W. KRITCHEVSKY (B.P. 331,194, 22.12.28).—The composition, for domestic use, comprises a hyposulphite or formaldehyde-sulphoxylate together with a solid acid or a salt having an acid reaction (oxalic or tartaric acid, aluminium sulphate, sodium hydrogen sulphite) and a sodium alkyl-naphthalenesulphonate. Zinc dust may replace the hyposulphite.

C. HOLLINS.

Rendering fibres insectproof [mothproofing]. R. M. RITTER (B.P. 313,043, 26.4.29. U.S., 5.6.28).—Alkyl-naphthalenesulphonic acids, preferably fixed by means of mordant metal salts, are used to protect animal fibres etc. against moths. The butyl, amyl, and propyl compounds are mentioned.

C. HOLLINS.

Dyeing process. A. ESCHACH and J. P. WORMS (U.S.P. 1,774,428, 26.8.30. Appl., 20.11.23. Ger., 25.11.25).—See B.P. 230,128; B., 1925, 352.

Dyeing cellulose ester and ether. L. G. LAWRIE, F. W. LINCH, and E. H. RODD, Assrs. to BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,772,515, 12.8.30. Appl., 21.6.28. U.K., 5.7.27).—See B.P. 297,897; B., 1928, 891.

Dyeing and printing cellulose esters and ethers. E. FISCHER and C. E. MÜLLER, Assrs. to GEN. ANILINE

WORKS, INC. (U.S.P. 1,765,142, 17.6.30. Appl., 20.4.27. Ger., 23.4.26).—See B.P. 269,934; B., 1928, 121.

Printing with vat dyes. E. PFEFFER, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,744,140, 21.1.30. Appl., 22.1.29. Ger., 26.1.28).—See B.P. 304,787; B., 1930, 416.

Production of [pattern effects on] fabrics or articles [containing cellulose acetate]. G. H. ELLIS and R. J. MANN, ASSTS. to CELANESE CORP. OF AMERICA (U.S.P. 1,773,975, 26.8.30. Appl., 17.5.27. U.K., 15.6.26).—See B.P. 277,414; B., 1927, 874.

[Re-lustring] treatment of threads, fabrics, or other materials composed of or containing artificial filaments and products. C. W. PALMER and S. M. FULTON, ASSTS. to CELANESE CORP. OF AMERICA (U.S.P. 1,774,184, 26.8.30. Appl., 16.11.25. U.K., 26.5.25).—See B.P. 259,266; B., 1926, 976.

Dye jigger machines. ASSOCIATED DYERS & CLEANERS, LTD., and H. J. CRICK (B.P. 333,447, 9.10.29).

Dye jigs. J. MACE (B.P. 333,366, 13.7.29).

Apparatus for dyeing piece goods. A. A. HUDSON (B.P. 332,979, 14.5. and 25.7.29).

[Shrinking] treatment of cloth. A. A. LINDQVIST (B.P. 319,236, 16.5.29. Swed., 18.9.28).

Singeing of textile fabrics and yarns. J. CHADWICK & Co., LTD., and F. SHARPLES (B.P. 333,084, 18.9.29).

Deodorising hair, feathers, etc. [by means of ozone]. W. W. TRIGGS. From AMER. HAIR & FELT Co. (B.P. 333,197, 4.4.29).

Azo dyes [on wool] (B.P. 310,011).—See IV. **Mixed fibre** (B.P. 319,354).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Reaction of calcium cyanamide with carbon dioxide at high temperatures. H. H. FRANK and B. MEPPEN (Z. angew. Chem., 1930, 43, 726–732).—At 500–1100° carbon dioxide reacts with calcium cyanamide as follows: $2\text{CaCN}_2 + \text{CO}_2 = 2\text{CaO} + 2\text{N}_2 + 3\text{C}$. At the higher temperatures the liberated carbon is converted into carbon monoxide by excess of carbon dioxide. Carbon monoxide reacts slowly with cyanamide below 1000° as follows: $\text{CaCN}_2 + \text{CO} = \text{CaO} + 2\text{C} + \text{N}_2$.

A. R. POWELL.

Fluoaluminates of the alkali metals. R. H. CARTER (Ind. Eng. Chem., 1930, 22, 888–889).—The fluoaluminates of sodium, potassium, lithium, and ammonium were prepared in the laboratory and their solubilities and the p_{H} of the solutions determined. The potassium salt can be readily obtained with a yield of 90% by adding hydrofluoric acid to a solution of aluminium sulphate containing potassium carbonate. The mixture is boiled and the fluosilicate separates as a gelatinous precipitate. If hydrofluosilicic acid is used, silica is precipitated, giving a lighter compound suitable for dusting as insecticide. If an alkaline solution of alumina is used as starting point products are obtained giving a practically neutral reaction to phenolphthalein, and these should be safer in use than are the fluosilicates.

C. IRWIN.

Solubilities of fluosilicates in water. R. H. CARTER (Ind. Eng. Chem., 1930, 22, 886–887).—The solubility curves of sodium, potassium, and barium fluosilicate between 0° and 100° were determined. The solubilities decrease in the order mentioned. Pure calcium fluosilicate is much more soluble than are these salts and is too soluble for use as insecticide. The calcium fluosilicate of commerce, which has been tested in this way, appears to contain only a small percentage of fluosilicate.

C. IRWIN.

Determination of small quantities of carbon monoxide. ANON. (Jahresber. VII., Chem.-Tech. Reichsanst., 1928, 195–201; Chem. Zentr., 1930, i, 1977–1978).—By the use of dry carbon monoxide, a relatively high gas velocity, and finely-divided cupric oxide deposited on quartz, quantitative oxidation to carbon dioxide occurs, and the adsorption of this gas on the small amount of cupric oxide is minimal. The gas is completely removed by passing air free from carbon dioxide for 20 min. at 350°.

A. A. ELDRIDGE.

Nitrous oxide for anaesthesia. V. E. HENDERSON and G. H. W. LUCAS (Canadian Chem. Met., 1930, 16, 211–212).—Passage of nitrous oxide through ordinary litmus solution under the conditions of the U.S.P. test causes a colour change by displacing carbon dioxide. Since, in addition, litmus is not very sensitive towards ammonia, the following alternative test is proposed. The change in p_{H} produced by bubbling 2000 c.c. of the gas through 100 c.c. of freshly boiled 0.00005% methyl-red solution should not be greater than that produced by 0.1 c.c. of 0.01N-hydrochloric acid or -ammonia. Damage to the lungs, observed in certain animal experiments, was due to dryness of the gas, and not to any harmful constituent.

H. E. F. NOTTON.

Utilisation of waste steam for sulphate production. REUTER.—See I. **Diffusion of insoluble carbonates.** RAE.—See VIII. **Determination of free lime.** BESSEY.—See IX. **Manganese arsenate insecticide.** DEARBORN.—See XVI.

See also A., Aug., 979, **Phosphorescent zinc sulphide** (COUSTAL). 997, **System sodium sulphate-sodium carbonate-water in natural soda lakes** (KURNAKOV and MAKAROV). 1003, **Synthesis of ozone** (BREWER and WESTHAVER).

PATENTS.

Denitrating mixtures of nitric and sulphuric acids and concentrating nitric acid. J. L. BENNETT, ASST. to HERCULES POWDER Co. (B.P. 333,264, 5.2.29. U.S., 26.10.28).—A mixture of aqueous nitric and sulphuric acids or other dehydrating agent, such as phosphoric acid or anhydrous sodium sulphate, is partly denitrated by passing it down a tower in counter-flow to a mixture of nitric acid vapour and steam. The partly denitrated mixture is heated in a vessel provided with steam coils, so as to generate steam and nitric acid vapour, a portion of the steam being used to denitrate further quantities of the mixture and to separate the nitric acid from the balance of the steam. Only sufficient steam is introduced into the tower to prevent undue dilution of the nitric acid, the vapours of which, on leaving the top of the tower, are condensed in another vessel.

W. J. WRIGHT.

Apparatus for decomposition of thiocyanic acid and its salts. C. J. HANSEN, Assee. of H. KOPPERS A.-G. (B.P. 310,549, 27.4.29. Ger., 28.4.28).—Corrosion of chromium-nickel steel apparatus, employed for the decomposition of thiocyanic acid or its salts by acid at normal or high temperatures for the production of ammonium salts, is avoided if sulphurous acid, or a liquor containing it in a free or combined state, is used for the decomposition. W. J. WRIGHT.

Manufacture of complex fluoboro-organic acids and salts thereof. I. G. FARBENIND. A.-G. (B.P. 316,987, 7.8.29. Ger., 7.8.28).—Boric acid is dissolved in hydrofluoric acid, and an organic acid is added to the solution, or hydrofluoric acid is caused to act on a mixture of boric acid and an organic acid or on a compound of boric acid with an organic acid, *e.g.*, boracetic anhydride. W. J. WRIGHT.

Catalytic oxidation of ammonia. SELDEN Co., Assees. of A. O. JAEGER (B.P. 313,153, 10.5.29. U.S., 9.6.28).—The catalyst comprises a zeolite-like body the base of which has been exchanged with a metal oxide having catalytic activity and the resulting product treated with a solution of a salt of a metal acid of group V or VI; *e.g.*, sodium cerium iron aluminosilicate is treated with a dilute solution of a manganese, silver, copper, cobalt, manganese, lead, or zinc salt and then with a 1% solution of ammonium vanadate, tungstate, molybdate, or chromate. A. R. POWELL.

Manufacture of ammonium chloride. J. I. BRONN, and CONCORDIA-BERGBAU A.-G. (B.P. 333,098, 4.10.29).—Dilute hydrochloric acid solutions are boiled, and the vapours are superheated to 150° and mixed with ammonia vapours similarly superheated. Solid ammonium chloride is deposited and the hot steam obtained as a by-product may be utilised in the process described in B.P. 308,028 (B., 1929, 433). More efficient collection of the ammonium chloride is effected by carrying out the reaction in a centrifuge. A. R. POWELL.

Preparing magnesium hydrate tablets. W. C. MCGOWAN, Assr. to MILNESIA INTERNAT., INC. (B.P. 310,919, 5.2.29. U.S., 3.5.28).—To obtain tablets of uniform smoothness, which readily disintegrate in the mouth without grittiness, the moist hydroxide is granulated, diluents such as calcium carbonate or starch are added, and the product is formed into tablets. W. J. WRIGHT.

Filtering material. H. BLUMENBERG, JUN. (U.S.P. 1,734,197, 5.11.29. Appl., 15.12.27).—Copper sulphate and calcium hypochlorite are ground together in molecular proportions. This material is mixed with cellular material such as diatomaceous earth or pumice in the ratio 100 : 30. Mineral distillates may be purified by treatment with 0.5–5% of this mixture, water with 25 lb. per million gals., and sewage or industrial waste liquors with 100–500 lb. per million gals. T. A. SMITH.

Manufacture of metal carbamates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,584, 20.3.29).—Calcium, barium, and zinc carbamates are obtained by double decomposition of salts of these metals with ammonium carbamate in ethyl- or methyl-alcoholic

solutions. Sodium carbamate is similarly obtained from solutions of sodium chloride in liquid ammonia.

A. R. POWELL.

Manufacture of solid carbon dioxide. K. C. PEGGS (B.P. 333,212, 6.5.29).—Instead of allowing liquid carbon dioxide to expand through valves or orifices, it is caused to expand, when the process has started, through the mass of the already formed solid product, thereby filling the pores and producing a denser material. W. J. WRIGHT.

Production of carbon disulphide. I. G. FARBENIND. A.-G. (B.P. 333,090, 24.9.29. Ger., 17.11.28).—To enable the residue from the retorts used in the manufacture of carbon disulphide to be removed continuously by mechanical means, layers of granular material, *e.g.*, broken brick, pumice, lava, coke, are introduced in alternate layers with the wood charcoal.

A. R. POWELL.

Catalytic oxidation of ammonia. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,765,352, 17.6.30. Appl., 14.4.28).—See B.P. 309,583; B., 1930, 764.

Chemical decomposition of crude potash salts. F. RÜSBERG, Assr. to KALI-CHEM. A.-G. (U.S.P. 1,744,040, 26.8.30. Appl., 4.9.29. Ger., 6.9.28).—See B.P. 314,725; B., 1929, 681.

Manufacture of calcium molybdate. A. KISSOCK (U.S.P. 1,763,712, 17.6.30. Appl., 6.11.26).—See B.P. 280,240; B., 1929, 393.

Preparation of magnesium dichromate. G. KRÄNZLEIN and A. VOSS, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,774,018, 26.8.30. Appl., 10.5.27. Ger., 22.6.25).—See F.P. 617,235; B., 1927, 814.

Production of aluminium compounds. T. R. HAGLUND, Assr. to INTERNAT. PATENT CORP. (U.S.P. 1,772,936, 12.8.30. Appl., 6.4.25. Swed., 17.4.24).—See B.P. 232,549; B., 1925, 997.

Catalysts for the catalytic production of methyl alcohol and other oxygenated organic compounds from oxides of carbon and hydrogen. R. G. FRANKLIN, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,774,432, 26.8.30. Appl., 13.10.27. U.K., 15.11.26).—See B.P. 290,399; B., 1928, 523.

Production of oxygen and nitrogen. G. CICALI (U.S.P. 1,772,856, 12.8.30. Appl., 20.6.25. It., 16.2.25).—See B.P. 248,977; B., 1926, 406.

Precipitation of boron. A. E. VAN ARKEL, Assr. to N.V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,774,410, 26.8.30. Appl., 18.8.26. Holl., 5.10.25).—See B.P. 264,953; B., 1927, 252.

Reduction of carbon monoxide (U.S.P. 1,741,306—8). Catalyst for methyl alcohol synthesis (U.S.P. 1,738,971).—See III. Salts of antimonie and stibinic acids (B.P. 309,184).—See XX.

VIII.—GLASS; CERAMICS.

Progress report on effect of furnace gases on quality of enamels. A. I. ANDREWS and E. A. HERTZELL (J. Amer. Ceram. Soc., 1930, 13, 522–529).—The effects of firing ground- and cover-coat enamels in atmospheres of sulphur dioxide, city gas, nitrogen,

and carbon dioxide were studied. Variations in the composition of ground-coat enamels had little effect on resistance to attack by sulphur gases. Similar variations in the cover coats, however, were more effective, but further work is needed to define the relationship clearly. The work showed that the presence of unburned or partially burned gas and sulphur dioxide in furnace gases is detrimental to enamels. F. SALT.

Formation of seeds and bubbles in glass pots. W. M. COHN (J. Amer. Ceram. Soc., 1930, 13, 555—559).—The production of bubbles or seeds in glass is related to the variation in the porosity of the pots, which increases up to a certain maximum and then falls to zero. For the removal of all gases from the glass melt, the refractory material must have a certain porosity throughout the melting operation. Alternatively, porous fireclay rods may be introduced into the melt. F. SALT.

Felspars and their peculiarities as ceramic solvents. A. S. WATTS (J. Amer. Ceram. Soc., 1930, 13, 550—554).—Brief notes are given on the constitution and mode of occurrence of felspar minerals and on associated minerals. Chemical analysis is shown to be an unreliable guide to the classification of felspars. A method of classification is proposed which is based on the fusion behaviour of samples of felspars, with and without additions of quartz, kaolin, and ball clay. F. SALT.

Impurities in commercial felspars often overlooked by enamellers. J. R. CRANDALL (J. Amer. Ceram. Soc., 1930, 13, 530—531).—Eight commercial felspars, varying in chemical composition from 13.32 to 9.06% K_2O and from 4.14 to 2.08% Na_2O , were examined for impurities. Fusion tests showed small black or brown specks on the fused surface of certain felspars, due to the presence of garnet, hornblende, tourmaline, or biotite mica. Muscovite mica readily goes into solution and therefore gives no trouble in the finished enamel. F. SALT.

Acid-resisting, white, dry-process cast-iron enamels. A. I. ANDREWS (J. Amer. Ceram. Soc., 1930, 13, 509—521).—In the development of such enamels, additions were made systematically to two simple three-component systems, viz., the sodium oxide-boric oxide-silica and the sodium oxide-lead oxide-silica systems. Two tests were applied to the enamels as developed: (a) the "acid spot" test, using a 7% solution of citric acid; (b) the solubility test, using hot 20% hydrochloric acid. The best enamel developed from the point of view of acid-resistance contained 18 Na_2O , 34 PbO , 48 SiO_2 , with 15% SnO_2 . The fineness of grinding, firing temperature, smelting conditions, and rate of cooling are without effect on the acid-resistance of dry-process enamels. Fluorine compounds are necessary to produce opacity, but they reduce the resistance to acids. F. SALT.

Lead content of the glazes on pottery manufactured in Ukraine. W. A. UGLOW, W. A. REBERG, and M. W. BOLTINA (Z. Unters. Lebensm., 1930, 59, 379—394).—The influence of boron, lead oxide, soda, potash, etc. on the properties of glazes is briefly reviewed.

An account is given of the preparation and application of lead glazes of low m.p., and attention is drawn to the dangers connected with this method. The methods used for testing glazes for soluble lead vary considerably as regards the nature and concentration of the acid and the time and temperature of extraction and the consequent discrepancies in the reported values are enumerated. The toxicity of Ukraine pottery is compared with that of ware from other countries. The utensils were tested by filling with 4% acetic acid and boiling for $\frac{1}{2}$ hr., lead being determined in the extract by adding a known amount of potassium dichromate and titrating the excess iodometrically. More than 600 analyses were made on all classes of ware. After six extractions some articles still gave appreciable amounts of lead, although the first (average 102 mg./litre) was always higher than any subsequent extraction (average 37.2 mg./litre). Individual samples showed great variation owing to lack of uniformity in the composition and methods of applying the glaze. The results indicated that many kinds of pottery gave undesirably large amounts of soluble lead, and recommendations are made for mitigating the trouble, such as a preliminary boiling with vinegar, whereby the amount of lead subsequently dissolved would be reduced by 60—70%. H. J. DOWDEN.

Grain size of ceramic bodies. V. SKOLA (Feuerfest, 1930, 6, 81—85).—The problem of grading ground material so as to produce a mixture of maximum density was studied both mathematically and practically. In the ideal case, the voids formed by one fraction will be filled by the next smaller fraction. The grading which most nearly approached this condition was: 4—2 mm. 43.9%, 1—2 mm. 10.9%, 0.5—1 mm. 12.5%, 0.2—0.5 mm. 11.1%, 0.12—0.2 mm. 9.4%, 0.06—0.12 mm. 6.9%, and < 0.06 mm. 5.2%. F. SALT.

Effect of various substances on diffusion of kaolin and certain insoluble carbonates. J. RAE (Pharm. J., 1930, 125, 223—224).—An account of some tests on the influence of added soluble substances on the rate of settling of kaolin and of bismuth, calcium, and magnesium carbonates in various media. S. I. LEVY.

Method for determining the loss of weight of clays during firing. W. R. MORGAN (J. Amer. Ceram. Soc., 1930, 13, 561—565).—By means of simple apparatus, consisting essentially of a balance from which a sample could be suspended in an electric furnace below, the progress of dehydration and oxidation was determined by the loss of weight of the sample during the heating operation. With different clays and bodies, the total loss of weight varied considerably (from 4 to 11%), but the losses in any temperature range expressed as a percentage of the total weight lost at 982° showed a much closer relationship. F. SALT.

Clay sewer-pipe manufacture. IX. Method of checking flue-gas analyses and its application to ceramic kilns. E. A. HERTZELL. **X. Measurement of moisture loss in kilns.** R. L. BLESSING. **XI. Cold-junction compensator for outdoor pyrometer installations.** R. E. ARNOLD (J. Amer. Ceram. Soc., 1930, 13, 566—570, 571—579, 580—586; cf. B., 1929, 644).—IX. The use of Bato's formula to

check the results of gas analysis obtained with the Orsat apparatus is explained. Calculations were applied to four tests on kilns fired with coal, natural gas, and fuel oil.

X. Data and experiences are presented on an attempt to determine, by loss-of-weight methods, the completion of the water-smoking period and to ascertain the point at which increased rate of heating would be safe. The results indicated two essentials for correct water-smoking, viz., strong draught during the early stages, and accurate control of the top temperatures. Measurement of loss of weight did not indicate the critical point in the water-smoking operation.

XI. A simple and inexpensive instrument for multiple-kiln installations is described. F. SALT.

Corundum and silicon carbide. R. SCHNEIDLER (Chem.-Ztg., 1930, 54, 625—627).—Batch working is preferred in the electrothermic manufacture of corundum because the removal of the impurities in the bauxite is easier and the physical structure of the product better. Furnaces are usually constructed of strong sheet iron, unlined, as the slag forms an excellent protection. The floor is covered with tar, coal, etc. Coke is laid upon this and the bauxite gradually added and melted by moving the electrodes, by hand or automatically, the current being kept at 6000 amp. and 90—120 volts. After completion of the process the cooled block of corundum is lifted out vertically. The usual yield is 55—60% of first-class corundum, calculated on bauxite. The usual corundum of commerce contains 95—97% Al_2O_3 ; inferior qualities are darkened in colour by iron and titanium. Silicon carbide is produced by the reduction of quartz sand containing at least 98% SiO_2 with good-quality coke or anthracite at 1800°. It is decomposed by heating to 2200°, such decomposition being catalysed by the presence of iron or other metals, which must therefore be excluded as far as possible. A resistance furnace built of refractories with a resistor core of coke is used and the current must be kept uniform. C. IRWIN.

See also A., Aug., 1010, Decomposition of aluminous silicates for analysis (FINN and KLEKOTKA).

PATENTS.

Stratified bodies, e.g., strengthened glass. TRIPLEX SAFETY GLASS CO., LTD., and J. WILSON (B.P. 332,551, 20.4.19 and 30.1.30).—A sheet of cellulose ester plastic, e.g., celluloid, is united to glass already coated with gelatin, by immersing the surfaces in an inert liquid of relatively high b.p. and heating and pressing the plates together. The inert liquid, e.g., cyclohexane, may contain also solvents, e.g., glycol ethers, tolyl phosphate, alkyl phthalates.

C. A. KING.

Firing of ceramic ware. A. S. WATTS (B.P. 332,692, 7.6.29).—The various operations of twice firing ceramic ware together with the necessary intervening stages of cooling are conducted in a tunnel kiln of sectional construction. The time of heat-treatment in the different zones is controlled by the length of the zones, which may be altered by the addition or removal of further kiln sections. C. A. KING.

Burning of bricks, tiles, and the like. A. HEUER (B.P. 310,315, 8.2.29. Ger., 23.4.28).—Bricks etc. of normal porosity and high strength are made by mixing intimately a high-grade fuel, e.g., coke, in a finely-divided state with the raw materials in nearly sufficient quantity to provide the necessary heat for the burning process. Less than this quantity of fuel may be mixed with the materials and the requisite additional fuel is then placed between the bricks etc. [Stat. ref.]

F. SALT.

Preventing deposition of carbon in and upon ceramic materials which come into contact with [furnace] gases at high temperatures. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 330,561, 7.3.29).—Copper or a copper compound is added to the materials (e.g., refractory bricks) during any stage of manufacture, or to the finished product, by impregnating, spraying, brushing, etc. F. SALT.

Treatment of carbonised clays and of refractory units prepared from such clays. W. McBRIDE (B.P. 330,745, 24.5.29).—The free carbon contained in refractory units made from carbonised clays produced by the process described in B.P. 149,440 (B., 1920, 658A) is removed by oxidation, preheated atmospheric air being introduced into the kiln during or immediately after the firing of the units. F. SALT.

Tunnel kiln. P. A. MEEHAN, ASSR. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,764,460, 17.6.30. Appl., 3.4.28).—See B.P. 312,786; B., 1929, 645.

Processes and apparatus for tempering glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY, ET CIREY (B.P. 333,839—40 and 333,843, [A] 30.11.29, [B, C] 2.12.29. Fr., [A] 7.8.29, [B] 8.8.29, [C] 21.9.29).

[Machine for] manufacture of [hollow] glass-ware. A. SCHILLER (B.P. 312,159, 7.5.29. Ger., 21.5.28).

Treatment of cast glass plates and sheets [to prevent buckling]. A. BRANCART (B.P. 333,500, 14.2.30. Belg., 23.2.29).

[Laminated] safety glass sheet and its manufacture. J. NEWTON (B.P. 333,242, 8.2.29).

Manufacture of ceramic bodies [from strands]. HERMSDORF-SCHOMBURG ISOLATOREN GES. (B.P. 317,364, 24.5.29. Ger., 14.8.28).

Grinding wheels. W. W. TRIGGS. From NORTON Co. (B.P. 333,658, 29.5.29).

IX.—BUILDING MATERIALS.

Glycerol method for determination of free lime. G. E. BESSEY (J.S.C.I., 1930, 49, 360—362 r).—A modification of the glycerol method of determining free lime in cements and sand-lime bricks is described, in which the free lime is completely extracted by warming for several hours at 60—80° with glycerol, and is then titrated, after dilution with alcohol, by an alcoholic solution of benzoic acid, using phenolphthalein as indicator. It is suggested that this modification has certain advantages over previous methods and is simpler to carry out. The applicability and accuracy of this and previous methods are discussed.

Relation of the strengths of cements of various binding powers. BURCHARTZ (Mitt. Materialprüf., 1930, 124—125).—The tensile, compression, and bending strengths of 1:3 and 1:5 mixtures of three different cements with two types of sand have been determined. The relation of tensile to bending strength is best in cement of low binding power, whereas the relation of tensile to compression strength is best with cement of high binding power. The use of carefully graded sand in making cement mortars has a more beneficial effect when low-grade cement is used; high-grade cement gives a good mortar with most types of sand. The effect of varying the quantity of water and of different shapes of test piece on the values of the various strengths has also been determined. A. R. POWELL.

Determination of free calcium hydroxide in set cements. Calorimetric method. G. E. BESSEY (Dept. Sci. Ind. Res., Building Res. Tech. Paper 9, 1930, 25 pp.).—The determination of free calcium hydroxide is dependent on the difference in the heat of hydration of the material when heated to 350° and 550°. Preliminary tests proved that no sulphates were decomposed below 1000° nor carbonates below 550°, the loss at 350—500° being due only to the dissociation of calcium hydroxide. About 10 g. (W) of cement passing 100-mesh are heated in an electric furnace; cooled, and sealed in a tube, which is broken in a calorimeter after stable conditions have been attained. Duplicate determinations of the heat evolved by material heated to 350° (Q_1) and 550° (Q_2) permit the computation of free CaO from the formula: $(Q_2/W_2 - Q_1/W_1)100fk/273$, in which f is a factor for incomplete decomposition of $\text{Ca}(\text{OH})_2$ at 530° and any adsorption of carbon dioxide, and k a factor involving the combination of calcium oxide with any other materials during ignition. The three correction factors may be condensed to 0.41 when applied to Portland cement. C. A. KING.

Mixer. ZAKARIAS.—See I.

See also A., Aug., 1010, **Decomposition of aluminous silicates for analysis** (FINN and KLEKOTKA).

PATENTS.

Rotary kilns. E. G. STONE (B.P. 332,761, 6.8.29).—A kiln for producing cement consists of two or more separate rotary-kiln sections arranged in series. All except the final section are divided into smaller tubes and the last section is mounted to tilt longitudinally. The first or slurry section is rotated at greater speed than are other sections. C. A. KING.

Manufacture of pavement materials. NAAML. VENN. KONINKLIJKE STEARINE KAARSENFABRIEK GOUDA (B.P. 333,433, 27.9.29. Holl., 4.2.29).—Increased adhesion is secured if broken road material is coated first with molten bitumen or a bitumen mixture and then with a bitumen emulsion. Coagulation of the emulsion is regulated by adding cement or other suitable filler. C. A. KING.

Hardening of alberene stone. P. MAHLER, Assr. to ALBERENE STONE Co. (U.S.P. 1,738,956, 10.12.29. Appl., 26.2.27).—Any natural stone containing one or more groups of silicates, e.g., soapstone, is hardened

by subjecting it, under controlled conditions as to time and temperature, to successively increasing temperatures, in order to drive off the water of constitution of the different silicate groups. F. SALT.

Dental cement. O. SIMON (B.P. 333,325, 27.5.29).—The hardness and density of silicate or zinc phosphate cements are increased by adding an insoluble material, e.g., wax or bitumen, together with acid or salts of aluminium, calcium, magnesium, strontium, or beryllium. The wax or oil may first be treated with an acid and the excess afterwards neutralised, and an organic solvent may be used to facilitate mixing. C. A. KING.

Wood veneer and its preparation. W. H. WOOD (U.S.P. 1,738,132, 3.12.29. Appl., 7.4.24. Renewed 26.7.28).—To facilitate the transport and storage of thin wood veneer and to maintain its pliable nature etc., it is impregnated with a water-soluble sulphate, which forms crystals containing water of crystallisation, preferably by soaking it in a hot, concentrated solution of magnesium sulphate. F. SALT.

Manufacture of [sand-faced] building bricks and similar clay or like products. C. S. JILLINGS (B.P. 333,346, 25.6.29).

Drying kilns [for lumber etc.]. B. F. STURTEVANT Co., Assees. of H. F. HAGEN (B.P. 333,566, 14.2.29. U.S., 1.10.28).

[Photographically] imitating [the grain in] marble or the like. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 313,007, 4.6.29. Ger., 4.6.28).

[Photographic] production of printing plates in imitation of the fibre in wood. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 310,897, 2.5.29. Ger., 2.5.28).

Concrete mixer. CHAIN BELT Co. (B.P. 333,138, 13.1.30. U.S., 11.1.29).

Rotary concrete and the like mixing machines. FILS DE J. WEITZ (B.P. 333,492, 6.1.30. Fr., 16.2.29).

Manufacture of sheets [e.g., wall-board] from plastic material. A. E. WHITE. FROM UPSON Co. (B.P. 333,161, 3.4.29).

Drying or like treatment of timber and other materials. C. GOODALL (B.P. 333,403, 20.8.29).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Rotary cylindrical furnace for melting foundry cast iron. A. LE THOMAS (Rev. Mét., 1930, 27, 256—264).—A 5-ton rotary cylindrical furnace for melting cast iron by means of a powdered-fuel flame injected at one end of the longitudinal axis is described. It comprises a riveted steel plate shell lined with a silicious refractory 20 in. thick, which will withstand 120—150 fusions. For high-temperature fusions the furnace is provided with a preheater for the air, by the use of which temperatures up to 1650° are readily obtained. This type of furnace has the advantage over the cupola type that there is only a very small oxidation of the

iron, and the composition of the cast metal may therefore be more accurately regulated. The fuel consumption averages 16–18% of the weight of molten metal obtained.

A. R. POWELL.

Value of an iron ore or other material added to the blast furnace as a function of the yield of cast iron. J. VALLET (Rev. Mét., 1930, 27, 248–255).—Expressions are derived for determining the market value of iron ore based on its composition and the yield of cast iron obtained from it; the effect of the nature and composition of the coke on the results is discussed.

A. R. POWELL.

Change in composition of the cementite constituent during the recovery of special steels. A. PORTEVIN and P. CHEVENARD (Compt. rend., 1930, 191, 408–411; cf. B., 1925, 504).—The authors' method of dilatometric analysis has been applied during the recovery of a steel containing 1.5% C and 2% Mn (previously rendered austenitic by water-tempering at 1180°) heated at the rate of 250°/hr. to t° (100–700°) and maintained at t° for 7 hrs. Curves in which t is plotted against dilatability, total expansion due to the thermal cycle, gain in hardness, resistivity, remanent magnetisation (field 900 gauss), and coercive field show the changes in concentration and proportion of the phases cementite, α - and γ -solid solutions. Below 200° a carbide (Curie point 120°) is precipitated, the iron returning from the γ - to the α -state at 200–225° with precipitation of another carbide (Curie point 170°). Above 260° the transformation austenite \rightarrow martensite-carbide aggregate occurs, the proportion of carbide becoming progressively smaller as t rises. At 400° the steel is completely in the form of sorbite, and at higher temperatures the carbide precipitated by heating becomes richer in manganese at the expense of the ferrite.

J. GRANT.

Absorption of sulphur and phosphorus by iron and steel in welding with impure acetylene. ANON. (Jahresber. VII., Chem.-Tech. Reichsanst., 1928, 83–86).—The amounts of sulphur and phosphorus entering the metal are small and without effect. Chemical purification of the acetylene is unnecessary.

A. A. ELDRIDGE.

Sulphur in cast iron. H. H. EBERT (Zentral-Europ. Giesserei-Ztg., 1930, 3, 3–4; Chem. Zentr., 1930, i, 1997).—Methods of desulphurisation are discussed.

A. A. ELDRIDGE.

Effect of the state of the cementite on the heat sensitivity, the tendency to core-hardening, and formation of hardness fractures in carbon steels. S. STEINBERG (Stahl u. Eisen, 1930, 50, 1164–1166).—The effect of various heat treatments on the micro-structure and mechanical properties of steels with 0.8 and 1.3% C has been investigated. The hypoeutectoidal steel after annealing at 950° and slow cooling becomes hard and tough after hardening at 735° and quenching, but hardening at 750–800° produces a more coarsely crystalline structure and the metal becomes brittle with a tendency to the development of hair-cracks. The hypereutectoidal steel requires very slow cooling after annealing at 950° and a hardening temperature of 800–850° to produce a tough structure. Lamellar

cementite causes brittleness and hair-cracks to develop on hardening, whereas granular cementite or troostite imparts a high degree of toughness to steel.

A. R. POWELL.

Effect of small additions of copper or nickel on the thermal expansion and growth of cast iron. O. BAUER and H. SIEGLERSCHMIDT (Mitt. Materialprüf., 1930, 110–111).—Cast iron containing 3.15% C, 1.12% Si, and 0.114% S had a coefficient of thermal expansion of 0.0000141, and after twice heating to 750° it showed a permanent elongation of 0.201% due to carbide decomposition. Addition of 0.55% Cu or 0.48% Ni had no effect on the thermal expansion, but reduced the permanent elongation to 0.155% and 0.153% respectively.

A. R. POWELL.

New methods in the study of corrosion and protection of metallurgical products against corrosion. J. COURNOT (Bull. Soc. chim., 1930, [iv], 47, 802–825).—Theories of corrosion and the experimental methods of investigation available are reviewed. A résumé is given of methods of protection of metals from corrosion, especially by coating with other metals or with paints, and by anodic treatment. E. S. HEDGES.

Matte smelting of oxidised copper ores in reverberatories. ST.-MAURICE (Rev. Mét., 1930, 27, 214–222).—Notes on the method of calculating the charge for matte smelting of silicious oxidised copper ore relatively low in iron and high in manganese are given. The ore is smelted with gypsum and coke mixed with a small amount of crude petroleum to prevent loss by dusting. The amount of gypsum added is sufficient to produce a matte containing more than 50% Cu, and the quantity of coke is adjusted to reduce ferric and manganic oxides to the ferrous and manganous states and the gypsum to calcium sulphide. The slags obtained average 0.2–0.3% Cu and have a very low iron content.

A. R. POWELL.

Copper alloy systems with an α -phase having variable limits and their use for the hardening of copper. M. G. CORSON (Rev. Mét., 1930, 27, 83–101, 133–153, 194–213, 265–281).—An account is given of the researches leading up to the development of the so-called "Corson alloys" which have been the subject of numerous patents in recent years. The alloys consist of copper with small percentages of chromium, cobalt, silicon, or silicides of nickel, cobalt, iron, or chromium, all of which constituents have a higher solid solubility at high temperatures than at the ordinary temperature. Between 20° and 1000° the solid solubility of chromium in copper varies from 0.05 to 0.8%, of cobalt from 0.35 to 3.4%, of Co_2Si from 0.3 to 3.3%, of Ni_2Si from 0.7 to 8.2%, of iron from 0.2 to 3.8%, and of silicon from 2.8 to 7.5%. Alloys containing silicides are best hardened by quenching from 800–1000° and ageing at 250–450°; these alloys should contain a slight excess of the second metal over that required to form its silicide so as to ensure maximum hardening on ageing without the serious fall in electrical conductivity which is produced by the retention of silicon in solid solution. Photomicrographs of numerous alloys of this type are reproduced, and the effect of heat treatment on the

mechanical and electrical properties and on the structure is illustrated by graphs and photographs.

A. R. POWELL.

Cyanidation of cupriferous gold ores. W. G. CLARKE and B. H. MOORE (*Chem. Eng. Min. Rev.*, 1930, 22, 353—355).—Gold ores containing copper and other carbonates cannot be efficiently cyanided in the usual way owing to the high cyanide consumption, and an acid leach is impracticable owing to the presence of calcium and magnesium carbonates. Good extraction of gold from an Australian ore of this type has been obtained by roasting at 600° for 1½ hrs., grinding the product finely, passing it over amalgamating plates to remove coarse gold, and cyaniding the tailings with 0.07% cyanide solution for 7 hrs. with the addition of 1 lb. of lime per ton of ore. Very little copper is dissolved in this period and the cyanide consumption is only 1½ lb. per ton of ore.

A. R. POWELL.

Rationalisation of the shaft furnace for treatment of lead mineral. N. C. KYRIACOU (*Rev. Mét.*, 1930, 27, 113—124).—The theory of the reactions which take place in the lead blast furnace is outlined and some modern types of such furnaces are described with reference to constructional diagrams. A. R. POWELL.

Sprayed molten metal coating process and results. R. L. BINDER (*J. Franklin Inst.*, 1930, 211, 173—217).—The development of the sprayed metal process is described in which wire is fed uniformly into an acetylene-oxygen flame and the metal blown by means of compressed air on to a receiving surface.

C. A. KING.

Detinning of scrap tinplate. W. W. SCOTT and N. E. DAVIS (*Ind. Eng. Chem.*, 1930, 22, 910—911).—A laboratory trial of the recovery of tin from scrap tinplate by agitation with dilute tartaric acid in contact with air is reported. The scrap tin was placed in a rotating cylinder partly immersed in the acid. The stannous tartrate was precipitated with hydrogen sulphide, and the loss of tartaric acid was 10% by wt. of the tin recovered. Tins require pretreatment to free them from grease and lacquer.

C. IRWIN.

Heat radiation of thermit. W. SHOULEJKIN and X. SOLOVOVA (*Z. physikal. Chem.*, 1930, 149, 434—438).—By means of a solarimeter the heat radiation from burning thermit mixtures has been measured. With ordinary iron-thermit the maximal effect amounts to 38 kw., and with manganese dioxide and potassium permanganate the values are, respectively, 256 and 2080 kw.

E. S. HEDGES.

Actual state of the metallurgy of electrolytic zinc according to recent publications. A. SANSON (*Rev. Mét.*, 1930, 27, 57—63, 125—132).—A review of modern practice in three American electrolytic zinc works, viz., those at Great Falls, Anaconda, and Kellogg (Idaho). In each case the process is briefly described with reference to flow sheets and the electrical conditions in the cells are discussed at some length.

A. R. POWELL.

Determination of cadmium and copper in spelter and zinc ores by rapid internal electrolysis. E. M. COLLIN (*Analyst*, 1930, 55, 495—501).—Zinc and

cadmium may be quantitatively separated by the internal electrolysis method as used for the separation of bismuth and lead (cf. Sand, B., 1930, 773), employing zinc anodes and tinned copper wire with platinum foil soldered to one end for connecting to the cathode. For spelter a sample (10 g.) is attacked with cold 10% sulphuric acid, the residue from which is collected and dissolved in 20% nitric acid, any tin present being filtered off. The filtrate is evaporated to fuming with 20% sulphuric acid, the nitric acid being thus removed and any lead precipitated. The filtrate from the lead is combined with the original solution and 10-vol. hydrogen peroxide and excess of ammonia (≈ 0.880) are added. The iron precipitate, after boiling, may be ignited and weighed as oxide or determined by means of permanganate. The remaining solution is neutralised with sulphuric acid, a slight excess of acid added, and the solution electrolysed for copper at 70°. The deposit is weighed and, after adjusting for acidity, the solution is electrolysed for cadmium. Antimony does not interfere under the conditions of experiment. The treatment of ores and residues differs only in the initial attack.

D. G. HEWER.

Cast-iron enamels. ANDREWS.—See VIII.

See also A., Aug., 1937, **Structure of copper-zinc alloys.** (BRONIEWSKI and STRASBURGER). **Age-hardening lead-calcium alloys** (SCHUMACHER and BOUTON). 988, **System iron-silicon** (STOUGHTON and GREINER). **System iron-vanadium** (ÖYA). **Formation of the violet copper alloy Cu₂Sb** (ARRIVAUT). **Silicon-aluminium alloys** (GUILLET and BALLAY). 999, **Passivity curves of some iron alloys** (BANISTER and EVANS). 1002, **Reduction of metal oxides by hydrogen** (TAYLOR and STARKWEATHER). 1004, **Electro-deposition of beryllium** (BOOTH and TORREY). 1011, **Electrotitrimetric determination of zinc in a mineral** (JOASSART and LECLERC).

PATENTS.

Metallurgical furnaces. W. ALBERTS (B.P. 332,850, 6.11.29. Ger., 4.7.29).—In order to increase the cooling surface of the arch of a furnace, profile bricks are arranged so as to give a wave-like surface in contact with the air, with alternating ridges and depressions.

C. A. KING.

Cupola furnaces. J. E. FLETCHER, and BRIT. CAST IRON RES. ASSOC. (B.P. 333,322, 24.5.29).—In a cupola surrounded by a wind chest from which the main and supplementary tuyères enter the cupola at different heights, a hollow spherical valve can be operated from outside the wind chest to regulate the supply of air through the main tuyère.

C. A. KING.

Crucible furnace. J. W. PENICUD (B.P. 332,701, 17.6.29).—The refractory lining of the furnace is composed of superposed rings having only horizontal joints, and surmounted by a dome-shaped top with plugged holes for admitting fuel. The internal crucible is covered by a truncated cone and the lip may extend through the furnace lining. Suitable means for coke- or oil-heating are provided.

C. A. KING.

Gas-fired metal-heating furnaces. VEREIN. STAHLWERKE A.-G., and E. SCHREIBER (B.P. 318,552,

4.9.29. Ger., 5.9.28).—In annealing furnaces, the heating gas is mixed with the required quantity of air for complete combustion before discharge from the port, and additional gas to produce a reducing atmosphere is admitted around the main heating ports.

C. A. KING.

Protective devices or alarms for metal-melting furnaces. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF J. L. MCFARLAND (B.P. 315,841, 19.7.29. U.S., 19.7.28).—In melting furnaces of the crucible type, the crucible is enclosed in a receiver to hold escaping metal, which makes electrical contact between the ends of two conductors encased in refractory material, thus causing a signal to be given.

C. A. KING.

Reduction of iron ores. A. LANGER (B.P. 309,998, 8.4.29. Austr., 19.4.28).—The charge of ore and reducing agent is fed on to the surface of a flowing bath of molten iron and caused by the action of a series of blast burners to flow in the opposite direction to the stream of iron. Part of the iron issuing from one end of the furnace is returned, with or without the addition of carbon, to the other end to accelerate the flow of metal through the furnace.

A. R. POWELL.

Manufacture of steel. E. C. SMITH, ASSR. TO CENTRAL ALLOY STEEL CORP. (U.S.P. 1,744,418, 21.1.30. Appl., 16.2.25).—The steel is deoxidised with manganese, silicon, and aluminium, and occluded slag is caused to coalesce and rise to the surface by the addition per ton of steel in the ladle of 0.5 lb. of a flux consisting of 8 pts. of litharge and 1 pt. of sodium fluoride.

A. R. POWELL.

Purification of steel. (SIR) G. C. MARKS. FROM LUSIFER PRODUCTS CO. (B.P. 332,890, 21.1.29).—After the completion of the usual finishing reactions and preferably after removal of the greater part of the final slag there is added to the molten steel less than 1% of a finely-divided mixture containing 50–98% Al_2O_3 , 20–1% Fe_2O_3 , 30–1% SiO_2 , and 0–4% TiO_2 , e.g., crude bauxite, alundum, or emery. This treatment serves to remove non-metallic impurities and occluded gases from the steel.

A. R. POWELL.

Production of iron and steel castings. J. E. FLETCHER (B.P. 333,494, 10.1.30).—The mould is pre-heated to such a temperature that in the subsequent heating of the mould by the molten metal the parts of the former in contact with the metal are just heated to the recalescence temperature when the metal in contact with them has itself cooled to the same point. Internal stress during cooling is thus avoided.

A. R. POWELL.

Casting of ingots. (HON. SIR) C. A. PARSONS AND H. M. DUNCAN (B.P. 332,654, 3.5.29. Addn. to B.P. 278,032; B., 1927, 912).—Heat is applied to the peripheral portion of the surface of the ingot to maintain horizontal isothermals and uniform segregation. Such heating may be by means of hot gases through tangential openings, or by electrical means.

C. A. KING.

Production of articles made of steel or cast iron and resistant to attack by corroding agents. P. F. M. AUBERT, and A. J. P. and H. A. M. DUVAL (AUBERT & DUVAL FRÈRES) (B.P. 304,783, 14.2.30).—The articles consist of steel or cast iron containing one or more of the metals aluminium, silicon, manganese,

nickel, chromium, molybdenum, tungsten, vanadium, titanium, zirconium; they are rendered resistant to corrosion by nitrogenisation at 500° in ammonia for 4–8 hrs.

A. R. POWELL.

Rendering austenitic nickel-chromium steels non-corrodible. W. H. HATFIELD and H. GREEN (B.P. 333,237, 5.2.29).—An austenitic steel containing 10–25% Cr and 25–5% Ni, with or without other alloying elements, is tempered at 100–400° after fashioning into suitable shape and polishing. Thus a steel containing 0.14% C, 8% Ni, and 18% Cr becomes resistant to tarnishing by organic acids after tempering at 200° and slowly cooling.

A. R. POWELL.

Manufacture of cast iron and steel containing copper. F. HEUSLER (B.P. 317,734, 20.8.29. Ger., 20.8.28).—The copper is added to the molten metal in the form of a manganese-copper, silicon-copper, or manganese-silicon-copper alloy just prior to casting. Up to 3% Cu may thus be introduced into mild steel without segregation occurring.

A. R. POWELL.

Steel alloy and its treatment. C. P. BEATH and B. J. BABBITT, ASSRS. TO WESTERN ELECTRIC CO., INC. (U.S.P. 1,743,309, 14.1.30. Appl., 19.11.27).—The steel contains 5–50% Co, 1–12% W, 0–7% Mn, 0.3–5% C, 6–18% Ni, and 0–12% Cr, the preferred composition being 20% Co, 9% W, 2.5% Mn, 0.8% C, 7% Ni, and 60.7% Fe. After heating at 820–830° for 2–3 hrs. and quenching in oil or water, the alloy has a high coercive force and magnetic remanence.

A. R. POWELL.

Magnetic [nickel-iron alloy] material. A. F. BANDUR, ASSR. TO WESTERN ELECTRIC CO., INC. (U.S.P. 1,743,089, 14.1.30. Appl., 26.4.28).—An alloy of iron with 30–60% Ni, preferably 35–38%, is rolled into thin strips which are oxidised by heating in oxygen at above 400° and rolled into a ring core, which is heated at 480° for 15 min., whereby an almost constant permeability over a wide range of flux densities is obtained.

A. R. POWELL.

Constructional steel. F. KRUPP A.-G. (B.P. 319,319, 15.8.29. Ger., 20.9.28).—The steel contains up to 0.4% C, up to 0.5% Si, 1.2–2% Mn, and 0.4–0.8% Cu; the preferred composition is 0.2% C, 0.35% Si, 1.6% Mn, and 0.45% Cu.

A. R. POWELL.

Surface plating of metals [e.g., iron or steel] with [chromium] alloys. RESEARCH CORP., ASSEES. OF M. FIEDLER (B.P. 310,812, 30.4.29. U.S., 30.4.28).—The metal is plated with nickel or cobalt, then with chromium, and heated by means of a high-frequency induction coil in such a way that only the plated skin is heated to a sufficiently high temperature to cause the plating metal or metals to alloy with the surface of the ferrous metal. The process may also be used for coating iron with zinc-copper alloys. [Stat. ref.]

A. R. POWELL.

Bright annealing of metals. METALGES. A.-G., H. VON FORSTER, and E. LAY (B.P. 332,656, 3.5.29).—The apparatus comprises a furnace chamber provided with means for passing a non-oxidising gas therethrough and a stuffing box in the lid carrying a vertically acting, movable rod terminating in a bottom plate which carries

the charge, *e.g.*, coils of wire, and closes the discharge outlet. When the annealing operation is finished, a cooling box is wheeled below this outlet, around which it fits tightly, and the charge is lowered into the box by means of the rod so that it can be cooled in a non-oxidising atmosphere.

A. R. POWELL.

Welding of copper. W. ANDREWS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,894, 29.1.29, and 333,466, 2.11.29).—(A) Copper for making welded articles contains 0.03–0.05% P or 0.05–0.1% Si, B, or Cd; it is welded by using a copper rod containing silver and coated with a flux of 45% $\text{NaNH}_4\text{HPO}_4$, 35–45% $\text{Na}_2\text{B}_4\text{O}_7$, up to 10% H_3BO_3 , 6% Mg_2SiO_4 , 3% PbO , and 1% LiF . (B) The welding rod consists of an alloy of copper with 0.5% Ag and 0.06% P, coated with a mixture of 1 pt. of fluorspar and 3 pts. of a deoxidiser alloy containing 15% Mg, 30% Mn, 50% Si, and 5% Fe, C, and other impurities. Alternatively, the silver may be plated on to the phosphor-copper rod and the rod then coated with a mixture of 3 pts. of 80% ferrosilicon, 2 pts. of 15% phosphor-copper, and 1 pt. of a 1:1 mixture of lime and felspar.

A. R. POWELL.

Production of scale-free welded tubing. AIR REDUCTION Co., INC. (B.P. 332,869, 29.11.29. U.S., 10.12.28).—The formed tubing is heated and the seam welded progressively while at the same time reducing gas is passed through the tube.

C. A. KING.

Production of bronze. G. BÄRTGES (B.P. 333,442, 2.10.29).—A bronze resistant to corrosion by sulphuric, phosphoric, and organic acids consists of copper with 7–13% Al and 2–6% Fe. The metals are melted under an alkaline borate flux and a small quantity of sodium, potassium, or magnesium is added as a deoxidiser. The alloy is cast, allowed to cool, remelted under the same slag, deoxidised again with a small quantity of magnesium, and cast at 950–1000° in moulds which admit of rapid cooling.

A. R. POWELL.

[Copper] alloy wire. W. H. BASSETT, Assr. to AMER. BRASS Co. (U.S.P. 1,744,717, 28.1.30. Appl., 18.6.28).—A strong alloy for lead-in wires for telephone installations consists of copper with 0.5–1.5% Cd and 2–7% Zn; its conductivity is 55% of that of copper and in the hard-drawn state it has a tensile strength of 54 tons/in.²

A. R. POWELL.

Bearing alloy [with a lead base]. J. V. O. PALM and E. C. KNUTH, Assrs. to CLEVELAND GRAPHITE BRONZE Co. (U.S.P. 1,745,314, 28.1.30).—The alloy comprises 67% Pb, 15–18% Cd, 5–10% Zn, and 6–15% Sb, preferably 67% Pb, 18% Cd, 5% Zn, and 10% Sb.

A. R. POWELL.

Melting of oxidisable material [brass]. HIRSCH KUPFER- u. MESSINGWERKE A.-G. (B.P. 313,856, 17.6.29. Ger., 16.6.28).—The apparatus comprises a melting crucible surrounded by an induction coil and surmounted by a conical chamber for holding the charge. Above this chamber is a preheating chamber with a bottom perforated plate, closed by a sliding door beneath which is a funnel communicating with the conical chamber. The first charge is melted with the door closed, the door is then opened to allow the vapours from the charge to

preheat a second charge in the top chamber, the molten metal is tapped from a taphole in the bottom of the crucible, and the preheated charge then allowed to fall through the vapours into the crucible, thus avoiding oxidation. [Stat. ref.]

A. R. POWELL.

Light-metal [magnesium] alloy. J. A. GANN, Assr. to DOW CHEM. Co. (U.S.P. 1,743,849, 14.1.30. Appl., 26.7.26).—The alloy consists of more than 80% Mg, 1–10% Al, 1–5% Cu, 0.5–4% Cd, 0.1–1% Zn, and 0.1–1% Mn, the preferred composition being 88% Mg, 8.25% Al, 2% Cu, 1% Cd, 0.5% Zn, and 0.25% Mn.

A. R. POWELL.

Removal of gases from molten light metals such as aluminium and its alloys. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 313,489, 21.5.29. Ger., 12.6.28).—An electrical heating unit is situated above molten metal in a melting chamber to maintain its temperature, and the gases above the metal are circulated in a conduit by mechanical means.

C. A. KING.

Treating aluminium with halogen gases. R. J. ANDERSON, Assr. to FAIRMONT MANUF. Co. (U.S.P. 1,743,515, 14.1.30. Appl., 1.5.28).—Carbides, nitrides, and oxides, as well as occluded gases, may be removed from molten aluminium by treating it with a stream of, *e.g.*, chlorine.

A. R. POWELL.

Flotation process for oxide ores [cassiterite]. J. E. BARNITZKE (B.P. 311,239, 7.5.29. Ger., 7.5.28).—The ore pulp, before or after addition of the flotation oils, is treated with hydrogen, or a gas mixture containing hydrogen, at the ordinary or at an elevated temperature, whereby superficial reduction of the tin oxide particles to metal is stated to take place so that they readily become wetted by the oils.

A. R. POWELL.

[Lead] alloys [for packing joints]. F. W. ANDREWS, Assr. to FRIGIDAIRE CORP. (U.S.P. 1,743,303, 14.1.30. Appl., 28.4.27).—The alloy, which has a coefficient of thermal expansion nearly equal to that of cast iron, comprises 90–96% Pb, 3–1% Sn, and 4–2% Bi.

A. R. POWELL.

Electrolytic deposition of gold and silver. A. MOZER (B.P. 333,462, 30.10.29. Ger., 11.12.28).—The metals are deposited with 0.1–0.3 volt at a current density of 100 amp./m.² from solutions of the iodide in potassium iodide solution, *e.g.*, gold from a bath containing 21 g. of aurous iodide and 140 g. of potassium iodide per litre, and silver from a bath containing 40 g. of silver iodide and 370 g. of potassium iodide per litre.

A. R. POWELL.

Manufacture of magnetic alloys. V. B. BROWNE (Re-issue 17,771, 12.8.30, of U.S.P. 1,570,229, 19.1.26).—See B., 1926, 282.

Magnetic alloy. B. FETKENHEUER and H. NEUMANN, Assrs. to SIEMENS & HALSKE A.-G. (U.S.P. 1,772,771, 12.8.30. Appl., 7.5.28. Ger., 20.5.27).—See B.P. 290,658; B., 1929, 1019.

Pickling of metals. E. CHAPMAN and A. HILL, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,773,024, 12.8.30. Appl., 12.4.29. U.K., 14.4.28).—See B.P. 309,314; B., 1929, 523.

Preparation of homogeneous alloys of lead [with sodium]. G. KRÄNZLEIN, R. K. MÜLLER, F. BRUNNTRÄGER, and H. JANZ, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,774,384, 26.8.30. Appl., 24.10.28. Ger., 5.11.27).—See B.P. 300,129; B., 1929, 1019.

Manufacture of soldering pastes. G. KNOTH (U.S.P. 1,772,952, 12.8.30. Appl., 29.3.29. Ger., 3.4.28).—See B.P. 309,029; B., 1930, 199.

[Electrolytic] production of aluminium and its alloys. T. R. HAGLUND, Assr. to INTERNAT. PATENT CORP. (U.S.P. 1,772,937, 12.8.30. Appl., 19.10.25. Swed., 15.11.24).—See B.P. 242,958; B., 1926, 754.

Electrolytic deposition of gold. W. F. GRUPE (B.P. 333,246, 3.5.29).—See U.S.P. 1,731,415; B., 1930, 20.

Ore carrier for circular Dwight-Lloyd type blast-roasting apparatus. "BERZELIUS" METALLHÜTTEN GES.M.B.H. (B.P. 333,461, 28.10.29. Ger., 27.3.29).

Water-cooled blowing tuyères for blast furnaces. F. D. BROWN, G. B. RICHARDS, and T. J. ROBERTS (B.P. 333,095, 1.10.29).

[Air-vibrator] means for producing an intermittent supply of air to blast, smelting, and like furnaces. E. HICKMAN (Y EMPARAN) (B.P. 311,269, 10.4.29. Spain, 8.5.28).

Sectional electroplating, particularly applicable for plating large objects with chromium. SIEMENS & HALSKE A.-G., Assees. of K. W. SCHWARTZ (B.P. 314,392, 25.6.29. U.S., 26.6.28).

Washing waste blast-furnace gases (B.P. 333,317).—See I.

XL—ELECTROTECHNICS.

Utilisation of waste steam. REUTER.—See I. **Corundum and silicon carbide.** SCHNEIDLER.—See VIII. **Electrolytic zinc.** SANSON. **Electrolysis of spelter and zinc ores.** COLLIN.—See X. **Rubber insulating compounds.** WIEGAND and BOGGS.—See XIV. **Treatment of boiler waters.** BARTOW.—See XXIII.

See also A., Aug., 973, **Rôle of the core metal in oxide-coated filaments** (LOWRY). 994, **Water-gas equilibrium and the electric discharge** (PETERS and KÜSTER). 1003, **Synthesis of ozone** (BREWER and WESTHAVER). 1004, **Electrodeposition of beryllium** (BOOTH and TORREY). 1011, **Electrotitrimetric determination of zinc in a mineral** (JOASSART and LECLERC).

PATENTS.

Electric-discharge devices. A. A. J. CROWNE (B.P. 332,734, 4.7.29).—A hot or cold radioactive cathode is surrounded by or plated with photoelectric material which emits electrons under the incidence of X-, γ -, or α -rays, and is enclosed in a vessel containing, if desired, gases which reduce the impedance of the device. [Stat. ref.] J. S. G. THOMAS.

Cathodes for electric-discharge tubes. M.-O. VALVE Co., LTD., and D. A. RANKIN (B.P. 332,695, 12.6.29).—A tungsten core coated with zinc oxide is

subjected to the action of barium vapour in the tube, whereby a coating of barium oxide is produced.

J. S. G. THOMAS.

Luminous electric-discharge tubes. G. CLAUDE (B.P. 318,198, 27.6.29. Fr., 30.8.28).—The filling for such tubes consists of helium with less than 5% of neon. J. S. G. THOMAS.

Photo-electric cells. G. B. ELLIS. From Soc. DES USINES CHIM. RHÔNE-POULENC (B.P. 332,733, 12.7.29).—The photo-electric current is increased by the emission of secondary electrons from the surface of a third electrode arranged inside the cell and raised to a suitable voltage. J. S. G. THOMAS.

[Positive electrodes for alkaline] electric accumulators. O. Y. INRAY. From I. G. FARBENIND. A.-G. (B.P. 332,884, 13.1.30. Ger., 5.2.29. Addn. to B.P. 325,581; B., 1930, 428).—The active mass, moistened, if desired, with glycerin, and mixed with graphite freed from impurities, more especially iron, is compressed, in order to increase the capacity of the electrodes. J. S. G. THOMAS.

Producing and maintaining constant the necessary supply of phosphoric acid in the electrolyte of lead accumulators. M. KUGEL (B.P. 332,660, 3.5.29. Addn. to B.P. 280,197; B., 1928, 864).—Pieces of glassy metaphosphoric acid of definite weight are added to the electrolyte. Alternatively a mixture of phosphates of metals which do not harm the accumulator may be added. J. S. G. THOMAS.

Electrolytic condenser. E. W. ENGLE and H. L. OLESEN, Assrs. to FANSTEEL PRODUCTS Co., INC. (U.S.P. 1,744,302, 21.1.30. Appl., 14.3.27).—The condenser comprises an acid-proof container filled with a compact roll of tantalum and molybdenum or tungsten sheets separated by thin sheets of asbestos moistened with dilute sulphuric acid. A. R. POWELL.

Moistening of gases prior to purification by electrostatic deposition of dust. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 333,048, 29.7.29. Ger., 1.6.29).—Steam, produced by injecting a liquid, e.g., water, into the products of combustion of a gas burning in a container, is passed into the gas to be treated. J. S. G. THOMAS.

Manufacture of electric insulating materials. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 333,290, 10.5.29).—Rubber, gutta-percha, balata, and similar materials are treated with urea or thiourea or derivatives of these, with the additional presence of proteolytic enzymes such as pepsin or trypsin, if desired. The rubber or other material may be treated as latex or in the ordinary form, using an aqueous solution of the urea compound. The rubber or other material after such treatment and subsequent washing contains a reduced proportion of protein and can be used advantageously for insulating submarine cables, preferably after hardening by admixture with purified gutta-percha or balata or with a bitumen of high m.p. and low content of mineral matter and of free carbon. D. F. TWISS.

Manufacture of electrically insulating material. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM.

ELEKTRICITÄTS-GES. (B.P. 308,315, 21.3.29, and 309,507, 11.4.29. Ger., [A] 21.3.28, [B] 11.4.28).—(A) Powdered metal, *e.g.*, lead or zinc, each grain being coated with a solid coating of material of smaller conductivity, *e.g.*, lacquer or resin, is compressed so that air gaps are removed. (B) In the manufacture of insulating material composed of alternate superposed layers of different electrical conductivities, films of lacquer, baked at 200—250°, are united with a web of fibrous material, *e.g.*, fabric, by a binder, *e.g.*, lacquer.

J. S. G. THOMAS.

Manufacture of insulated cables. J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 332,891, 24.1.29).—Metallic conductors, covered with a non-distillable polymerisation production of butadiene, mixed, if desired, with india-rubber or similar vegetable rubber, together with a filler, *e.g.*, lampblack, and sulphur, and, if desired, a vulcanisation accelerator, and/or an anti-ager, are vulcanised at 100—150°. J. S. G. THOMAS.

Arc-welding electrode. B. TURNER, AssT. to FERRO-ARC WELDING Co., LTD. (U.S.P. 1,773,653, 19.8.30. Appl., 19.6.28. U.K., 26.8.27).—See B.P. 295,514; B., 1928, 760.

[Electrolytic] means for indicating the presence of saline, alkaline, acid, or other impurities in water and other fluids. W. C. CROCKATT (U.S.P. 1,774,319, 26.8.30. Appl., 10.11.26. U.K., 16.11.25).—See B.P. 266,047; B., 1927, 303.

Electric furnaces [with removable heating units]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. L. IPSEN and A. N. OTIS (B.P. 333,457, 21.10.29. U.S., 23.10.28).

Electric heating units [for bakers' ovens etc.]. A.-G. KUMMLER & MATTER (B.P. 313,056, 5.6.29. Switz., 5.6.28).

Heat fuses for use with electric and other furnaces. BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 333,199, 25.4.29).

Regulating the electrodes in tilting furnaces. W. W. TRIGGS. From FLÖDINJERN AKTIEBOLAG (B.P. 333,318, 21.5.29).

Magnetic separators. O. C. JONES (B.P. 333,042, 24.7.29).

Manufacture of electrical resistances. I. SILBERSTEIN (B.P. 333,837, 27.11.29. Ger., 18.12.28).

[Laminated] magnetic cores. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. J. FRANK (B.P. 317,830, 23.8.29. U.S., 23.8.28).

Selenium [light-sensitive] cells. W. PRIOR and C. CRISP (B.P. 333,293, 11.5.29).

Light-sensitive apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of T. A. EDISON (B.P. 316,940, 1.8.29. U.S., 6.8.28).

Light-sensitive devices. [Photo-electric cells.] WESTINGHOUSE ELECTRIC & MANUF. Co., Assees. of V. K. ZWORYKIN (B.P. 333,116, 6.11.29. U.S., 6.11.28).

Photo-electric cells. G. W. WALTON (B.P. 333,548, 14.2.29).

Electron-discharge devices. LOEWE-RADIO GES. M.B.H., and E. RÖMHILD (B.P. 308,978, 2.4.29. Ger., 30.3.28).

Electric glow-discharge tubes. C. R. BURCH, F. E. BANCROFT, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 333,692, 27.6.29).

Devices for irradiation of substances with ultra-violet rays. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 332,977, 13.5.29).

Electric incandescence lamps. GEN. ELECTRIC Co., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 333,858, 20.1.30. Ger., 10.8.29).

[Heater units for] thermionic cathodes of vacuum electric tube devices. A. S. CACHEMAILLE. From WESTINGHOUSE LAMP Co. (B.P. 333,011, 24.6.29).

[Chromium as getter in gas-filled] incandescence electric lamps. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of P. A. CAMPBELL (B.P. 312,613, 28.5.29. U.S., 28.5.28).

Galvanic batteries [with electrolyte reservoir]. H. D. ELKINGTON. From STICKSTOFFWERKE GES. M.B.H. (B.P. 319,785, 27.9.29).

[Filling device for] batteries. S. J. RUBINSTEIN (B.P. 333,291, 10.5.29).

Insulating material (B.P. 330,712).—See II. **Alarms for melting-furnaces** (B.P. 315,841). **Steel alloy** (U.S.P. 1,743,309). **Magnetic alloy** (U.S.P. 1,743,089). **Plating metals with alloys** (B.P. 301,812). **Copper alloy [lead-in] wire** (U.S.P. 1,744,717). **Electrolytic gold and silver** (B.P. 333,462).—See X.

XII.—FATS; OILS; WAXES.

Classification of cacao butter and its substitutes by the f.p. method. A. G. AVENT (Analyst, 1930, 55, 477—487).—The f.p. is determined on 75 g. of the fat or, in a modified test, on 10 g., which are placed in a boiling-tube maintained at not above 65°. When the fat has completely melted, the tube is placed in cold water, stirred until the temperature falls to 40°, wiped, the cork carrying the special thermometer and stirring rod fitted into the tube, and the fat stirred again until the temperature reaches 33°, when the tube is placed in an insulated jar, the neck plugged with cotton wool, stirring stopped at 31°, and the first reading (continued at 1-min. intervals) taken. The fat is stirred for 1 min. after the first visible separation of crystals, and then three times during each half minute, the procedure being continued for 10 min. after the maximum temperature is reached. Values for the maximum and minimum temperatures, the difference between them, and the time taken from 31° to the maximum when plotted give for each kind of fat a typical curve. Cacao butters with high maximum temperatures generally show high acid values. Tables of typical figures are given for cacao butters, veberine, kayao, Pontianak illipé, and various "butters." The more divergent is the cooling curve of a fat from that of a cacao butter, the less suitable was the fat found to be for chocolate making, also the greater its similarity, the more satisfactory was the substitute.

D. G. HEWER.

M.p. range of cacao butter. J. D. VAN ROON (Chem. Weekblad, 1930, 27, 498—502).—The change of volume of the fat on melting is found to follow a characteristic course under standard conditions. A definite volume is introduced into a dilatometer, the instrument kept at 8° for 1 hr., then at 24° for 15 hrs., and finally the temperature is raised at the rate of 4° per hr.; the readings plotted as ordinates against temperatures as abscissæ give a well-defined S-curve, the shape of which is modified by presence of adulterants; 10% of substitute suffices to mask the S-shape entirely. The reproducibility and the variations in the curve arising from variations in the pretreatment are discussed.

S. I. LEVY.

Analytical methods for cottonseed. G. S. JAMIESON and R. S. MCKINNEY (Oil and Fat Ind., 1930, 7, 291—293, 315).—Details are given of provisional standard methods for the sampling of cottonseed, and for the determination of moisture, before and after fuming and grinding, and of free fatty acids.

E. LEWKOWITSCH.

Fatty acids of high mol. wt. [arachidic and erucic acids] in linseed and soya-bean oils. J. GROSSFELD (Z. Unters. Lebensm., 1930, 59, 412—418).—Wallis and Burrows have reported the presence in the fatty acids contained in soya-bean oil of 1% of arachidic acid (cf. B., 1924, 838). Using the method previously developed (cf. Grossfeld and Simmer, B., 1930, 790), pure linseed and soya-bean oils as well as a sample of linseed oil suspected of adulteration with soya-bean oil were tested for fatty acids of high mol. wt. and the values compared with those for stearic acid. The results indicated the following contents of arachidic acid: linseed 0.73%, soya 0.85—0.88%, suspected sample 0.34%. Seven other samples of linseed oil gave values of 0.07—0.47%, calc. as arachidic acid. By fractional precipitation of the lead salt from dilute alcohol it was found that the potassium salt of the supposed arachidic acid formed a jelly in the presence of ether. Erucic acid was thus suspected and its presence was confirmed from the iodine value. The results gave an average value for linseed oil of 1.78% of erucic acid. When the presence of this acid is considered with reference to the total fatty acids in linseed and soya-bean oils it is found that the stearic acid content is extremely low, whilst the mean value for the palmitic acid content is 9%, assuming that 1% of the lead salt remains in solution.

H. J. DOWDEN.

Selective hydrogenation of unsaturated fatty acids and their constitution. I. Linoleic acid. B. SUZUKI and Y. INOUE (Proc. Imp. Acad., Tokyo, 1930, 6, 266—268).—The reduction of methyl linoleate with tetra- or deca-hydronaphthalene and palladised barium sulphate proceeds in two stages; methyl oleate is formed as the intermediate. Oxidation of methyl linoleate with potassium permanganate in acetone containing a small amount of sodium hydrogen carbonate gives hexoic and azelaic acids, showing that linoleic acid is Δ^8 -octadecadienoic acid. Similar oxidation of a half-reduced linoleic acid affords nonoic and azelaic acids, thus confirming the production of oleic acid. Similar hydrogenation of linolenic acid proceeds in three stages.

H. BURTON.

Acetyl value of unsaturated fatty oils. S. UENO and N. KUZEI (J. Soc. Chem. Ind., Japan, 1930, 33, 234—236 B).—In the case of old samples of oils (except beef tallow) the acetyl value by the Lewkowitsch filtration method was greater than that obtained by the distillation process, the latter being nearer the true acetyl value. The difference was greatest (e.g., 33.6 and 12.6 for linseed oil) in the case of the most unsaturated oils; this is attributed to the formation of soluble dibasic acids during acetylation etc. The values for refined oils by both methods were in closer agreement and accorded with the values by André's procedure. The acetyl value of most fresh unsaturated oils was below 10; that of hardened fish oils was lower than that of the original oils.

E. LEWKOWITSCH.

California olive oil. P. F. NICHOLS and B. E. LESLEY (Oil and Fat Ind., 1930, 7, 295—296, 315).—Various tests for rancidity were examined, of which the Kreis test, though not infallible, gave the most reliable results. Californian olive oil has characteristics of colour, viscosity, and free acidity similar to those of imported oils, but the deviations from the average are greater. Lovibond values of 80 yellow, 5 red, 2 blue, relative viscosity (efflux time compared with that of water at 20°) 15—16, and 1% of free fatty acids are recommended as tentative standards for examination, a slight positive Kreis reaction being permissible.

E. LEWKOWITSCH.

Dolphin oil. A. KLEIN and M. STIGOL (Pharm. Zentr., 1930, 71, 497—500).—Dolphin and cod-liver oils may be distinguished by certain colour reactions. E.g., on shaking the oil with (a) an equal volume of nitric acid (d 1.4) and a saturated solution of resorcinol in benzene, or (b) an equal volume of hydrochloric acid (d 1.19) and an ethereal solution of phloroglucinol (1:1000), or (c) light petroleum and nitric acid (d 1.4), red colours are produced with dolphin oil and yellow with cod-liver oil. An oil from the Black Sea dolphin had: d 0.9285, acid value 0.365, saponif. value 209.5, iodine value 129.7, Reichert-Meissl value 32.98, Polenske value 3.29, unsaponifiable matter 0.2%, ether-insoluble acid polybromides 29.04%, and non-volatile fatty acids 87.32% (Hübl iodine value 147.57). The total fatty acids (95.01%) present in the oil had iodine value 130.94, mean mol. wt. 255.3, and contained 15.43% of "solid acids" (Twitchell) (m.p. 48—50°, iodine value 22.26, mean mol. wt. 258.7) and 83.57% of "liquid acids" (iodine value 142.05, mean mol. wt. 275.4). Linoleic and isovaleric acids were identified in the oil, which contained also stearic, palmitic, oleic, and highly unsaturated (clupanodonic) acids and probably one or more solid unsaturated acids.

E. LEWKOWITSCH.

Composition of commercial palm oils. I. Fatty acids and component glycerides of some palm oils of low free acidity. T. P. HILDITCH and (Miss) E. E. JONES (J.S.C.I., 1930, 49, 363—369T).—Four commercial palm oils of low free acidity have been investigated and their characteristics are fully recorded. The fatty acids present had the following setting points and composition: Belgian Congo (neutral), 44.4°, myristic 1.2%, palmitic 43.0%, stearic 4.4%, oleic 40.2%, linoleic acid 11.2%; Malaya (crude), 45.6°.

myristic 2.5%, palmitic 40.8%, stearic 3.6%, oleic 45.2%, linoleic acid 7.9%; Drewin (Gold Coast) (crude), 40.4°, myristic 2.3%, palmitic 34.3%, stearic 5.6%, oleic 49.5%, linoleic acid 8.3%; (neutral), 41.0°, myristic 1.4%, palmitic 32.7%, stearic 7.5%, oleic 51.7%, linoleic acid 6.7%; soft Cameroons (crude), 44.0°, myristic 1.0%, palmitic 38.9%, stearic 5.9%, oleic 43.9%, linoleic acid 10.3%; (neutral), 44.2°, myristic 1.4%, palmitic 40.1%, stearic 5.5%, oleic 42.7%, linoleic acid 10.3%. The glyceride structure of the four oils has been studied by the isolation and examination of the fully-saturated glycerides by means of oxidation with permanganate and acetone solution, and the percentages of fully-saturated glycerides found in the neutralised Belgian Congo, Malay, Drewin, and Cameroons oils were respectively 10, 9, 7, and 8. The fully-saturated glyceride content in palm oils appears to depend on the proportion of unsaturated acids in the total fatty acids of the fat and, correspondingly, the molecular proportion of saturated acids linked with 1 mol. of unsaturated acids in the form of mixed saturated-unsaturated glycerides varies from about 0.8 to 0.6:1. These relationships differentiate palm oil from palm-kernel oil and from seed fats in general, and indicate that the mode of assembly of fatty acids in the glyceride molecules in palm oil is somewhat analogous to that in such animal fats as tallows and butters. The saturated fatty acids both of the original oil and of the fully-saturated glyceride portions contained 85–90% of palmitic acid, and consequently tripalmitin is the main component of the fully-saturated glycerides of palm oil (6–7% of the whole fat). The greater part of palm oil is made up of mixed glycerides in which mono-oleodipalmitins and dioleomonopalmitins must predominate.

Testing of oils and fats for rancidity. J. DAVID-SOHN (Chem.-Ztg., 1930, 54, 606–607).—Cf. B., 1930, 871.

Fatty acids from oxidation of mineral oils. COLLIN.—See III. Pine resin in mixtures. DONATH.—See XIII.

See also A., Aug., 1920, **Determination of degree of unsaturation of oils and fats** (SMIT). 1056, **Composition of fat of silver-black fox** (SCHUETTE and THOMAS). 1057, **Neutral fat of liver and other tissues of the ox** (BLOOR and SNIDER). **Unsaturated acids of oil of *Labeo Rohita*** (CHOWDHURY and SARKAR). 1057, **Determination of neutral fat in animal substances** (ZINZADZE).

PATENTS.

Bleaching fats and oils. H. F. TAYLOR, E. F. SCHERUBEL, and G. W. PHELPS, Assrs. to SWIFT & Co. (U.S.P. 1,744,843, 28.1.30. Appl., 26.4.24).—Bleaching earth (1–10%) and sulphuric acid (e.g., 2–8% of acid of d 1.70) are added simultaneously, or in immediate succession, to the heated oil (55–106°) during vigorous agitation which is continued until the desired bleaching has been obtained (e.g., $\frac{1}{2}$ –1 hr.); the oil is then allowed to settle or is neutralised with, e.g., calcium carbonate, and filtered.

E. LEWKOWITSCH.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of the transparency to light of turbid media. ANON. (Jahresber. VII., Chem.-Tech. Reichsanst., 1928, 222–229; Chem. Zentr., 1930, i, 1976).—Two methods for the determination of the covering power of paints are described.

A. A. ELDRIDGE.

Fastness [of colours] to light. H. J. STERN (J. Oil Col. Chem. Assoc., 1930, 13, 184–192).—A lecture, in which the methods previously proposed for measuring the fastness of colours to light are critically reviewed; a system combining the advantages of “the exposure for a given time” methods with those of the “reference to standard types” methods is recommended. Fading lamps will only grade a colour and are not considered satisfactory as it is impossible to say that results are equivalent to so many hours of sunlight. In some instruments employing artificial light, the internal temperature and humidity are so high as to cause undue fading.

F. C. HARWOOD.

Detection and determination of pine-rosin in various mixtures. E. DONATH (Chem.-Ztg., 1930, 54, 667).—The author's nitric acid test for the detection of rosin in waxes (cf. Dingler's Polytech. J., 1872, July, 2) can be applied quantitatively with the aid of a simple colorimeter. This colour reaction may be used to determine rosin in impregnated papers in the presence of wool fat etc. when the Storch-Morawski reaction is inapplicable.

E. LEWKOWITSCH.

X-Ray examination of lithopones. W. A. WOOD (J.S.C.I., 1930, 49, 300–301 r).—The question as to whether zinc lithopone is a mixture of zinc sulphide and barium sulphate or a compound or a solid solution of one in the other is investigated with the help of X-ray methods. The type of problem in which X-rays might be used with advantage is discussed. Measurements are given showing definitely that the material is not a compound but a mixture. A similar result is found for cadmium lithopone.

Mixer. ZAKARIAS. See I. **Testing of acetylcellulose.** FERMAZIN.—See V.

PATENTS.

Preparation of β -elæostearin and coating compositions containing it. E. I. DU PONT DE NEMOURS & Co. (B.P. 312,052, 15.4.29. U.S., 18.5.28).— β -Elæostearin is “run” at approx. 260°, being thus rendered soluble in high-boiling gasoline or naphtha and suitable when incorporated with resins, driers, pigments, etc. for use as coating compositions.

S. S. WOOLF.

(A, c) **Coating compositions.** (B) **Varnish.** C. I. B. HENNING, C. E. BURKE, and E. E. REID, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,742,507–9, 7.1.30. Appl., [A] 27.7.28, [B] 30.7.28, [C] 5.7.29).—The heavy-metal salts of monoalkyl phthalates are added (A, c) to nitrocellulose lacquers or (B) to oil varnishes. Examples are (A) zinc *n*-butyl phthalate (0.5 pt.) with pyroxylin (1 pt.) and a volatile solvent; (B) cobalt butyl phthalate in a varnish containing tung oil, linseed

oil, rosin, and turpentine; (c) cupric, ferrous, or ferric *n*-butyl phthalate with pyroxylin and a solvent.

C. HOLLINS.

Manufacture of lacquers, priming compositions, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,895, 15.3.29).—The resins made by condensing acylated polyhydric alcohols, in which at least one free hydroxyl group is retained, with polybasic carboxylic acids are incorporated with cellulose ethers in organic solvents. Example: cellulose ethyl ether with the resin from phthalic acid and glycerol mono- or dilinoleate or ricinoleate, or colophony glyceride, or β -hydroxyethyl linoleate; cellulose benzyl ether with the resin from glycerol, colophony, palmitic acid, and maleic acid.

C. HOLLINS.

Preparation of [resinous] condensation products of acetylene and phenols. C. F. SCHRIMPE, Assr. to BAKELITE CORP. (U.S.P. 1,742,519, 7.1.30. Appl. 8.3.27).—4:4'-Dihydroxy- α -diphenylethane (ethylidene-diphenol) is obtained by passing acetylene through a mixture of phenol with a little sulphuric acid and mercuric sulphate in the absence of water or added solvent.

C. HOLLINS.

Manufacture of synthetic resins. I. G. FARBENIND. A.-G. (B.P. 310,816 and Addn. B.P. 332,963, 30.4.29. Ger., [A] 30.4.28).—(A) Crude solvent naphtha in the presence, if desired, of a phenol or a naphthol is treated with a fluoroboro-fatty acid, e.g., fluoroboro-acetic acid, the temperature being kept below 70°, e.g., by addition of an indifferent solvent. The reaction product is treated with an alkali or alkaline-earth oxide and the volatile constituents are removed by known methods from the filtered resin solution. (B) A compound of boron fluoride with an aliphatic hydroxycarboxylic acid, e.g., lactic acid, or with an aliphatic unsaturated acid, e.g., oleic acid, is used in place of the fluoroboro-fatty acid.

S. S. WOOLF.

Manufacture of phenol-formaldehyde condensation products. KUNSTHAARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 312,905, 13.3.29. Austr., 1.6.28).—In the processes described in B.P. 267,901 and 290,963 (B., 1928, 720; 1929, 404) phenols, carbamides, etc. are added to the condensation solution towards or at the end of the condensation process for the purpose of binding free formaldehyde. An initial solution of low viscosity is obtained by working at lower temperatures for shorter times with smaller amounts of catalyst, in order to allow for the increase of viscosity caused by the subsequent addition.

S. S. WOOLF.

Production of shaped articles from urea or thiourea, solid polymerised formaldehyde, and fillers. BAKELITE GES.M.B.H. (B.P. 332,964, 28.1.29. Ger., 3.2.28. Cf. B.P. 305,211; B., 1930, 828).—The dry constituents are mixed in the hot and the reaction mass is shaped by hot-pressing with no intermediate treatment.

S. S. WOOLF.

Floor coverings [e.g., linoleum]. ARMSTRONG CORK Co., Assees. of J. C. MCCARTHY (B.P. 317,756, 20.7.29. U.S., 21.8.28).

Esters of fatty acids (U.S.P. 1,739,315). Driers for paints (U.S.P. 1,742,506).—See III. Azo pigments

(B.P. 331,247).—See IV. Plasticisers (B.P. 312,606). Cellulose derivative compositions (B.P. 308,657).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Solubility of oxygen in rubber and its effect on rate of oxidation. I. WILLIAMS and A. M. NEAL (Ind. Eng. Chem., 1930, 22, 874—878).—The solubility of oxygen in rubber follows Henry's law, the quantity of gas dissolved being proportional to the pressure; it decreases with increase of temperature. The oxidation of raw or vulcanised rubber (the former with the addition of copper stearate) is found to be independent of the oxygen concentration as long as this is above a certain minimum. The process probably involves a series of reactions, the controlling reaction being a slow one in the rubber preceding the oxidation. Rate of chemical absorption increases with temperature, being doubled at lower temperatures for a rise of 7—10°, but the increase appears to be much less at higher temperatures, e.g., at 70°, this reduction arising from the inability of oxygen to penetrate by diffusion as fast as it is being consumed. Near or above 70° the deterioration of rubber seems to be hastened by a second type of oxidation which is influenced by the concentration of oxygen and by high pressures. In artificial ageing tests it is necessary to consider the concentration of the oxygen in the rubber. The concentration of oxygen in rubber suspended in air at 70° will be less than that required for normal oxidation at the maximum rate, and this will limit the value of tests in air to thin slabs of highest grade, the rate of oxidation of which will be small relative to the rate of diffusion. The solubility of oxygen at 70° and atmospheric pressure is slightly greater than that necessary for uniform oxidation, and suffices for the satisfactory accelerated ageing of thin slabs of rubber of good or medium quality. Simultaneous use of high temperatures and high pressures for accelerated ageing should be avoided. D. F. TWISS.

Effect of pigmentation on the work of retraction of rubber compounds. F. S. CONOVER (Ind. Eng. Chem., 1930, 22, 871—874).—Although various reinforcing agents and mineral fillers exert different effects on the work involved in the extension of the compounded rubber, the energy of retraction as indicated by the area between the retraction curves and the elongation axes for mixtures at comparable degree of vulcanisation is substantially independent of the kind or proportion of the compounding ingredient; e.g., vulcanised mixtures containing, respectively, carbon black 5 vols., clay 20 vols., and zinc oxide 30 vols. per 100 vols. of rubber exhibit almost the same work of retraction from equal elongations. Barytes constitutes an exception, possibly because of the presence of vacuoles in the stretched rubber. Work of retraction is influenced, however, by the state of vulcanisation of the mixture.

D. F. TWISS.

Carbon black in rubber insulating compounds. W. B. WIEGAND and C. R. BOGGS (Ind. Eng. Chem., 1930, 22, 822—825).—Carbon black may be incorporated in rubber without impairing its insulating and dielectric properties; fresh, uncompounded carbon black must

be employed. The ability of carbon black to adsorb water and dissolved electrolytes from the rubber causes an increase sometimes exceeding 50% in the dielectric qualities. The useful maximum of carbon black is about 10% on the rubber content.

D. F. TWISS.

Behaviour of various clays with crude and reclaimed rubber. H. A. WINKELMANN and E. G. CROAKMAN (Ind. Eng. Chem., 1930, 22, 865—869).—Tests on a number of commercial clays showed no relationship between the iron oxide content and the rate of accelerated ageing of rubbers containing them, either in the oven test or the bomb test. Chemical analysis generally gives little information concerning the probable behaviour of clays in rubber, but acidity or alkalinity should be determined on account of its influence on rate of vulcanisation. Microscopical examination permits a rapid estimation of the reinforcing value to be made. In general, the effect of clays is the same in reclaimed as in crude rubber.

D. F. TWISS.

Influence of rubber "resins" in zinc oxide accelerator mixings. W. S. DAVEY (J.S.C.I., 1930, 49, 338—343 T).—A comprehensive survey of the activating effect of different amounts of zinc oxide, with or without rubber "resin," on a number of typical accelerators is recorded. The results generally confirm those previously obtained by Martin and Davey (*ibid.*, 1923, 42, 98 T; 1925, 44, 317 T), viz., that fatty acids exert their maximum effect in the presence of 5 pts. of zinc oxide per 100 of rubber. A smaller amount of zinc oxide (say 1 pt.) is insufficient to produce the full activating effect, even in the presence of fatty acids. A larger amount of zinc oxide (say 20 pts. or more) is sufficient to satisfy the requirements of the accelerator and therefore the presence of fatty acids is relatively unimportant. Fatty acids ensure an even distribution of zinc oxide, so that with 5 pts. of zinc oxide there is sufficient zinc oxide surface available for maximum activation in the presence of fatty acids, but not in their absence.

PATENTS.

Treatment of [rubber] latex. NAUGATUCK CHEM. Co., Assees. of E. HAZELL (B.P. 333,391, 7.8.29. U.S., 5.10.28).—Treatment of rubber latex with a substance of the constitution M·O·Hal, where Hal represents a halogen atom and M is an alkali or alkaline-earth metal, an alkyl group, or hydrogen, increases the adhesiveness of the unvulcanised rubber, and also deodorises the latex, increases its mechanical stability, its resistance to putrefaction, and its filterability, and facilitates its creaming. The preferred agent is a hypochlorite, the activity of which can be increased by decreasing the p_H of the mixture, an emulsifying agent, e.g., a complex sulphonic acid, being simultaneously applied, if desired, to assist in stabilising the latex. A thiosulphate may be added subsequently to remove surplus hypochlorite.

D. F. TWISS.

Accelerating the vulcanisation of natural and artificial varieties of rubber. A. CARPMAEL, From I. G. FARBENIND. A.-G. (B.P. 333,174, 2. and 14.5.29).—Vulcanisation is accelerated by a condensation product of ammonia with an unsaturated aldehyde, such as crotonaldehyde, acetaldehyde, or homologues of the latter.

The accelerators, e.g., tricrotonylidenetetramine, may be used singly or jointly.

D. F. TWISS.

Accelerating the vulcanisation of natural or artificial varieties of rubber. I. G. FARBENIND. A.-G. (B.P. 312,069, 11.5.29. Ger., 18.5.28).—Liquid derivatives of guanidine, e.g., pentamethyl- and pentabutylguanidine, have an advantage as accelerators of vulcanisation in that they are easily incorporated in rubber. They possess a dissociation constant exceeding 1×10^{-8} (cf. B.P. 12,661 of 1914; B., 191, 1062).

D. F. TWISS.

Manufacture of rubber or like [seamless, multi-compartment] articles. DUNLOP RUBBER Co., LTD., and E. W. B. OWEN (B.P. 333,331, 31.5.29).

Artificial ebonite (B.P. 330,712).—See II. **Polymerisation of diolefines** (B.P. 331,265).—See III. **Insulating materials** (B.P. 333,290). **Insulated cables** (B.P. 332,891).—See XI. **Leavening rubber** (B.P. 311,389).—See XIX.

XV.—LEATHER; GLUE.

Madagascar barks containing tannins. A. DEFORGE, J. MAHEW, and H. DE BALSAC (Halle aux Cuirs, Tech. Suppl., 1929, 49—57, 84—92, 119—123, 144—149, 175—183; Chem. Zentr., 1930, i, 1573).—Wattle bark contained tans 24.3—27.8, non-tans 5.4—10.4, insoluble matter 51.9—57.2, and water 9.4—12.7%. The tannin is of the catechin type. The bark of *Terminalia Catappa*, L., contains pyrogallol and catechin tannins 11.8, non-tans 4.4, insoluble matter 73.2, and water 10.6%. "Rotra" (*Eugenia*) bark contains tans 12.6, non-tans 12.2, insoluble matter 61.5, and water 13.7%. The tannins are of the pyrogallol and catechin groups.

A. A. ELDRIDGE.

Mechanism of vegetable tanning. I. Acidity characteristics of [tan] liquors and extracts. D. H. CAMERON and G. D. McLAUGHLIN (J. Amer. Leather Chem. Assoc., 1930, 25, 325—358).—Acetic acid was determined in a number of tan liquors by distilling it over in steam. Direct distillation removes some lactic acid. The amount of diffusible material in the tan liquors was determined by diffusion through a "Cellophane" sheet in 24 hrs., and the organic acids were determined by extracting the diffused material with ether. Pyrocatechol, resorcinol, and quinol were also identified in the ethereal extract. Gallic acid was determined in the ethereal extract by diluting, evaporating to dryness, and titrating with 0.1N-sodium hydroxide. A modified fluorescein indicator was evolved for the determination of the "total titratable acidity" of dark coloured tan liquors. Analytical data were thus obtained for samples of actual process tan liquors including those of chestnut, quebracho, hemlock, oak bark, and cutch extracts. It is shown that the acids present in tan liquors, chiefly acetic, lactic, and gallic, are responsible for the characteristic p_H values of the tan liquors, and that whilst the tannins themselves are capable of reacting with alkali, such action is most marked at p_H values above 6.

D. WOODROFFE.

"Red heat" in salted hides (Miss) D. J. LLOYD [with R. H. MARRIOTT and (Miss) M. E. ROBERTSON] (Collegium, 1930, 270—296).—See B., 1930, 251.

Red discoloration of salted hides and salt stains. M. BERGMANN (Collegium, 1930, 255—269).—See B., 1930, 433.

Inclusions in fruits. NIETHAMMER.—See XIX.

See also A., Aug., 1994, **Relation of hydration of gelatin to swelling** (NEVILLE and THEIS).

PATENTS.

Manufacture of tawed leather. J. Y. JOHNSON. From E. STIASNY (B.P. 333,221, 28.1.29).—Skins are treated with a solution of a complex compound of aluminium, in which one radical of carboxylic acid, or an acid synthetic tanning agent, and one sulphurous acid radical are combined with the aluminium. Alternatively, the skins may be treated first with an aqueous solution of a soluble aluminium compound, *e.g.*, alum, sodium chloride, and sodium lactate, and subsequently with an aqueous solution of the ingredients forming the desired complex compound with the aluminium, *e.g.*, alum, lactic acid, sodium sulphite, and a synthetic tannin. [Stat. ref.] D. WOODROFFE.

Manufacture of tanning agents. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,216, 25.3.25).—Resorcinol or pyrogallol (3 mols.) is condensed with benzaldehyde (1—1.5 mols.) in water containing a little acid, reaction being stopped by neutralisation before all the phenol has reacted. Clear brown syrups are obtained which have powerful tanning properties. Substituted benzaldehydes (salicylaldehyde, *o*-chlorobenzaldehyde) may also be used, in which case any crystalline product separating out is removed. C. HOLLINS.

Manufacture of tanning agents [chlorinated products of degraded lignite etc.]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,204 and 332,270, 15.4.29).—The dried chlorinated products obtained according to B.P. 284,670 or 323,781 (B., 1929, 505; 1930, 311) are (A) extracted with an organic solvent, *e.g.*, crude wood spirit, methyl or ethyl alcohol, and the solution obtained is either evaporated, or precipitated by the addition of a salt of a weak acid, a base, or an organic precipitant (*e.g.*, benzene), or are (B) incorporated either with gelatin-precipitating, water-soluble condensation products of hydroxyaryl compounds (*e.g.*, resorcinol) and aldehydes not containing sulphonyl groups, or with condensation products of di- or poly-hydroxy-benzenes and formaldehyde, with or without the addition of an acid, a neutralising agent, and a synthetic or acid vegetable tanning agent. D. WOODROFFE.

Depilating hides and skins. M. BERGMANN (U.S.P. 1,765,199, 17.6.30. Appl., 27.6.25. Ger., 12.2.25).—See B.P. 247,826; B., 1926, 377.

Phenolic tanning agents (B.P. 302,666 and 302,938).—See III.

XVI.—AGRICULTURE.

Microbiological analysis of soils. Influence of continuous unbalanced manuring on the growth and development of *Azotobacter chroococcum*. T. BAUNGARTEL and C. BIHLER (Landw. Jahrb., 1930, 71, 855—876).—Active forms of *Azotobacter chroococcum* were present in all soils irrespective of the fertiliser treatment, but development in enrichment cultures

was in some cases characteristic of the fertilisers used. The use of phosphate fertilisers definitely improved the development of the organism. In cultures from soils receiving nitrogen but no phosphate, development was retarded or weakened. With all fertiliser mixtures which included nitrogen and potash, or nitrogen, potash, and phosphate, growth and development of the organism was similar. A study of the growth of *Azotobacter* gives valuable information as to the physical and chemical properties of soils. A. G. POLLARD.

Methods for determining the nature and abundance of soil organic matter. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1930, 30, 97—116).—Existing methods for the examination of soil humus are investigated and discussed. Total soil organic matter is best determined as $1.72 \times$ total organic carbon content. Determinations of "humus" or "humic acid" in soils by alkaline extraction or by oxidation cannot be used as a basis of calculation of the total organic matter present. From 90 to 95% of the total organic matter may be classified in groups of definite chemical complexes by the following procedure. Fats, waxes, and resins are extracted with ether followed by alcohol or with a 1:1 alcohol-benzene mixture. The extracted residue is decomposed with sulphuric acid and is subsequently extracted with water. In the solution, sugars (as a measure of total carbohydrates) and total and ammoniacal nitrogen may be determined. In the weighed residue the carbon and nitrogen contents are determined and the proportion of lignin is calculated from the C:N ratio. Organic matter in soils consists largely of "soil lignins" and "soil proteins" with small proportions of fats, waxes, resins, and certain carbohydrates including hemicelluloses and cellulose-like substances. A. G. POLLARD.

Exchangeable cations and lime requirement in differently fertilised soils. S. RAVIKOVITCH (Soil Sci., 1930, 30, 79—95).—Methods for the determination of exchangeable bases in soils by neutral salt leaching are examined. Hissink's correction for dissolved calcium carbonate was unsatisfactory in limed soils, but the methods of Gedroiz and of Tyurin gave almost identical results for the value. The exchangeable calcium content of soils was increased in all cases by applications of calcium carbonate, and to a certain extent by superphosphate, but was reduced markedly by ammonium sulphate and to a lesser extent by potassium chloride on both limed and unlimed soils. Farmyard manure increased the total base-exchange capacity of soils and the exchangeable calcium content of limed soils. A. G. POLLARD.

Weathering complex of soil. R. GANSSEN (Mitt. Lab. Preuss. Geol. Land., 1929, No. 8, 1—9; Chem. Zentr., 1930, i, 1851).—The method of van Bemmelen and Hissink is criticised. Determinations of the exchange acidity and of the p_H are nearly parallel with the mol. ratio of adsorbed bases to alumina. A. A. ELDRIDGE.

Composition of natural organic materials and their decomposition in the soil. V. Decomposition of various chemical constituents in plant materials, under anaerobic conditions. F. G. TENNEY and S. A. WAKSMAN (Soil Sci., 1930, 30, 143—160; cf. B., 1929

904).—Plant materials generally decompose much more slowly under anaerobic than under aerobic conditions. Differences in the rate of decomposition of lignins and organic nitrogen complexes as compared with that of cellulose and hemicellulose are particularly marked. The anaerobic decomposition of maize stems, rice straw, oak leaves, and sphagnum is examined and the results are used to explain differences in peat formation. Low-moor peat is formed from plants in which cellulose and hemicelluloses decompose rapidly and lignins and proteins accumulate. In high-moor peats from sphagnum, the polysaccharides are less easily decomposed and cannot serve as energy sources for micro-organisms. The latter are unable to utilise the nitrogen rendered available by the more readily decomposed organic nitrogen complexes.

A. G. POLLARD.

Potash in bog soils. TACKE (Ernähr. Pflanze, 1930, 26, 1–5; Chem. Zentr., 1930, i, 1674).—Bog soils lack potash. Results of potash fertilisation for potatoes and grain are recorded.

A. A. ELDRIDGE.

Presence of *Azotobacter* and absence of *Thiobacillus thio-oxidans* in peat soils. C. E. SKINNER and I. J. NYGARD (Ecology, 1930, 11, 558–561).—*Azotobacter* are active in peat soils only at $p_H > 5.9$. *A. chroococcum* were found in the more basic soils and *A. Beijerinckii* in those approaching the acid limit. *Thiobacillus thio-oxidans* was not found in peat except where sulphur had previously been used as a fertiliser.

A. G. POLLARD.

Availability of manganese and of iron as affected by applications of calcium and magnesium carbonates to the soil. H. B. MANN (Soil Sci., 1930, 30, 117–141).—The acidity of the soils examined was reduced by equivalent amounts by moderate applications of calcium and magnesium carbonates. The effect of the latter is relatively smaller when applied in larger amounts. Liming decreased the solubility of manganese in the soils, smaller applications having a relatively greater effect. The solubility of iron was increased by moderate liming but decreased with larger applications. The increased solubility produced by magnesium carbonate was more persistent than with calcium carbonate. Soya beans grown in soils treated with calcium or magnesium carbonates and fertilisers responded to lime. Heavy dressings of calcium carbonate reduced the crop and large amounts of magnesium carbonate were toxic. Manganese absorption by plants varied with solubility of manganese in the soil, absorption decreasing at the point of maximum yield. Manganese chlorosis was observed in a sandy loam limed to $p_H 8.4$, and was remedied by applications of manganese sulphate to the soil or in very dilute solutions to the leaves. Applications of iron, magnesium, copper, or barium salts to soil or foliage were without effect on the chlorosis. Manganese chlorosis is not a systemic deficiency, since localised applications of manganese cause a complete recovery of the part treated. Excessive liming of light soils produces a chlorosis specifically due to manganese deficiency and not associated with a deficiency of iron.

A. G. POLLARD.

Effect of fertilisation on the permeability of soil. B. GANOSSIS (Praktika, 1928, 3, 696–698; Chem. Zentr., 1930, i, 1852).—Continuous fertilisation with

stable manure, but not that with artificial manures, considerably diminishes the permeability of the soil to water.

A. A. ELDRIDGE.

Nature of the yield curve [of plants] with increasing applications of potash. H. WIESSMANN, and K. NAUMANN (Landw. Jahrb., 1930, 72, 105–123).—The yield curve of oats and rye grass with increasing supplies of potash approximated to the logarithmic type of Mitscherlich. Later sowing of the seed caused slight increases in the “effect factor” of potash. The mean values of the “effect factor” of potash with rye grass and with oats were similar, but with the former crop there was a slightly decreased value with the higher applications of potash. In general, values of the “effect factors” for potash were slightly smaller than those of Mitscherlich.

A. G. POLLARD.

Fertilisation of acid soil with potassium salt. H. KAPPEN (Ernähr. Pflanze, 1930, 26, 6–10; Chem. Zentr., 1930, i, 1674).—Yields of rye, oats, and potatoes were not increased, but addition of Thomas meal or chalk gave high yields. The results support Ehrenberg's lime-potash law.

A. A. ELDRIDGE.

Action of ammonium sulphate and of saltpetre on development of sugar beet and maize in relation to composition of the nutrient solution. I. G. DIKUSSAR (Landw. Jahrb., 1930, 72, 79–104).—Sand-culture experiments are described in which a continuous flow of nutrient of controlled p_H value was arranged. The growth of sugar beet in the absence of peat was normal. The effect of sulphate and nitrate of ammonium and nitrate and nitrite of sodium as sources of nitrogen for maize and sugar beet depended on the p_H of the nutrient. At $p_H 5.5$ the efficiency of these substances was in the order $\text{NaNO}_3 > \text{NH}_4\text{NO}_3 = \text{NaNO}_2 > (\text{NH}_4)_2\text{SO}_4$. At $p_H 7.0$ ammonium sulphate was superior to all other forms of nitrogen. An increase in the concentration of the calcium, magnesium, and potassium salts in the nutrient improved the efficiency of ammonium sulphate, but did not affect that of sodium nitrate. On the other hand, at $p_H 7.0$ a decreased concentration of these salts weakened the effect of ammonium sulphate and increased that of sodium nitrate. The relative efficiencies of ammonium sulphate and sodium nitrate are not definite, but can be controlled by the nature and proportions of the other constituents of the nutrient solution. Ammonium salts produced a smaller proportion of ash contents in the plants than did sodium nitrate. This is traced to the inhibitory effect of the ammonium ion on the permeability of the roots to calcium and magnesium ions.

A. G. POLLARD.

Behaviour of tricalcium phosphate in cultivated soils. H. DANNEEL and K. W. FRÖHLICH (Landw. Jahrb., 1930, 72, 51–58).—Tricalcium phosphate does not dissolve in water in the molecular form. The ratio $\text{CaO}:\text{P}_2\text{O}_5$ in solution varied from 1:1.1 at dilutions of 1 in 200 to 1:2.6 at dilutions of 1 in 50,000. The bearing of this on the composition and solubility of naturally occurring phosphates is discussed.

A. G. POLLARD.

Physical and chemical properties of commercial arsenical insecticides. I. Manganese arsenate.

F. E. DEARBORN (J. Econ. Entom., 1930, 23, 630—635).—Commercial manganese arsenate is a mixture of manganese and calcium arsenates, oxides of manganese, and small proportions of iron, alumina, and silica. No common standard of composition exists among commercial samples. A. G. POLLARD.

"Penetrol" [insecticide] as an activator for nicotine. J. L. HOERNER (J. Econ. Entom., 1930, 23, 174—177).—"Penetrol" (a sulphonated oxidation product of petroleum) was more effective than potassium fish-oil soap as an activator for nicotine used against several species of aphids. A. G. POLLARD.

Reducing the unit charge of nicotine [in plant sprays]. R. S. FILMER (J. Econ. Entom., 1930, 23, 165—169).—Laboratory and orchard tests show that the toxic concentration of nicotine to aphids is reduced to 1:5000 when 0.5% of sodium oleate is included in the spray. Neither sodium oleate nor potassium oleate injured orchard tree foliage, even when the leaves were coated with lead acetate or sulphur from previous sprays. A. G. POLLARD.

Preliminary report on *p*-dichlorobenzene solutions for the control of the lesser peach borer (*Algeria pictipes*, G. and R.). O. I. SNAPP (J. Econ. Entom., 1930, 23, 636—638).—Favourable results are recorded. The highest percentage kill was obtained by painting infected portions of trees with a solution containing 1 lb. of *p*-dichlorobenzene in 2 quarts of crude cottonseed oil. A. G. POLLARD.

Naphthalene fumigation at controlled concentrations. A. HARTZELL and F. WILCOXON (J. Econ. Entom., 1930, 23, 608—618).—Red spider mites, cyclamen mites, and thrips were controlled by a concentration of 0.008 lb. of naphthalene per 1000 cub. ft. of air at 30.5° (average) and at R.H. 60%. Plant injury under these conditions was restricted to a very few species. A. G. POLLARD.

Carbon dioxide as an aid in the fumigation of certain highly adsorptive commodities. R. T. CORTON (J. Econ. Entom., 1930, 23, 231—233).—Admixture of carbon dioxide with fumigant vapours (e.g., chloropicrin and ethylene oxide) considerably facilitated the destruction of the flour beetle (*Tribolium confusum*) in nut-meats. A. G. POLLARD.

Comparative insecticidal value of different species of *Derris*. E. R. DE ONG (J. Econ. Entom., 1930, 23, 619—624).—Five species of *Derris* all proved of insecticidal value. The toxic material was present in all parts of the plants. *D. elliptica* is probably slightly superior to the other species. A. G. POLLARD.

Relation of evaporation to killing efficiency of soap solutions on the Harlequin Bug and other insects. B. B. FULTON (J. Econ. Entom., 1930, 23, 625—630).—Several minutes' contact is necessary for the penetration of a toxic dose of soap owing to the insects' power of closing the spiracles. The efficiency of a soap solution is inversely proportional to the rate of its evaporation. The addition of hygroscopic substances did not appreciably increase the efficiency. A. G. POLLARD.

Sodium in crops. V. VINCENT and J. HERVIAUX (Ann. Sci. agron. Franç., 1929, 46, 444—460; Chem.

Zentr., 1930, i, 1809).—Sodium is most abundant in those plant organs which display greatest physiological activity. Soil acidity appears to favour the accumulation of sodium. Sodium alone is not a fertiliser, but sylvinit, rich in sodium chloride, affords better yields of beet than does potassium chloride.

A. A. ELDRIDGE.

Economic principles of stall manure management. M. RAUTERBERG (Landw. Jahrb., 1930, 71, 921—1010).—A comprehensive survey of the uses and making of stall manure under varying natural and farm conditions. A. G. POLLARD.

Yields of crude and assimilable nutrient by double and treble mowing of meadows after various fertiliser treatments. H. JANTZON and W. KIRSCH (Wiss. Arch. Landw., 1929, B, 1, 166—178; Chem. Zentr., 1930, i, 1850).

Peat-growing plants. WAKSMAN.—See II. Fluosilicates and fluoaluminates. CARTER.—See VII. Elements in cereal grains. SCHMORL. Finland cows' milk. VIRTANEN. Fodder and quality of milk. HERRMANN. Coagulability of milk. WURSTER. F.p. of milk from diseased cows. VAN RAALTE.—See XIX.

PATENTS.

Preparation of organic mercuric compounds suitable for use in seed disinfectants. (SIR) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 331,494, 21.1.29).—A mercuric salt is caused to react with an alkyl, aryl, or aralkyl derivative of lead or tin, or with a mixture of sodium-lead alloy and an alkyl salt; a solvent is preferably present. Mercuric chloride and lead tetraethyl in alcohol give ethyl mercurichloride, m.p. 193°. The phenyl mercurichloride, m.p. 252°, cyclohexyl mercuriacetate, and benzyl mercuriacetate are also described. The best yields are obtained with mercuric acetate in acetic acid. C. HOLLINS.

Fertilisers (B.P. 331,546).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Effect of temperature on the sulphur dioxide content of corn syrup in mixtures of sugar and corn syrup. R. H. MORGAN (Analyst, 1930, 55, 488—495).—Since corn syrup and sugar may contain, respectively, 450 and 70 p.p.m. of sulphur dioxide, and since the longer is the time of boiling, the more sulphur dioxide is expelled, it has been possible to use certain bleaching agents without detection. However, the addition of such agents may be ascertained if a curve be plotted of residual sulphur dioxide against temperatures of boiling. The proportions of sugar and corn syrup are determined by analysis and the temperature to which the mixture has been boiled is indicated from the dry solids content. Assuming the maximum permissible amount of sulphur dioxide, a corresponding curve may be drawn giving the maximum amounts of sulphur dioxide in p.p.m. to be expected at the various temperatures, for it is shown that residual sulphur dioxide diminishes with rise in temperature to which the mixture is heated, and further that the amount of sulphur dioxide evolved increases with this

temperature, and that a determination of dry solids is a measure of the boiling temperature.

D. G. HEWER.

PATENTS.

Manufacture of starch products. INTERNAT. PATENTS DEVELOPMENT CO. (B.P. 332,680, 27.5.29. U.S., 26.12.28).—Starch products of colloidal character, when mixed with cold water, are produced by gelatinising by heat and pressure the raw material, which consists of a starch and water mixture containing non-starch substances and taken from the wet-milling process of making starch from maize or similar grain. The gelatinising apparatus consists of a pair of hollow, heated rolls, a hopper to contain the moist starch, arranged above the rolls, and a mechanism for spreading the starch from the hopper into a thin layer and feeding it between the rolls.

C. RANKEN.

Production of starch capable of swelling in cold water. O. MEYER (U.S.P. 1,773,056, 12.8.30. Appl., 8.5.26. Ger., 14.12.25).—See B.P. 286,377; B., 1928, 345.

XVIII.—FERMENTATION INDUSTRIES.

Calculation of the original gravity of beer. R. SAAR (Chem.-Ztg., 1930, 54, 639).—The original gravity of beer was formerly calculated from $S = 100 \times (E + 2.0665A)/(100 + 1.0665A)$, where S is the gravity, E the extract, and A the alcohol, all the values being expressed in wt.-%. A first approximation to the value of the original gravity is obtained from the expression $E + 2A$, and if this is designated by s , the new formula becomes $S = s - 0.0107A(s - 6.24)$, where 6.24 is the value of S at which it is exactly equal to $E + 2A$.

C. RANKEN.

Composition of light lager beers. E. REMY (Z. Unters. Lebensm., 1930, 59, 402–406).—Examination of eight varieties of lager beer has shown that the physical constants and chemical constitution are generally similar. The content of alcohol and extractable material may be readily calculated from the surface tension by means of formulæ (i) $E = 3.3 \times [s_1 + (s_1 - s_2)]$, (ii) $A = 2.7s_2$, where E and A are the extractable material and alcohol content, respectively, and s_1 and s_2 the relative surface tensions of the beer and the alcoholic distillate, respectively. The calculated values agree to within $\pm 0.04\%$ with those obtained by direct determination. The average values were alcohol 3.19%, extractable material 4.88%, mineral matter 0.227%, total nitrogen 0.069%, refraction of beer and of distillate at 17.5° 39.6 and 19.4, respectively. Tryptophan and tyrosine could not be detected, but positive evidence of the presence of vitamins-B and -C was obtained. For the measurement of p_H bromocresol-green was found very satisfactory when the beer was diluted 1:9.

H. J. DOWDEN.

Wine distillates and wine brandies. H. ZELLNER (Z. Unters. Lebensm., 1930, 59, 431–433).—Mainly polemical against Büttner and Miermeister (cf. B., 1930, 527).

H. J. DOWDEN.

[Wine distillates and wine brandies.] BÜTTNER and MIERMEISTER (Z. Unters. Lebensm., 1930, 59, 433–434).—A reply to Zellner's criticism (cf. preceding abstract).

H. J. DOWDEN.

Standardisation and deterioration of rennin. L. D. HAVENHILL (J. Amer. Pharm. Assoc., 1930, 19, 720–724).—Examination of 10 specimens of rennin showed that deterioration had occurred to the extent of 60–99% during 17 years. Various recommendations regarding standardisation and storage of rennin are made.

H. BURTON.

See also A., Aug., 1009, **Rapid p_H determination [in beer and wort]** (FLETCHER and WESTWOOD).

PATENTS.

Nutritive media for cultivating pathogenic bacteria and preparation of vaccines therefrom. A. BECKER (B.P. 313,514, 17.1.29. Ger., 13.6.28).—The culture medium is prepared by adding a vegetable thickening agent such as agar to the undiluted vegetable juice obtained by expressing a vegetable substance rich in sugar and containing lævulose. For the manufacture of the vaccines, bacteria are grown on the medium at 37–40° for from 2 to 20 hrs., according to the type of bacteria. The bacteria are washed off with sodium chloride solution with 0.5% of added phenol, killed by heating to 54–60°, and diluted as necessary.

C. RANKEN.

Manufacture of spirit and yeast by working-up molasses. F. SIMMER, ASSR. to A./S. DANSK GAERINGS INDUSTRI (U.S.P. 1,774,406, 26.8.30. Appl., 13.1.28. Austr., 21.1.27).—See B.P. 283,969; B., 1929, 262.

Dehydration of impure ethyl alcohol. E. RICARD, ASSR. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,763,722, 17.6.30. Appl., 20.11.25. Belg., 20.11.24).—See F.P. 614,913; B., 1927, 857.

XIX.—FOODS.

Elements in cereal grains. K. SCHMORL (Z. ges. Mühlenwesen, 1930, 6, 152–153, 167–169; Chem. Zentr., 1930, i, 1809).—The metal and non-metal content of the ash of rye shows greater differences than that of wheat. With increasing moistness during growth the content of phosphate, calcium, and potassium increases, whilst that of nitrogen decreases. In chalky soils the calcium content is increased, and the potassium content diminished. Clay soils increase the potassium and sulphate and diminish the calcium content. Sandy soils give the highest nitrogen content. No direct relation exists between the mineral content and the fertilisation. In comparison with the straw the grain contains more phosphorus and less silicon.

A. A. ELDRIDGE.

Detection of the age of flours. W. HARTMANN (Z. Unters. Lebensm., 1930, 59, 364–379).—An investigation has been made of the influence of storage on rye and wheat flours and grain. By the action of atmospheric nitrogen, moisture, and enzymes changes are produced in the fat extracted by light petroleum or chloroform resulting in a lowering of the refraction (at 40°) and an increase in the acidity as measured by titration with 0.5N- or N-caustic soda against phenolphthalein. During the first 7–8 weeks of storage a decrease in the lecithin-phosphoric acid content of the extracts was also detected, which thus accounts for the rapid fall in the refraction. The acidity of freshly-milled 60% rye flour was 54–76

for commercial samples and 44 for an experimentally milled specimen, whilst for 60% wheat flour the corresponding values were 46–64 and 39, respectively. After storage for 6 months the values for 60% rye flour and 60% wheat flour were 120–150 and 90–100, respectively. It is considered that flours having acidities higher than the upper limits should be regarded as unserviceable.

H. J. DOWDEN.

Composition of cows' milk in Finland. A. I. VIRTANEN (Milchwirt. Forsch., 1930, 9, 355–364; Chem. Zentr., 1930, i, 1869).—The low albumin (3.12%) and high lactose (4.94%) contents are characteristic. The albumin content is lowest in May and highest in September and October. With increase of albumin and fat the lactose decreases, and conversely. The calcium and phosphorus contents are practically constant. The variations are attributed to long-continued stall feeding and lack of exercise.

A. A. ELDRIDGE.

Relation between the calcium content of Allgäu soils to the calcium content of milk and its coagulability by rennin. K. WURSTER (Milchwirt. Forsch., 1930, 9, 258–285; Chem. Zentr., 1930, i, 1869–1870).—The coagulability by rennin varied little; the feed and lactation conditions had no significant effect. Considerable variations in the calcium content are attributed chiefly to the conditions of lactation. The manner of combination, rather than the amount, of milk-calcium determines the behaviour towards rennin.

A. A. ELDRIDGE.

Fodder and quality of milk in meadow fertilisation with calcium cyanamide. L. HERRMANN (Milchwirt. Forsch., 1929, 9, 142–160; Chem. Zentr., 1930, i, 1713).—Changes in the acidity, fat content, and density of the milk were observed.

A. A. ELDRIDGE.

Effect of increasing doses of coconut and palm-kernel cake on the fat content of milk. F. HONCAMP, W. HELMS, G. KÖDDER, and A. PETERMANN (Milchwirt. Forsch., 1929, 9, 161–178; Chem. Zentr., 1930, i, 1713).—The favourable effect, although proportionality does not exist, on the fat content and quantity of milk was confirmed.

A. A. ELDRIDGE.

Fat in the fodder of milch cows. A. BUSCHMANN (Tierernähr., 1930, 1, 129–178; Chem. Zentr., 1930, i, 1962).—Up to 17.8% of the fat digested passes without marked change in composition into the milk. Moderate administration of fat usually has a favourable effect on the milk fat, but for each fat there is an upper limit of dose.

A. A. ELDRIDGE.

Iodine [feeding] question. J. HANSEN (Tierernähr., 1929, 1, 119–124; Chem. Zentr., 1930, i, 1962).—Administration of "ancora" iodised salt to cows or sows was not followed by an increased milk yield or live weight.

A. A. ELDRIDGE.

F.p. of milk from cows having foot-and-mouth disease. A. VAN RAALTE (Chem. Weekblad, 1930, 27, 480).—An analysis is given of the f.p. of 78 samples, which varied from -0.532° to -0.566° , the values being somewhat lower than that for normal milk.

S. I. LEVY.

Composition of "calciamolke" (whey). A. WASITZKY (Z. Unters. Lebensm., 1930, 59, 394–398).—

Whey prepared by the precipitation of casein with calcium lactate (4–5 g. per litre of milk) has been compared with wheys obtained by the treatment of milk with acetic acid and rennet. The results obtained with three samples of whole milk are tabulated, giving η_{sp} , density, total solids, fat, albumin, lactose, and ash, together with the percentage composition of the ash. The differences were very slight except in the case of the fat content, which was practically zero (0.0–0.02%) for the "calciamolke," but appreciable though small (0.02–0.27%) for the other two wheys.

H. J. DOWDEN.

Rapid determination of the bacterial content of meat products and sausages. F. W. BICKERT (Z. Unters. Lebensm., 1930, 59, 345–364).—The most suitable apparatus for disintegrating the product prior to its examination was found to be a ball-mill, using fused quartz sand as the abrasive and physiological saline as the medium. The best results were obtained when the proportions were 20 g. of sand, 80 c.c. of saline, and 20 g. of meat, the mixture being ground for 1 hr. at 120 r.p.m. at 0° . Various objections are cited against the plating-out methods of determining the bacterial content. A more rapid and accurate method is described in which the standard of comparison is a suspension of saprophytic spores which had been blackened by immersion in silver nitrate solution followed by pyrogallol. The suspension is mixed with the liquid under test in the proportion 1:9, the bacteria being then stained with carbol-fuchsin and a count made on a thin film of the mixture.

H. J. DOWDEN.

Preservation of raspberry juice with hydrofluoric acid. R. COHN (Z. Unters. Lebensm., 1930, 59, 422–428).—A criticism of Hanak's statements (cf. B., 1930, 482).

H. J. DOWDEN.

[Preservation of raspberry juice with hydrofluoric acid.] A. HANAK (Z. Unters. Lebensm., 1930, 59, 429–430).—A reply to Cohn's criticism (cf. preceding abstract).

H. J. DOWDEN.

Toxicological aspect of the preservation of raspberry juice with hydrofluoric acid. E. DEUSSEN (Z. Unters. Lebensm., 1930, 59, 421–422).—A recapitulation of a publication on the toxicology of fluorine compounds (cf. Z. ges. richt. Med., 1923, 2, 141) with reference to Hanak's statement that small daily doses of hydrofluoric acid (0.006–0.009 g.) are cumulative and dangerous (cf. B., 1930, 482).

H. J. DOWDEN.

Formaldehyde titration of lemon juices. A. NIETHAMMER (Z. Unters. Lebensm., 1930, 59, 420).—When examined by the method of Tillmans and Kiesgen (cf. B., 1927, 456) natural and commercially prepared lemon juices were readily distinguishable.

H. J. DOWDEN.

Microchemical detection of [glucosidic] inclusions in certain fruits. A. NIETHAMMER (Z. Unters. Lebensm., 1930, 59, 418–419).—The inclusions in the flesh of *Mespilus germanica*, *Ceratonia siliqua*, and *Phoenix dactylifera* have been isolated by sublimation under reduced pressure at 350° . The crystalline forms are characteristic of glucosidal tannins and the colour reactions with potash and with hydrochloric acid

and vanillin gave positive results. Acetaldehyde and tannins were detected in the fruits. H. J. DOWDEN.

Ethylene oxide as a fumigant for food and other commodities. E. A. BACK, R. T. COTTON, and G. W. ELLINGTON (J. Econ. Entom., 1930, 23, 226—233).—Stored foodstuffs may be fumigated successfully with ethylene oxide alone or in conjunction with carbon dioxide. The mixture is non-inflammable and non-explosive. Doses necessary for various materials are recorded and vary from 2—3 lb. of ethylene oxide per 1000 cub. ft. for 1—3 hrs., or, with 14 lb. of carbon dioxide, either the dose or the period of treatment may be reduced. (Cf. Cotton and Roark, B., 1928, 732.)

A. G. POLLARD.

Stalk treatment of fruit and vegetables for prevention of stalk rot, with special reference to bananas. R. G. TOMKINS and R. M. WOODMAN (J.S.C.I., 1930, 49, 285—287 T).—As a result of a number of trials of various substances for stem end treatment for the prevention of stalk rot, the use of paraffin wax, vaseline, and, possibly, boric acid crystals is recommended.

Mixer. ZAKARIAS.—See I. Cacao butter and its substitutes. AVENT. M.p. range of cacao butter. VAN ROON.—See XII. Carbon dioxide fumigation [against the flour beetle]. COTTON.—See XVI.

See also A., Aug., 1070, Beef extract as source of vitamin- B_2 (HOAGLAND and SNIDER).

PATENTS.

Butter manufacture. E. G. N. and (Mrs.) E. S. SALENUS (B.P. 333,233, 7.5.29).—Cream or whole milk is boiled at low temperature *in vacuo*, the agitation thus produced forming butter. Suitable apparatus is described.

E. B. HUGHES.

Leavening of foodstuffs, india-rubber, and the like. E. O. WING (B.P. 311,389, 10.5.29. U.S., 10.5.28).—The mass to be leavened contains a ketonecarboxylic acid capable of decomposition by heat, with the aid of additional substances such as alkaline material if desired. For foodstuffs, acetonedicarboxylic acid is preferred because it is physiologically harmless and also gives rise only to carbon dioxide and acetone, the latter contributing to the leavening effect. This acid, if carefully prepared, can be kept for months with little decomposition. For use as a baking powder it is conveniently mixed with three times its weight of a starch product, e.g., flour or starch of rice or maize, the moisture content of which has been reduced below 10%. Such ketonecarboxylic acids can also be used for producing a cellular structure in other materials, e.g., in rubber.

D. F. TWISS.

Pasteurisation or sterilisation processes and plants. A. GRAHAM-ENOCK, and GRAHAM-ENOCK MANUF. CO., LTD. (B.P. 332,949 and 332,950, 26.3.29).

Manufacture of biscuits. T. & T. VICARS, LTD., and E. M. CROSLAND (B.P. 333,158, 2.4.29).

Treatment of edible nuts [in the shell with dyes]. BENNETT DAY IMPORTING CO., INC., Assees. of H. C. GOOD (B.P. 315,398, 14.6.29. U.S., 13.7.28).

Food preservatives (B.P. 331,274).—See XXIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Reactions of sulphonal and trional. L. EKKERT (Pharm. Zentr., 1930, 71, 550).—Sulphonal may be distinguished from trional by the colour reactions with resorcinol and sulphuric acid, followed by dilution and addition of ammonia, the former giving a rose colour and the latter a brownish-yellow or yellow-brown.

S. I. LEVY.

Pharmacology of ergot: biological assay and standardisation. IX. **Summary with conclusions and recommendations.** M. R. THOMPSON (J. Amer. Pharm. Assoc., 1930, 19, 705—720; cf. B., 1930, 881).—When fluid extract of ergot (U.S.P. X) is freed from alcohol by evaporation in a vacuum, and the residue made up to the original volume by addition of saturated sodium hydrogen carbonate solution, the alkaloids are precipitated, whilst the non-specific amine fraction (mainly histamine) remains dissolved (cf. B., 1930, 639). The alkaloids are redissolved in dilute alcoholic hydrochloric acid. The various methods for the assay of ergot extracts are reviewed and the "isolated rabbit uterus method" is recommended as the official method. Ergotamine tartrate is suggested for use as a standard. Recommendations regarding storage of ergot and extracts are made.

H. BURTON.

Infusum radices ipecacuanhæ. K. H. BAUER and K. HEBER (Pharm. Zentr., 1930, 71, 513—518).—Infusions of concentrations below 1:400, prepared by the method of D.A.B. VI, contain the bulk of the alkaloids present as determined in the original drug by the D.A.B. VI method (ammoniacal ether extraction). More concentrated infusions contain less of the original alkaloids; thus a 1:20 infusion contains only about one half. Concentration *in vacuo* of dilute infusions results in considerable decrease of alkaloid content. Decoctions prepared by the above method contain the same amounts of alkaloid as infusions of equal concentration, and prolonged treatment does not raise the yield in either case. When the residue from a concentrated infusion is again extracted, a further quantity of alkaloid is removed, but the total yield remains deficient. Infusion of such a residue with dilute hydrochloric acid brings the total yield almost to that indicated by the D.A.B. VI determination. Infusion of the original drug with dilute hydrochloric acid (2 g. with 40 c.c. of water and 10 c.c. of 0.5N-hydrochloric acid, followed by a second infusion with 50 c.c. of water) gives, however, higher values for the alkaloid content than the German official method, the figures from the latter representing only about 87% of the alkaloid present. The original drug, therefore, contains alkaloid in a form (possible glucosidic) that is removed neither by infusion nor by ammoniacal ether extraction.

R. CHILD.

Decomposition of quinine on keeping or irradiating its aqueous solution. R. DIETZEL and K. SÖLLNER (Pharm. Ztg., 1930, 75, 955—957; cf. B., 1929, 147, 869; A., 1930, 795).—A 1% solution of quinine hydrochloride undergoes no change on keeping in diffused daylight for 1—2 days; on keeping for 60 days or after more than 5 hrs.' exposure to the radiation from a mercury-vapour lamp, the solution shows, besides

the development of colour, a decrease in its power of absorbing ultra-violet light, although no new bands appear in the absorption spectrum. At the same time the optical rotation decreases in value and the b.p. falls slightly (*e.g.*, after 6 hrs.' irradiation the b.p. of a solution had fallen 0.09° and the optical rotation decreased from -177.4° to -168.9°). These changes, which are uninfluenced by passing oxygen through the solution during irradiation, are ascribed to polymerisation of the cinnamic-truxillic acid type involving the unsaturated linking in the vinyl group of the quinine molecule. Direct confirmation of this by a chemical method has not, however, yet been obtained.

R. CHILD.

Reactions of morphine and other opium alkaloids. L. EKKERT (Pharm. Zentr., 1930, 71, 550—551).—The colour reactions with *p*-dimethylaminobenzaldehyde and sulphuric acid are described. S. I. LEVY.

Analyses of chewing-tobacco. W. KOENIG (Z. Unters. Lebensm., 1930, 59, 407—411).—Determinations of moisture, ash, foreign inclusions, water-soluble extract, and nicotine have been made on 26 samples from 10 factories. Nicotine was determined by (a) a method using kieselguhr and tungstic acid, (b) Pfyl and Schmitt's method (*cf.* B., 1927, 955) and (c) a modification of the author's original method (*cf.* A., 1911, ii, 472). In (c), the sample (20 g.) is treated with 60 c.c. of 15% caustic soda and the mixture then extracted with 100 c.c. of xylene. After shaking and keeping for 24 hrs., the xylene solution is poured off, dried with anhydrous sodium sulphate, and filtered. A portion of the filtrate (75 c.c.) is shaken with 15 c.c. of 1*N*-sulphuric acid and, after separating and filtering the nicotine sulphate solution, 10 c.c. of it are shaken with 5 c.c. of 50% caustic soda and 25 c.c. of xylene. The xylene extract is separated, dried, and polarised. A table of factors is given for conversion of rotation into the percentage of nicotine. The tobaccos were also examined for artificial sweetening and preservative. The results showed wide variations. The averages of the three nicotine determinations, which agreed well, indicated nicotine contents of 1.9—4.8%. Adulteration with artificial sweetening and foreign vegetable materials was not detected, but traces of zinc and arsenic as well as boric and benzoic acids were noted in several samples. The considerable loss on drying (max. 22%) and the high mineral content (max. 3%) gave rise to serious criticism.

H. J. DOWDEN.

Microchemistry in the domain of essential oils and perfumery material. L. ROSENTHALER (Perf. and Essent. Oil Rec., 1930, 21, 277—286).—A review. Details are given, with descriptions of apparatus, for the microchemical examination of such raw materials as benzoin, peru and tolu balsams, storax, camphor, valerian root, nutmeg and mace, cloves, tonquin beans, cinnamon, and aniseed; for the identification of pure compounds, including benzaldehyde, bourbonal, menthol, piperonal, and thymol; for the examination of essential oils, and for the detection of such adulterants as nitrobenzene in benzaldehyde, and coumarin in vanillin. The microdetermination of hydrogen cyanide is also described.

R. CHILD.

Bacteriological chemistry of heavy metals.

E. A. COOPER and S. D. NICHOLAS (J.S.C.I., 1930, 49, 386—388 *r*).—Cadmium compounds are actively germicidal, being more potent than lead salts, but less so than mercury salts. The soluble complex pyrophosphates of sodium with cadmium and bivalent mercury are strongly inhibitory to bacteria. The mercurous compound is active in concentrations of 1 in 1,000,000, and the cadmium derivative in 1 in 10,000. Inhibitory power has been measured by studying the effect of the germicides on (a) rate of bacterial growth and (b) rate of dissolved oxygen absorption. In certain cases metallic compounds have been found to exert a stimulating effect on bacteria. Methods are also described for the preparation of colloidal solutions of cadmium compounds. Cadmium carbonate produces a very stable sol, but the phosphate and molybdate are unstable. In the case of lead, however, the sols of the phosphate and molybdate are extremely stable.

Mixer. ZAKARIAS.—See I. Nitrous oxide for anaesthesia. HENDERSON and LUCAS.—See VII. Diffusion of kaolin and insoluble carbonates. RAE.—See VIII.

See also A., Aug., 1031, Phenylurethane anaesthetics (RIDER). 1046, Preparation of amino-alcohols, derivatives of atophan, carbostyryl, and of quinoline, and arsenicals (FOURNEAU and others). 1047, Synthesis of isomerides of proflavine and neutral acriflavine (BOGERT and others). Dialkyl-barbituric acids (SHONLE and others). 1050, Alkaloids of ergot (SMITH and TIMMIS). 1053, Reactions of adrenaline and novocaine (EKKERT). Specific colour reactions of adrenaline and tyrosine (KISCH).

PATENTS.

Methylenedisalicylic acid derivatives. S. L. SUMMERS (U.S.P. 1,743,630—6, 14.1.30. Appl., [A—F] 12.10.28, [G] 8.12.28).—Methylenedisalicylic acid is converted by way of an ester into the amide. (A) The amide is etherified with a nitrobenzyl chloride, the nitro-groups are reduced, and the product, after being heated with pyruvic acid in alcohol at 130° to introduce two pyruvyl groups into the two amide groups, is diethylated to give the compound $\text{CH}_2[\text{C}_6\text{H}_3(\text{O} \cdot \text{CH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{NHet}) \cdot \text{CO} \cdot \text{NH} \cdot \text{CO} \cdot \text{COMe}]_2$. (B) The amide is converted into *O*-acyl derivatives, *e.g.*, by heating with pyruvic or acetic acid and sulphuric acid. (C) The amide is etherified with a nitrobenzyl chloride, the nitro-groups are reduced, and the product is tetrapyruvylated or (D) tetra-acetylated. (E) The amide is converted into its *O*-diacetate, which is then diiodinated. (F) The amide is converted into its *NN'*-dibenzylidene derivative, which is diiodinated, or is dipyruvylated and diiodinated. (G) The amide is diiodinated. All the products have antirheumatic and antiseptic properties.

C. HOLLINS.

Manufacture of guanidine derivatives. Soc. CHEM. IND. IN BASLE (B.P. 316,195, 24.7.29. Switz., 24.7.28).—Compounds containing a heterocyclic NH group react with cyanamide to give guanyl derivatives. Examples are guanyl derivatives of α -pipecoline (hydrochloride, m.p. 190—192°), ethyl *p*-nipecotylamino-benzoate [hydrochloride, m.p. 116° (decomp.); picrate, m.p. 226°], tetrahydroquinaldine (hydrochloride, m.p.

184—185°; nitrate, m.p. 179°, *d*-coniine (nitrate, m.p. 154—155°), piperidine (hydrochloride, m.p. 184°), stilbazoline, 2:4:6-trimethylpiperidine (nitrate, m.p. 160—162°), ethyl 2:4:6-trimethylpiperidine-3:5-dicarboxylate (nitrate, m.p. 162°), nipecotyl-diethylamine, nipecotylethyl-*n*-butylamide, nipecotyl- β -phenylethylamine, nipecotyl-4-*o*-anisoxylaniline, and decahydroquinaldine (hydrochloride, m.p. 242°). C. HOLLINS.

Organic arsenic compounds. [Str.] G. C. MARKS. From PARKE, DAVIS, & Co. (B.P. 331,195, 22.12.28).—Arylarsinic acids are warmed in water with salts of carboxylic acids containing a thiol group. The products, Ar·As(S·R·CO₂Na)₄, or Ar·AsO(S·R·CO₂Na)₂, or Ar·AsO(ONa)(S·R·CO₂Na), are valuable against spirochaetes and trypanosomes. Sodium thioglycollate is condensed with 3-amino-4-hydroxybenzenearsinic acid, with phenylglycineamide-4-arsinic acid, and 3-nitro-4-hydroxybenzenearsinic acid; cysteine with arsanilic acid, and phenylglycinamide-4-arsinic acid.

C. HOLLINS.

Manufacture of salts of antimonie acid and of organo-stibinic acids. I. G. FARBENIND. A.-G. (B.P. 309,184, 5.4.29. Ger., 7.4.28. Addn. to B.P. 311,448; B., 1929, 577).—The salts of *p*-aminobenzenestibinic acid with primary or tertiary amines, diamines, etc. are therapeutically valuable, as also are amine salts generally of antimonie acid. Diethylamine antimonate, diethylamine 3-chloro-4-acetamidobenzenestibinate, ethylenediamine *p*-aminobenzenestibinate, and quinine *p*-acetamidobenzenestibinate are described. C. HOLLINS.

Isolation of certain physiologically active substances. C. FUNK (U.S.P. 1,745,788, 4.2.30. Appl., 4.1.27. Ger., 15.1.26).—Picric, picrolonic, and flavianic acids, and other analogous nitrophenols, are used as precipitants, *e.g.*, in the purification of insulin, and isolation of the hormone of the posterior lobe of the pituitary gland, vitamins-B, -C, and -D, etc.

C. HOLLINS.

Separation of α -terpineol from pine oil. L. T. SMITH, Assr. to HERCULES POWDER Co. (U.S.P. 1,743,403, 14.1.30. Appl., 11.8.27).—Pine oil is fractionated, preferably under reduced pressure, and the fraction, b.p. 214—220°, is cooled to -15° and seeded. C. HOLLINS.

Manufacture of derivatives of quinoline [atophan]. R. WOLFFENSTEIN (U.S.P. 1,773,837, 26.8.30. Appl., 10.1.29. Ger., 23.1.28).—See B.P. 304,655; B., 1929, 835.

Drying of tobacco. "UNIVERSELLE" CIGARETTEN-MASCHINEN-FABR. J. C. MÜLLER & Co., and C. W. MÜLLER (B.P. 333,416, 4.9.29).

Magnesium hydroxide tablets (B.P. 310,919).—See VII. **Dental cement** (B.P. 333,325).—See IX. **Vaccines** (B.P. 313,514).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See also A., Aug., 1005, Colour-selectivity of latent photographic images (WEIGERT and SHIDEI).

PATENTS.

Production of diazo-types. KALLE & Co. A.-G. (B.P. 331,459, 19.10.29. Ger., 19.10.28).—Hydroxyarylguanidines or -diguandines having a free position

o- or *p*- to the hydroxyl group are used as coupling components in diazo-type processes. Examples are: 7-hydroxy-1-naphthylidiguandine with diazotised *p*-aminodimethylaniline (blue) or tetrazotised tetramethoxybenzidine (blue); 7-hydroxy-2-naphthylidiguandine with diazotised 4-amino-4'-methoxydiphenylamine (reddish-brown).

C. HOLLINS.

Packing for photographic plates. I. G. FARBENIND. A.-G. (B.P. 333,864, 14.3.30. Appl., 16.3.29).

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Methods of influencing the combustion of ignition compositions, explosives, etc. A. J. STEPHENS. From ASOCIACION DE PRODUCTORES DE YODO DE CHILE (B.P. 333,364, 11.7.29).—The addition of iodine or oxygen-free compounds of iodine, such as cuprous iodide, to match-head compositions, explosives, and flash-light powders, and to rubbing surfaces for matches, promotes uniform combustion without explosion. The use of 5% of cuprous iodide in rubbing surfaces permits a reduction in the proportion of red phosphorus from 50 to 10%. W. J. WRIGHT.

Denitrating "mixed" acid (B.P. 333,264).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Hygienic evaluation of surface water. O. ACKLIN (Gas- u. Wasserfach, 1930, 73, 409—416).—From numerous examples it is shown that a satisfactory hygienic evaluation of a water may be obtained by determining the ratios non-volatile residue/total nitrogen as ammonia and total oxygen/oxygen consumption after keeping for 5 days, both ratios being measured immediately after filtration of the water through a sand filter. Simultaneous high values for both ratios indicate a thoroughly satisfactory water for domestic consumption.

A. R. POWELL.

Determination of ammonia in water. M. W. E. EVERS (Chem. Weekblad, 1930, 27, 475—480).—The limit of HCO₃' concentration below which it is necessary to add alkali in order to ensure the removal of all ammonia by distillation is experimentally determined as 46 mg. per litre; when more than this is present, all the ammonia is driven off, the ratio of calcium to magnesium having no influence. Calcium carbonate is the most suitable material for the addition, but objections urged against the use of magnesia and sodium carbonate are shown to be unfounded. S. I. LEVY.

Testing of river water by determination of the permanganate demand and the chlorine number. A. BEHRE, H. CHRISTLIEB, and M. KONGEHL (Z. Unters. Lebensm., 1930, 59, 398—402).—In an attempt to determine the cause of the unpleasant taste of the Hamburg water supply during the winter months, samples were taken at three stations over a period of 9 months and subjected to the permanganate test and Froboese's chlorine test (cf. Arb. Reichsges.-Amt., 1920, 52, 211). During the summer months the chlorine value was 33—50% lower than the permanganate demand, but during December—March the former often exceeded the latter, thus indicating the presence of protein matter. The figures support the view that

pollution is being caused by sewage, especially as the beet-sugar factories are not in operation during the period when the water is most contaminated. The cold chlorination to which the water supplies are subjected does not affect the values given by the tests; bacteria are destroyed, but organic matter is not attacked (cf. Keiser, B., 1926, 222).

H. J. DOWDEN.

Purification of drinking water by slow sand filtration. H. DORNEDDEN (Gas- u. Wasserfach, 1930, 73, 289—294, 319—325, 340—343).—Laboratory and large-scale tests on the comparative filtering properties of quartz sand and graded basaltic lava are described in detail. Although the lava filters were somewhat more retentive of bacterial slime, they did not behave so satisfactorily as the sand filters under conditions which were continually varying. Again, the lava grains had a decided tendency to break up during mechanical purification, so that re-grading was necessary before the lava could be used again; this more than compensated for the longer life of the lava filter beds. Examples are given of the composition of the filtrate and of the deposited slime after filtration of various waters through sand and lava filters, and the effects of pore size, rate of filtration, temperature, and pressure on the filtration processes are discussed.

A. R. POWELL.

Electro-osmose treatment of boiler waters. E. BARTOW (J. Amer. Water Works' Assoc., 1930, 22, 1115—1121).—The electro-osmose process (cf. B., 1927, 958) gives a water which is equivalent to distilled water for steam-raising purposes. Tap water and zeolite-softened water are equally suitable for use as the raw water; practically all dissolved solids are removed except a trace of organic matter and a little silica which is thought to be present in the molecular rather than in the ionic form and to be possibly of colloidal size. A partly-purified water containing a total residue of approx. 65 mg./litre can be obtained at an operating cost which compares favourably with a barium carbonate-calcium hydroxide treatment operating at the same rate. The cathode wash-water, which contains hydroxide, may be used industrially in any process where lime water is used. The anode water can be used as a bleaching or disinfecting wash-water. The removal of the first 50% of the dissolved solids can be accomplished at one third the cost of complete removal.

C. JEPSON.

See also A., Aug., 1020, **Preparation and bactericidal properties of penta-, hepta-, and nona-decoic acids** (GREER and ADAMS).

PATENTS.

Manufacture of materials resistant to or active against micro-organisms [food preservatives and disinfectants]. A. CARPMAEL. From T. SABALITSCHKA (B.P. 331,274, 28.3.29).—The *m*- or *p*-hydroxybenzoic esters of alcohols having 2 or more carbon atoms (ethyl, propyl, benzyl) are added to fruit juice, gelatin, or other material liable to bacterial decomposition. Mixtures of the esters are especially effective; e.g., a mixture of 0.045% of ethyl ester and 0.035% of *n*-propyl ester added to fruit pulp permanently prevents attack by mould or yeast.

C. HOLLINS.

Disinfectant, antiseptic, and germicidal products.

J. M. SCHAFER (U.S.P. 1,745,507—8, 4.2.30. Appl., [A] 11.10.28, [B] 20.10.28).—(A) The use of *C*-acylated resorcinols, e.g., acetyl, propionyl, butyryl, valeryl, hexoyl, heptyl, and octoyl derivatives, and (B) *C*-amyl- and/or *C*-hexyl-phenols, is claimed.

C. HOLLINS.

Insecticides. (SIR) G. C. MARKS. From ROHM & HAAS Co. (B.P. 330,911, 13.12.28).—Chloroacetic acid derivatives having a vapour pressure lower than that of the butyl ester are non-lachrymatory and at considerable dilution kill flies, aphids, Japanese beetle, cockroaches, etc. Glycerol tri(chloroacetate), cyclohexyl chloroacetate, glycol di(chloroacetate), sodium chloroacetate, 2:3-dichlorophenacyl chloride, chloroacetamide, dichloroisopropyl chloroacetate (b.p. 133—135°), and nitrophenyl chloromethyl ketone are specially mentioned.

C. HOLLINS.

Preventing the formation of scums and films due to growth of organisms on surfaces in contact with water. J. C. BAKER, ASST. to WALLACE & TIERNAN PRODUCTS, INC. (U.S.P. 1,745,141, 28.1.30. Appl., 21.7.27. Renewed 14.11.29).—The formation of such slimes, in condensers and similar surfaces in contact with running water, may be prevented by the intermittent addition to the water of chlorine in sufficient quantity to leave a small residuum.

C. JEPSON.

Chlorinating apparatus [for water]. W. C. BLACKMOND (U.S.P. 1,752,723, 1.4.30. Appl., 29.3.29).—A closing apparatus for chlorine is described in which a definite volume of gas is released periodically by means of a pulsating device. The period required to complete a cycle of operations is claimed to be under easy control and the whole apparatus simple in operation and unlikely to develop leaks.

C. JEPSON.

Water softener. W. N. McCONKEY (U.S.P. 1,753,661, 8.4.30. Appl., 24.9.27).—A mixture consisting of a colloidal suspension of aluminium hydroxide (5—10%), caustic soda (20—30%), trisodium phosphate (5—10%), soda alum (1—2%), and glycerin or castor oil (1—2%) is claimed as an efficient agent for purifying and softening water for many industrial and domestic uses.

C. JEPSON.

Deodorising, disinfecting, or preserving organic or offensive matter and production of fertilisers therefrom. FIDASCO, LTD., and W. H. HASLAM (B.P. 331,546, 2.4.29).—Dried pulverised common seaweed is claimed to be a suitable medium for packing and preserving fresh fish, fruit, etc., for use as an absorbent or partial drying agent, and for the production of a stable and inoffensive fertiliser when mixed with fish offal and subsequently dried and powdered.

C. JEPSON.

Production of antiseptic agents. H. WIELAND, ASST. to C. H. BOEHRINGER SOHN CHEM. FABR. (U.S.P. 1,772,975, 12.8.30. Appl., 14.6.26. Ger., 20.6.25).—See B.P. 253,918; B., 1927, 894.

Filtration material for gas masks [from fibrous material and activated charcoal]. V. HORÁK (B.P. 309,914, 15.4.29. Austr., 17.4.28).

Filter insertions for respirators [from activated carbonaceous felt]. V. HORÁK (B.P. 307,835, 12.3.29. Czech., 14.3.28).

Filtering material (U.S.P. 1,734,197).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCT. 24 and 31, 1930.*

L—GENERAL; PLANT; MACHINERY.

Grinding capacity of flint-ball mills. A. II. M. ANDREASEN and J. J. V. LUNDBERG (Trans. Ceram. Soc., 1930, 29, 239—250).—Calcined Danish ball-flint was wet-ground in a flint-ball mill, and during a period of grinding the variation of fineness was determined by the pipette method. During the test nine samples were taken and data showing hours of grinding, current used, revolutions per minute, total number of revolutions, grain sizes, and increase of surface are tabulated. Rittinger's law, that the increase in surface is directly proportional to the energy consumed, does not apply for the system under test. R. J. CARTLIDGE.

Cascading v. cataracting in tube mill. H. A. WHITE (J. Chem. Met. Soc. S. Africa, 1930, 31, 1—18).—The theory of the use of balls for grinding in tube mills is discussed and formulæ for the optimum speed of rotation and power required are given. Experiments bearing on the theory have been carried out and the effect of moisture was investigated. D. K. MOORE.

Fluids handling. I. W. L. BADGER and E. M. BAKER (Chem. Met. Eng., 1930, 37, 370—373). II, III. W. L. BADGER and W. L. McCABE (*Ibid.*, 430—434, 494—499).—A description is given of the various methods of jointing iron and steel pipes and of types of valves. Air lifts, ejectors, and reciprocating pumps are dealt with, followed by varieties of rotary and centrifugal pumps, compressors, and blowers, all being treated from the engineering point of view.

C. IRWIN.

Determination of macro-pore volume of porous substances. STADNIKOV. Absorption pipette for gas analysis. OTT.—See II.

See also A., Sept., 1102, **Intensive drying of liquids** (SMITS). 1115. **Measurement of particle size in ultra-violet light** (HASLAM and HALL).

PATENTS.

Open-hearth furnace. W. GAST (U.S.P. 1,756,484, 29.4.30. Appl., 23.6.21).—The air ports of the furnace are situated immediately over the regenerators and each is tapered, forming a throat which is more or less obstructed by a sliding gas nozzle. Preferably the adjustment of the gas nozzle simultaneously adjusts the gas supply. B. M. VENABLES.

Heat-interchanging apparatus. SERCK RADIA-TORS, LTD., and C. O. WAGNER (B.P. 333,764, 5.9.29).—An exchanger suitable for heating air, or cooling liquids by means of air, comprises a number of gilled tubes between inclined plates; the upward ends of the latter open into a common uptake or chimney producing a natural draught. B. M. VENABLES.

Heating in a gaseous atmosphere, particularly in a reducing or oxidising atmosphere. H. SCHAEFER (B.P. 333,192, 1.2.29).—The articles are heated by the sensible heat only of a gas, before combustion, which has been highly heated in a reversible regenerator. After passing through the goods the gas is mixed with air and burned in the other side of the regenerator. For an oxidising atmosphere the air would be heated.

B. M. VENABLES.

Cooling or heating apparatus particularly applicable to viscous fluids. R. SELIGMAN (B.P. 333,157, 28.3.29).—The apparatus comprises an inclined rotating cylinder cooled by exterior water sprays. Scrapers or "doctors" are provided on the interior to control the thickness of the layer of material, but these scrapers extend through only part of the length, the remaining part at the outlet end being occupied by shelves or scoops to discharge the material.

B. M. VENABLES.

Apparatus for drying, heating, and evaporating. W. HOBSON (B.P. 332,951, 27.2.29).—Material such as clay is run or pressed into tubular containers while in a fluid state. The tubes are impervious or pervious according to whether steam or a drying gas is used for heating. Apparatus of both kinds may be combined with a heat exchanger, so that live or exhaust steam may be used in one and the vapour produced may give up its heat to gases in the exchanger and the gases dry another batch of clay contained in pervious tubes.

B. M. VENABLES.

Drying drums. A. CARPMAEL. From BÜTTNER-WERKE A.-G. (B.P. 333,517, 8.5.29).—A drying drum of large size is sectionalised so that the parts will come within the limits of railway loading gauge.

B. M. VENABLES.

Separating arrangement for pneumatic centrifugal dryers. E. BARTHELMESS (B.P. 333,859, 22.1.30. Ger., 7.2.29).—After leaving the dryer, the air-borne material is caused to pass downwards and then upwards through a conduit with a return bend of small radius, at the bottom of which are a port and sack or other container to collect heavy or large particles.

B. M. VENABLES.

Grinding mills. H. DRYSDALE (B.P. 333,404, 21.2.29).—A mill of the type described, having a convex surface rotating about a (usually) vertical axis and engaging with a smaller concave surface rotatable about an inclined axis, is provided with positive drive to both elements, the relative speed being variable; the pressure between the elements can be varied, one element can be swung aside, and temperature control (water-cooling) can be applied through the hollow axis of one or both elements. B. M. VENABLES.

* The remainder of this set of Abstracts will appear in next week's issue.

Grinding mills. E. F. STIMSON (B.P. 333,635, 28.2.30).—In a mill of the type described in B.P. 333,404 (preceding abstract) both elements are positively driven; of the upper member only the rim is pressed by springs towards the lower member, and a definite minimum clearance is maintained. Liquid seals are provided to prevent lubricant from the upper member reaching the grinding surfaces and to prevent dirt reaching the driving mechanism of the lower member.

B. M. VENABLES.

Grinding mills. MACAO-WALZENMÜHLEN-GES.M.B.H. (B.P. 313,611, 14.6.29. Ger., 15.6.28. Addn. to B.P. 291,803; B., 1928, 878).—A mill acting on the principle described in the prior patent comprises the following parts in order downwards, the rotating parts being driven by a vertical shaft:—(1) agitator-feeder; (2) single-faced annular disc, non-rotating but adjustable axially; (3) set of double-faced planetary discs, the gear wheels being between the faces, and the sun pinion on the shaft serving also as a collar so that the planets may be adjusted axially by raising the shaft; (4) double-faced annular disc, non-rotating (or oppositely rotating) but having a limited axial freedom; (5) set of planets as in (3); (6) double-faced annular disc, stationary; (7) rotating annular disc having limited axial freedom on the shaft; (8) footstep for shaft adjustable in height. The construction is such that all wearing parts may be made of porcelain and the annular discs are preferably grooved.

B. M. VENABLES.

Grinding mills. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 334,058, 22.8.29).—The mill is in the form of a vertical, double-thrust ball-bearing of which the middle race is driven by power; the feed is to the outside of the upper ring of balls, and the ground material is borne away by upward internal air currents and passes through classifiers which return coarse material to the grinding zones.

B. M. VENABLES.

Grinding and subdividing apparatus. J. E. GERNELLE-DANLOY (B.P. 311,329, 3.5.29. Fr., 9.5.28).—A number of superposed discs are rotated by a vertical shaft, and grinding is effected by horizontal rollers on their upper faces. The material flows over the edge and through a central aperture of alternate discs which are coned and the rollers provided with spiral grooves to assist the flow.

B. M. VENABLES.

Method and mechanism for grinding and separating materials. W. J. CLEMENT, ASSR. to BOSSERT CORP. (U.S.P. 1,755,573, 22.4.30. Appl., 7.4.28).—A disintegrator of the hammer type is provided with an outward bulge on the circumference of the casing. Partly filling the bulge is a hollow element of which the concave face forms a continuation of the normal curve of the casing, but the outer convex face is formed of screening (or even left entirely open) so that fine material can be withdrawn through it by means of an air current.

B. M. VENABLES.

[Disc] mills for grinding paints, enamels, inks, and other viscous substances. DISC BAR MILLS, LTD., and E. A. WHITE (B.P. 333,436, 30.9.29).—The fixed element comprises a hollow member of wavy, cruciform, or star shape of which the leading grinding

edges are smooth and the trailing edges are grooved across their thickness. The rotating disc is horizontal.

B. M. VENABLES.

Device for reducing [the size of] materials. H. G. LYKKEN (U.S.P. 1,756,253, 29.4.30. Appl., 9.12.25).—The apparatus comprises rotors on a vertical shaft within a vertical casing, the material being admitted at a point above the bottom and air in controllable quantity through the bottom. The lower rotors produce an eddying motion which effects the reduction, passage of the air-borne material straight upwards is prevented by baffle plates, there is a comparatively quiet zone above the eddying rotors, and the material is drawn out through the top by a fan on the same shaft. The wall of the casing may be provided with deep grooves to retain a substantial layer of solid material, and with a side pocket to catch uncrushable material.

B. M. VENABLES.

Machines for crushing coal, stone, and similar materials. H. MARTINEZ and R. H. KIRK (B.P. 333,642, 21.5. and 20.11.29).—A crusher of the swing-jaw or other type is operated through resilient members so that the crushing member can yield to uncrushable pieces at both the inlet and outlet ends, preferably in such a way that the crushing motion changes to sliding or rubbing.

B. M. VENABLES.

Pulverising mills. INTERNAT. COMBUSTION ENG. CORP., ASSEES. OF H. KREISINGER (B.P. 317,081, 1.8.29. U.S., 11.8.28).—In a grinding system for coal etc., in which a substantial part of the air travels in a closed circuit, heated air or gas for drying purposes is admitted to the grinding mill in such a way that it will not come in contact with stationary accumulations of coal which inevitably collect about the lower part of the grinder; to this end the heated gas is admitted as an upper stratum to the main stream of returning air immediately before it enters the mill.

B. M. VENABLES.

Mixing, dissolving, or similar apparatus. H. S. BEERS, ASSR. to TURBO-MIXER CORP. (U.S.P. 1,756,236, 29.4.30. Appl., 29.5.29).—A tank is provided with an impellor near its suitably curved bottom and with a set of parallel spaced plates having teeth formed on them by which fibrous or viscous material is broken down.

B. M. VENABLES.

Troughs and the like for kneading, mixing, etc. BAKER PERKINS, LTD., and R. H. WEBB (B.P. 333,993, 18.6.29).—The inner lining of a jacketed vessel is formed without rivet holes or joints, recesses are formed on the back of the lining, and pads forming distance pieces for the jacket space are ball-paned into the recesses. A somewhat similar method is used for attaching the stuffing boxes for the stirrer shaft. The apparatus is particularly applicable for mixing cellulose acetate.

B. M. VENABLES.

Homogenising mill. W. EPPENBACH, ASSR. to UNITED STATES COLLOID MILL CORP. (U.S.P. 1,755,576, 22.4.30. Appl., 23.7.27).—A form of mill having flat annular working surfaces and bottom discharge is described.

B. M. VENABLES.

Heating of decomposable liquids. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 333,241, 8.2.29).—In an apparatus for heating liquids that deposit carbon

or other scale, of the type in which the heating means is kept in motion, such means are shaped so as to produce as little stirring action as possible, *e.g.*, they may be in the form of strips, electrically or otherwise heated, with their length parallel to the axis of rotation and with a cross-section in streamline form with the axis of symmetry tangential to the circle of rotation.

B. M. VENABLES.

Line filter. C. G. HAWLEY, ASSR. to CENTRIFIX CORP. (U.S.P. 1,755,780, 22.4.30. Appl., 8.3.26).—A stationary device, suitable for insertion in a pipe-line, which embodies no true filtering or straining action, but depends on inertia and gravity, is described. The liquid to be purified (*e.g.*, water) is impelled towards three substantially motionless bodies of the fluid in succession, the purified liquid being withdrawn sideways from the stream. The first action is in a straight line downwards to separate the larger heavy impurities, the second is in a spiral path downwards to separate the remaining heavy particles, and the third straight upwards to separate light particles, gases, etc.

B. M. VENABLES.

Apparatus for straining liquids. F. BAILEY and F. H. JACKSON (B.P. 333,850, 11.12.29. Holl., 5.1.29).—A strainer for large quantities of liquid, *e.g.*, circulating water, has a fixed vertical screen divided into sectors which is cleaned sector by sector by means of a back flush of strained water applied through a rotating flush box on one side and exhausted through a corresponding waste box on the other side. Automatic means are provided so that the flush water is only applied for short intervals when the flush and waste boxes coincide exactly with a sector of the screen. The screen may be on the suction side of the circulating pump and the flush water taken from the delivery of the same pump.

B. M. VENABLES.

Straining or filtering apparatus. AUTO-KLEAN STRAINERS, LTD., and W. R. BELDAM (B.P. 334,029, 27.7.29).—A form of construction of an edge strainer formed of thin discs spaced apart by distance pieces is described. The discs are annular and are threaded on and rotated by a cage of rods passing through internal lugs; fixed scrapers also of thin sheet extend into each filtering space.

B. M. VENABLES.

Distillation of liquids [especially crude glycerin]. W. E. SANGER, ASSR. to O. H. WURSTER (U.S.P. 1,743,488, 14.1.30. Appl., 27.4.25).—Glycerin is distilled in an apparatus such as that described in U.S.P. 1,743,289 (B., 1930, 492), preferably making use of a vacuum.

B. M. VENABLES.

Releasing the pressure of mixtures of solids and liquids existing under high pressure. C. F. R. HARRISON, S. LABROW, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 333,210, 4.5.29).—The condition necessary to avoid abrasion of let-down devices is found to be that at least part of the potential energy of the fluid must be converted into work without development of any considerable kinetic energy. The material is therefore exhausted through a pump used as a motor. Pumps of the screw-viscosity, drum, gear, or ram types may be used.

B. M. VENABLES.

Fractional distillation. W. F. STROUD, JUN., and

R. B. CHILLAS, JUN., ASSRS. to ATLANTIC REFINING CO. (U.S.P. 1,744,421, 21.1.30. Appl., 17.12.28).—To reduce the volumes of the vapours and the reflux liquid in the upper part of a fractionating tower, liquid is withdrawn from one or more intermediate points, preferably at a greater rate than the downflow, is cooled, and part or all returned to a tray or trays not below that from which it was withdrawn.

B. M. VENABLES.

Apparatus for vapour fractionation. J. C. MORRELL, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,744,134, 21.1.30. Appl., 16.5.27).—The upflow passages in a fractionating column are in the form of pipes with closed upper ends, the upper cylindrical walls being perforated. Below the trays are diaphragms, as described in U.S.P. 1,738,386 (B., 1930, 590), with the addition of unobstructed apertures through the centre.

B. M. VENABLES.

Purification of liquids. STANDARD OIL DEVELOPMENT Co., ASSCES. of F. W. ISLES (B.P. 314,016, 8.6.29. U.S., 21.6.28).—A liquid is treated with another which is immiscible and of different density by flowing them countercurrent in a substantially horizontal conduit. Turbulence is promoted by a number of jets of the heavier liquid inserted through the upper wall of the conduit, and the liquids are afterwards separated by settling in columns.

B. M. VENABLES.

Preparation of dispersions. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 334,100, 9.10.29).—A dispersion of, *e.g.*, 55% of asphalt in 41% of water, is prepared by the aid of 4% of dispersing agent by known means such as a mixer followed by a high-speed beater to reduce the viscosity, then, as a separate operation, further quantities of asphalt and water are added to bring the final dispersion to 61% of asphalt without any further addition of dispersing agent, but with control of the p_H at one or more stages.

B. M. VENABLES.

Apparatus for mixing and emulsifying liquids. P. L. A. THIBERGE (B.P. 312,649, 29.5.29. Fr., 29.5.28).—The constituents are contained in separate reservoirs and delivered to the emulsifier by individual pumps of equal capacity, driven by a common shaft. The proportioning is effected by three-way valves which by-pass more or less of the constituents back to the reservoirs. The emulsifier proper may be in the form of a ball bearing and the constituents enter at different points.

B. M. VENABLES.

Spraying or atomising of liquids. K. W. BRANCZIK (B.P. 333,701, 8.7.29).—The liquid is passed through a conduit which is bent to form a helix of increasing radius and terminates in a spraying nozzle.

B. M. VENABLES.

Apparatus and method for crystal growth. IMPERIAL CHEM. INDUSTRIES, LTD., and H. E. COCKSEGE (B.P. 333,598, 18.4.29).—The crystallising vessel has a vertical cylindrical partition and curved bottom, forced circulation being maintained downwards in the centre and upwards in the annular compartments. Crystals are drawn off downwards through an elutriation column in which the rising current is either fresh liquor or is crystallising liquor drawn off from the upper part of

the crystalliser through a screen and slightly reheated or diluted to destroy small crystals. B. M. VENABLES.

Salinometers and the like. EVERSHED & VIGNOLES, LTD., and C. E. PERRY (B.P. 333,727, 2.8.29).—A salinometer of the type in which the electric conductance of a column of liquid is measured is provided with temperature compensation in the form of an insulating mass which, under control of a thermostat, is more or less submerged in, and changes the cross-section of, the column of liquid. B. M. VENABLES.

Apparatus for separation of gas mixtures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,907, 15.4.29).—Partial separation of the constituents of a gas is effected by diffusion into a liquid of which a large surface is exposed and is simultaneously vaporising, the evolution of the desired constituent being effected by condensing the vapour. *E.g.*, a mixture of 72% of hydrogen and 28% of nitrogen is passed upwardly around an annular mass of Raschig rings, which are irrigated with water at 90°, the vapour being condensed on a central cooled tube; the gas, after passing once through the apparatus, contains 82% of hydrogen.

B. M. VENABLES.

Separation of gaseous mixtures by diffusion. I. G. FARBENIND. A.-G. (B.P. 308,792, 11.3.29. Ger., 31.3.28).—A non-porous foil of a metal that will combine with or dissolve the desired constituent of the gas is supported on a suitable permeable support and subjected to the mixed gas on one side at much higher pressure (preferably at least 20 atm. in excess) than that on the other. *E.g.*, a disc of palladium (10 cm. in diam. and 0.04 mm. thick) supplied with industrial hydrogen on one side at 70 atm. pressure will yield by diffusion 900 litres of very pure hydrogen per hr. when the temperature is 400° and the lower pressure 1 atm.

B. M. VENABLES.

Separation of gaseous mixtures by liquefaction. L'AIR LIQUIDE SOC. ANON. PUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 333,127, 29.11.29. Fr., 18.12.28. Addn. to B.P. 263,732; B., 1927, 320).—In the modified process the wanted gas of low b.p. which has been cooled and purified in the triple-flow exchanger is, before expansion, further cooled (and purified) by itself after expansion in an ordinary exchanger.

B. M. VENABLES.

Treatment of vapours, gases, etc. N. G. LINDERBORG (U.S.P. 1,756,693, 29.4.30. Appl., 28.4.23).—Vapours etc., *e.g.*, those produced during the manufacture of lubricating oil, are withdrawn under vacuum and simultaneously condensed by a cooling medium, *e.g.*, water, in an ejector device; after leaving the ejector the mixture is passed through an eddying chamber and then to a separating chamber. Any vapours arising from the separating chamber may be scrubbed and absorbed in separate devices.

B. M. VENABLES.

Method and apparatus for performing chemical synthesis. HIRSCH KUPFER- u. MESSINGWERKE A.-G. (B.P. 314,999, 4.7.29. Ger., 6.7.28).—Metallic or other conducting bodies are placed with the material in an autoclave and are heated and stirred by induced alternating currents and fields.

B. M. VENABLES.

Vacuum drying apparatus. A. E. JONSSON (U.S.P. 1,775,397, 9.9.30. Appl., 1.6.28. Swed., 13.6.27).—See B.P. 292,105; B., 1929, 496.

Apparatus for production of dispersions of solids in liquids. W. H. WHATMOUGH, ASSR. to STANDARD PRODUCTS CORP. (U.S.P. 1,774,910, 2.9.30. Appl., 4.10.28. U.K., 14.10.27).—See B.P. 304,178; B., 1929, 268.

Colorimeter. F. TWYMAN and J. PERRY, ASSRS. to A. HILGER, LTD. (U.S.P. 1,775,148, 9.9.30. Appl., 30.8.29. U.K., 18.8.28).—See B.P. 324,351; B., 1930, 400.

Furnace roofs. J. CHASSEUR (B.P. 333,943, 24.5.29).

Heat-exchanging devices, particularly for condensers of refrigerating apparatus. ELECTROLUX, LTD. From PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 334,333, 19.7.29).

Refrigeration apparatus for vehicles fitted with cold-storage rooms, more particularly railway refrigeration cars. SILICA GEL CORP., Assees. of E. B. MILLER (B.P. 310,336, 22.4.29. U.S., 23.4.28).

[Shaking-table] separation of dry materials [by means of upward air currents]. C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 333,622, 17.5.29).

Spraying or atomising devices suitable for use as liquid fuel burners or road sprayers. C. R. G. BEADLE and J. A. GAULD (B.P. 333,883, 20.2.29).

[Internally-lined] valves for corrosive fluids. P. JULIEN (B.P. 334,150, 27.12.29. Fr., 29.1.29).

Rotary furnaces (B.P. 331,673). **Combined gas and steam producer** (B.P. 309,048).—See II. **Treatment of filtering materials** (U.S.P. 1,742,433).—See VII. **Annealing furnaces** (B.P. 333,596).—See VIII. **Purification of gases** (B.P. 316,626 and 333,427).—See XI. **Distilling water** (B.P. 334,141).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Asturian coal. III. **Enrichment of a gas oil.** B. A. BUYLLA (Anal. Fis. Quím., 1930, 28, 959—966; cf. B., 1930, 540).—On berginisation for 1 hr. of an oil (32% of volatile matter) at a maximum temperature of 450° and a maximum pressure of 246 atm., a yield of 22.2% of oil and 64% of solid was obtained. The solid residue contained 29% of volatile substances, 9.4% of primary tar, and 59.5% of hard and coherent coke.

H. F. GILLBE.

Coal-tar pitch as a binding agent for bituminous coal briquettes. A. SPILKER [with G. BORN] (Brennstoff-Chem., 1930, 11, 307—318).—Previous work on the briquetting of coal, particularly brown coal, is reviewed. Experiments have been carried out to determine the influence of size of coal and the proportion and quality of pitch on the strength of briquettes made from a bituminous coal under otherwise similar conditions. The tensile strength and breaking strength of the briquettes were determined by the methods used in the cement industry, and the cohesion was determined by a modified "trommel" test. The briquettes were allowed to dry to constant weight before being tested.

By varying the size of coal within the limits usual in briquetting it was possible to bring about greater variations than were produced by varying the proportion of pitch by 25%. Other conditions being equal, coal of size 3—0 mm. gave the strongest briquettes. An increase in strength was produced also by using a more finely-ground pitch. The results emphasise the importance of a uniform distribution of pitch in the mixture. Softer pitches gave briquettes of higher cohesion but lower tensile strength; with too soft a pitch, however, the trommel test failed.

A. B. MANNING.

Apparatus for determining the plastic range of bituminous coals. R. KATTWINKEL (Brennstoff-Chem., 1930, 11, 329—330).—The coal, either powdered or briquetted, is heated in a small silica tube placed in the centre of a thick-walled aluminium cylinder. The tube is provided with a ground stopper and gas outlet tube. A narrow slot in the cylinder permits the sample of coal to be observed. The cylinder is heated by a gas burner, rather rapidly to 250°, and thereafter at 5°/min. Temperatures are taken at the following points: (a) the bitumen decomposition point, characterised by the condensation of oil in the stopper and outlet tube; (b) the softening point, at which the coal begins to swell; and (c) the solidification point, at which swelling ceases. The plastic range is the interval between (b) and (c). After determining the solidification point the stopper is removed and the tube heated at 520° for 10 min. The yield of semi-coke, given by the weight of the residue, agrees to within 1—2% with that given by Fischer's aluminium assay. A number of typical results are tabulated.

A. B. MANNING.

Analytical characteristics of coals. W. FUCHS (Brennstoff-Chem., 1930, 11, 332—334).—The chemical methods available for the characterisation of coals are grouped into four classes: (a) ultimate analysis; (b) proximate analysis; (c) decomposition analysis, *i.e.*, determination of moisture, ash, bitumens, humins, humic acids, and humates (*cf.* B., 1929, 5); and (d) determination of definite atomic groupings, *e.g.*, CO_2H , OH, double linkings, etc. The application of these methods is briefly discussed.

A. B. MANNING.

Lignite. I. Analysis of lignites of varying degrees of carbonisation. II. Properties and composition of humic acid. R. ODA (J. Soc. Chem. Ind., Japan, 1930, 33, 302—304 B, 304—305 B).—I. To follow the change of composition with carbonisation, recent, middle-aged, and old lignites have been analysed. The water content increases with age, 13—17—24%, also the amount of ash, 1—8—21%, but the bitumen present remains constant at about 2%. The humic acid content rises from 5.65% (containing 3.51% OMe) in recent lignite to 34.2% (1.20% OMe) in middle-aged lignite, and decreases to 2.2% (1.53% OMe) in old lignite. Cellulose, determined (a) by treatment with 42% hydrochloric acid and (b) by Schmidt's chlorine dioxide method, gives figures (a) 19.6—9.2—2.2%, and (b) 19.5—4.4%—trace for the three lignites. The recent lignite contains neither humin nor humus carbon, but much lignite (56.9%), although Karrer's acetyl bromide method indicates only 4.12%; this showed that the lignin is already somewhat altered. In the middle-

aged and old lignites the amount of humin and humus coal increases to 19 and 47.5% respectively, whilst the lignin figures determined by Karrer's method are 1.92% and a trace.

II. The humic acid from middle-aged lignite has the following composition: C 57.53, H 4.69, OMe 1.20, carbonyl oxygen 3.25, ash 5.94%; it shows strongly acid properties, and on the assumption that it is a true acid the acidity in equivalents of evolved carbon dioxide is 18.65%. Humic acid liberates iodine from potassium iodide solution, and reduces Fehling's solution. Methylation by treatment with hydrogen chloride in absolute methyl alcohol, followed by four-fold treatment with diazomethane, gives a methoxyl content of 12.06%, which is composed of 7.66% ester-methoxyl, determined by von Fellenberg's method, and 6.60% ether-methoxyl. From these figures it is calculated that humic acid contains 1.87% OH and 11.70% CO_2H .

C. W. SHOPPEE.

Behaviour of coal during carbonisation. I. Change of moisture-absorbing power of coal by carbonisation. II. Change of electrical conductivity of coal during carbonisation. III. Change of composition of coal by carbonisation. S. IKI (J. Soc. Chem. Ind., Japan, 1930, 33, 320—321 B, 321—322 B, 322—323 B).—I. Samples of anthracite, bituminous coals of different caking power, and brown coals were pulverised to a definite degree of fineness (below 100-mesh), heated for 15 min. at 100—1000°, and the resulting cokes pulverised to below 100-mesh. The power of absorbing moisture gradually decreases by heat-treatment, becoming minimal for a sample carbonised at 400° for bituminous coal and 600—700° for brown coal; by heating at higher temperatures it increases rapidly and becomes maximal at 700—800° for high-caking bituminous coal, but lower-caking bituminous coal, anthracite, or brown coal only attain this maximum at 1000°. The relationship between moisture-absorbing power and the temperature of carbonisation of wood charcoal is nearly the same as that of coal, especially low-grade brown coal. There is a close relationship between the moisture-absorbing power of coke and the caking power of the parent coal; the higher the caking power of the original coal, the smaller is the power of moisture absorption of the coke. The moisture-absorbing power of carbons decreases with increase of the degree of carbonisation, *i.e.*, retort carbon > blast-furnace coke > gas coke > coalite > charcoal > activated carbon; in the same sample of retort carbon or blast-furnace coke the lustrous hard portion, which appears to be the decomposition product of bitumen, has a smaller power of moisture absorption than the dull porous portion. The moisture-absorbing power of cokes and carbons thus forms an important indication of their origin, temperature of carbonisation, and properties.

II. In continuation of previous work (B., 1929, 344), determinations of the electrical conductivity during carbonisation of the samples used above has been carried out by Sinkinson's method (B., 1928, 734). This value for coal increases gradually from 400°, rapidly from 700°, becoming nearly constant at 1000°. The change of electrical conductivity of wood by carbonisation at various temperatures is nearly the same as

for coal, especially low-grade brown coal. Coke prepared from higher-caking coal has a greater conductivity than that from lower-caking coal. The electrical conductivity of various carbons increases with the degree of carbonisation in the order coalite < gas coke < blast-furnace coke < retort carbon < graphite; in the same sample of retort carbon or blast-furnace coke the lustrous hard portion has greater conductivity than the dull soft portion. The degree of carbonisation of coalite, charcoal, and activated carbon is thus indicated by the electrical conductivity.

III. Changes in the amounts of volatile matter and fixed carbon are hardly noticeable below 400°, but become important from 400 to 500° and occur up to 700°. Low-grade coal exhibits these changes up to a higher temperature than bituminous coal. The amount of volatile matter decreases with increased carbonisation, and constitutes a method of estimating the degree of carbonisation. Below 400° the content of bitumen-A and -B increases in high-caking coal and decreases in low-caking and low-grade coals, and in all cases bitumens become readily extractable by heating; at about 500° all bitumen disappears. The content of humic substances in brown coal gradually decreases by heating, becoming nearly zero at 600°. These facts explain to some extent the cause of the change of moisture-absorbing power of coal by heating. C. W. SHOPPEE.

Heat transfer in the low-temperature carbonisation of coal. V. C. ALLISON (Ind. Eng. Chem., 1930, 23, 839–843).—A formula has been developed to fit the experimental results obtained in a series of nine tests where Pittsburg seam coal was carbonised in vertical cylindrical metal retorts varying in radius from 1.03 to 6.28 in. and at retort temperatures of 590–780°. Low-temperature carbonisation was considered to be complete when the centre of the middle of the charge attained a temperature of 390°, as this was the only definite "break point" on the time-temperature curve above the 100° water-evaporation flat section, and is immediately followed by increased heat-penetrability of the charge, as is shown by the increased steepness of the curve. Of the total loss in weight obtained under low-temperature carbonisation conditions, 95% has occurred when this 390° point is reached, for this particular coal. From the formula the carbonising time can be calculated. The formula is extended to retorts of various shapes. The mechanism of heat penetration of a charge of coal is also discussed. H. S. GARLICK.

Circulating-stream coke ovens. O. PEISCHER (Stahl u. Eisen, 1930, 50, 761–767).—This type of coke oven has twin flues and a portion of the burnt gases is drawn by suction back into the incoming stream of heating gas and air so as to retard the combustion, avoid overheating, and regulate the heat flow from gas to coking retort. A furnace of this kind is described and illustrated diagrammatically, and a brief account is given of its operation with details of the temperature distribution in the coking chambers and flues, and of the yield of coke, fuel consumption, and methods of charging and discharging. A. R. POWELL.

Determination of the sp. gr. and macro-pore volume of coke and other porous substances. G.

STADNIKOV (Brennstoff-Chem., 1930, 11, 330–331).—A piece of coke is immersed in a bath of paraffin wax, the pressure on which is first reduced in order to remove all air from the macro-pores, and is then increased to 1.5–2 atm. When cold the impregnated coke is withdrawn and a piece of suitable size is cut from the mass. The volume of this piece is determined in a pycnometer, and the wax therein is then extracted with light petroleum in a Soxhlet apparatus, recovered by evaporation of the solvent, and weighed. The volume of the wax, calculated from its known sp. gr., gives the volume of the macro-pores. The true sp. gr. of the coke is given by wt. of coke/(vol. of impregnated coke—vol. of macro-pores). In metallurgical and gas cokes this figure agrees with the sp. gr. determined directly on the powdered material. It appears, therefore, that the volume of the micro-pores is negligible in these cokes. A similar comparison, however, shows that in low-temperature cokes the micro-pores possess an appreciable volume.

A. B. MANNING.

Determination of the "lump density" of coke by coating the surface with paraffin wax. F. G. HOFFMANN (Brennstoff-Chem., 1930, 11, 297–299).—Weighed pieces of coke are immersed in a bath of paraffin wax at 125°, are held therein until the bath has cooled to 75°, and are then withdrawn and allowed to drain. The wax thereby penetrates a few mm. into the pores of the coke, but leaves a layer of only negligible thickness on the outer surface. The volume of the coated coke is then determined by displacement, using a mixture of alcohol and water as displacement liquid, and a 2-litre beaker as a pycnometer. A simple attachment provided with a pointed screw permits the beaker to be filled to exactly the same level each time. By using only a few pieces of coke of total weight at least 500 g. the density can be determined with an accuracy of ± 0.1 –0.2%. A. B. MANNING.

Reproduction of coke sections. ZIPPERER and LORENZ (Gas- u. Wasserfach, 1930, 73, -606–607).—The coke section is coated electrolytically with a layer of metal. It is then polished and used as a block for printing directly on to ordinary paper.

A. B. MANNING.

Existence of two varieties of amorphous carbon. M. OSWALD (Chim. et Ind., 1930, 24, 280–292).—Technical amorphous carbons are, in general, intimate mixtures of two amorphous varieties of carbon, brown and grey, respectively. The brown form is the only truly amorphous modification of carbon, and its formation is favoured by the use of aliphatic substances for carbonisation, by the presence of oxygen atoms in the molecule, by low temperature of carbonisation, rapid cooling, and high rigidity of the medium. The reverse conditions favour the production of the grey form, which is pseudo-amorphous, giving an X-ray diffraction pattern like that of graphite. The grey form is produced by the carbonisation of aromatic compounds and is favoured by the presence of sulphur or nitrogen atoms in the molecule. E. S. HEDGES.

Liquid products of the berginisation of coal. J. M. PERTIERRA (Anal. Fis. Quím., 1930, 28, 792–806).—A sample of coal on treatment by the Bergius process

gave phenols 32%, bases 8.2%, and neutral oil 59.8%; the initial pressure had but little influence on these proportions. The fraction of the liquid products boiling between 95° and 145° contained 52% of aromatic hydrocarbons (chiefly benzene, toluene, and *m*- and *p*-xylenes), 28.7% of paraffins, and 7.3% of unsaturated compounds. Among the phenolic products phenol, *o*- and *m*-cresols, and pyrocatechol have been identified, whilst pyridine and 2:4-dimethylpyridine have been detected in the basic fraction.

H. F. GILLBE.

Elasticity of production as a factor in the progressive economy of gas works. R. MEZGER (Gas-u. Wasserfach, 1930, 73, 557–560, 589–595, 607–612, 627–632, 655–659).—In order to meet the varying demand for gas throughout the year the plant should conform with the following requirements: (a) high elasticity, *i.e.*, high range of variation in the rate of gas production; (b) low cost of production per unit, with the smallest variation in cost with varying rate of production; (c) high ratio of cost of operation plus cost of raw materials to fixed charges at full load; and (d) ease of control, *i.e.*, possibility of rapidly varying the rate of production. The cost of production and the elasticity of operation with horizontal and vertical retorts and coke ovens have been calculated; variations in carbonisation time, volatile content of coal, degree of de-benzolisation of the gas, simultaneous production of water-gas in the retort, and addition of rich gas to the gas used for heating the retorts have all been taken into consideration; the results have been tabulated and plotted. Similar series of results have been calculated for gas generators and for combinations of retorts with generators. In many cases the peak load can be economically covered by the utilisation of carburetted water-gas plants in conjunction with horizontal or vertical retorts.

A. B. MANNING.

Gas dehydration. K. FRANK (Gas-u. Wasserfach, 1930, 73, 637–638; cf. Mezger and Pistor, B., 1930, 594).—Gas dehydration by cooling can simultaneously bring about the removal of the naphthalene (cf. Lenze and Rettenmaier, B., 1926, 938). The question is raised as to whether the use of calcium chloride can also lower the naphthalene content of the gas, and whether the life of the solution is affected by the separation of tarry polymerised hydrocarbons. The utilisation of waste heat in bringing about refrigeration of the gas is cheaper than cooling by compression. It would seem preferable to place the refrigerating plant after instead of before the gas holder because (a) in spite of the oil insulating layer there is a danger of the gas again taking up water, (b) there is possibility of corrosion of the seams of the holder which are not "wetted" by oil, and (c) the temperature of the gas leaving the holder is always lower than that entering.

A. B. MANNING.

[Gas dehydration.] MEZGER and PISTOR (Gas-u. Wasserfach, 1930, 73, 638).—A reply to Frank (preceding abstract).

A. B. MANNING.

[Gas-works'] benzol recovery. F. GOLDSCHMIDT (Gas-u. Wasserfach, 1930, 73, 636–637).—Some recommendations are made regarding details of practice. The naphthalene content of the wash oil should not be

allowed to rise above 10–15% or difficulties may arise due to the deposition of naphthalene in the pipes behind the scrubbers or in the purifiers; by cooling the gas before it reaches the scrubbers its naphthalene content may be lowered and the useful life of the wash oil thereby lengthened. Fresh oil should be added to the wash oil to replace that removed with the benzol. The first separation of the crude benzol from the wash oil should be carried out rapidly; to avoid undue loss of benzol thereby the condensing system should be provided with a sufficiently large area of cooling surface. The danger of decomposition in heating the wash oil as high as 130–145° is negligible. Steam-distillation of the benzolised oil can be continued until the benzol content falls to 0.3%.

A. B. MANNING.

Absorption pipette for exact gas analysis. E. OTT (Gas-u. Wasserfach, 1930, 73, 801–802).—The "double pipette," in which oxygen, carbon dioxide, and carbon monoxide are absorbed in one bulb and hydrocarbons in a second one, has been improved by the addition of a further pipette, packed with glass tubing like that used for hydrocarbons and charged with acid iodine pentoxide for carbon monoxide absorption. Absorption is complete in 1–2 min., and acid vapours must be removed with caustic potash before the reading is taken. The presence of carbon monoxide is indicated by a brown or green coloration on the liquid surface, which disappears when absorption is complete.

C. IRWIN.

Determination of carbon monoxide in illuminating gas. J. DUBOIS (Przemysl Chem., 1930, 14, 313–318).—The time necessary for the complete absorption of carbon monoxide from illuminating gas can be reduced from 60 to 20 min. by shaking the gas twice for 2 min. with 10 c.c. of acid cuprous chloride reagent, and 3 times with 5-c.c. portions of ammoniacal cuprous chloride. Complete absorption can also be attained by using Damiens' reagent (acid cuprous sulphate solution) in three 3-c.c. portions, shaking each time for 3 min.; carbon dioxide, olefines, and oxygen must, however, first be removed from the gas, as these are also quantitatively absorbed.

R. TRUSZKOWSKI.

Self-carburization of industrial gases from brown coal. M. FREUND and E. BECKER (Brennstoff-Chem., 1930, 11, 335–337).—Brown-coal tar and pitch were cracked in the laboratory by being dropped through an electrically-heated tube packed with iron shavings. The maximum efficiency of gasification, *i.e.*, on the thermal basis, was obtained at a cracking temperature of 750–800°, and amounted to 48% with the tar, and 37% with the pitch. The composition of the gas produced was dependent principally on the cracking temperature, and only to a small extent on the raw material or the working pressure.

A. B. MANNING.

Formation of benzine and tar from ethylene by heating under ordinary pressure without catalysts. H. I. WATERMAN and A. J. TULLENERS (Brennstoff-Chem., 1930, 11, 337–340).—Ethylene was passed through an electrically-heated silica tube, and the variation in the yields of tar and light oil with temperature were observed. The rate of passage of the gas was about 1 g./min., the tube was 20 mm. in diam., and the

heated length 60 cm. The yield of light oil was negligible at 600°, and increased to a maximum of about 13% at 750°. The formation of tar began at 650° and passed through a maximum of 24.4% at 800°. Above 800° the yield of solid carbon increased rapidly. The bromine value of the light oil decreased rapidly as the temperature rose (from 123 at 650° to 0 at 900°), due probably to the increasing proportion of aromatic hydrocarbons.

A. B. MANNING.

Knock rating of straight-run Pennsylvania gasoline in relation to b.p., density, and index of refraction. M. R. FENSKE (Ind. Eng. Chem., 1930, 22, 913).—The gasoline was fractionally distilled through a column consisting of a 35-ft. iron pipe (diam. 3 in.), suitably lagged and packed with alternate 6-in. sections of glass rings and chain. Several sections were electrically wound, and the head of the column was arranged to give any desired reflux ratio. Heat losses were controlled by thermocouples. The distillation lasted for 45 hrs. About 16% of the gasoline consisted mainly of butanes and pentanes which were condensed in Dry-Ice, and the gasoline (b.p. 43–256°) was separated into 165 fractions. The values of d and n of each fraction were determined, and type fractions were tested for knock characteristics. Several fractions boiling at the same temperature show remarkable variations in these characteristics. Generally, the fractions having the highest values for d and n had the highest knock ratings, and *vice versa*. The data for those fractions for which any of the above-mentioned properties show a maximum or minimum are tabulated.

H. S. GARLICK.

Explosion pressures of mixtures of benzene and petroleum. ANON. (Jahresber. VII Chem.-Tech. Reichsanst., 1928, 249–252; Chem. Zentr., 1930, i, 2039).—The explosion pressures of mixtures of benzene, petroleum, and air at 0–60° were determined; maximal pressures (9 kg. per cm.² at 0°) were attained with approximately equal concentrations (9 wt.-%) of the fuels. With rise of temperature smaller values of pressure were obtained.

A. A. ELDRIDGE.

Explosion of gasoline and oxygen. C. K. FRANCIS (Ind. Eng. Chem., 1930, 22, 896).—Fifty c.c. of gasoline from a blend of straight-run and cracked distillates were placed in a $\frac{3}{4}$ -in. steel bomb, and oxygen was admitted and allowed to escape. On re-admitting oxygen no pressure was registered on the gauge, and on increasing the pressure of oxygen a disastrous explosion resulted. It is concluded that the oxygen-bomb test for potential gum formation offers too much hazard.

H. S. GARLICK.

Explosion-proof lubricants. ANON. (Jahresber. VII Chem.-Tech. Reichsanst., 1928, 112–126; Chem. Zentr., 1930, i, 2038).—Experiments with glycerol, paraffin oil, and rape oil demonstrated the advantage of the first-named. Experiments on ignitability in Mackey's apparatus are described.

A. A. ELDRIDGE.

Motor spirits containing alcohol. I, II. W. SWIENTOSLAWSKI (Przemysl Chem., 1930, 14, 337–345).—The phenomena of azeotropism are applied to the study of motor spirits composed of alcohol and petrol, with the addition of ether, benzol, solvent naphtha, etc.

R. TRUSZKOWSKI.

Combustion at a gasoline burner in a confined atmosphere. E. HAUSER (Anal. Fis. Quím., 1930, 28, 742–748).—The variations of composition of the air in an enclosed vessel containing a burning jet of petroleum vapour have been studied. The carbon monoxide content rises rapidly to a maximum, and then diminishes as the moment of extinction of the flame approaches; the carbon dioxide content, however, rises rapidly for about one third of the total time, becomes almost constant, and finally rises rapidly. Small quantities of acetylene, as well as of other hydrocarbons, appear immediately prior to the extinction of the flame.

H. F. GILLBE.

Determination of total sulphur in combustible liquids. L. BERMEJO (Anal. Fis. Quím., 1930, 28, 807–818).—Available methods for the determination of sulphur in liquid fuels are criticised and compared, and a new method is proposed based on the oxidation by hypobromite of the sulphur dioxide produced by combustion, and precipitation of the sulphate formed as barium sulphate. Greater accuracy is claimed for this procedure than is obtainable by the bomb method.

H. F. GILLBE.

Sludge of transformer oils. IV. Effect of various metallic soaps of naphthenic acids on the oxidation of less refined insulating oils, with special reference to the antioxidative action of tin naphthenate. T. YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 318–319 B; cf. B., 1930, 750).—The oxidation tests are carried out on oils, to which 2% of various metallic naphthenates have been added, at 120° for 100 hrs. in an open vessel. Copper, iron, manganese, silver, cobalt, and cerium naphthenates produce a large sludging effect, whilst calcium, barium, mercury, potassium, magnesium, and tin naphthenates exert an anti-sludging action. The addition of 0.1% of the last-named prevents sludge formation and discoloration under the above conditions. The addition of tin naphthenate decreases the breakdown voltage of the oils by less than 0.1%.

C. W. SHOPPEE.

Effect of various metallic soaps of naphthenic acids on the oxidation of highly refined transformer oils, with special reference to the antioxidative action of tin naphthenate. T. YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 319 B; cf. preceding abstract).—The addition of tin naphthenate prevents sludging and inhibits the formation of acids.

C. W. SHOPPEE.

Selection of petroleum oil for spraying purposes. E. R. DE ONG (Ind. Eng. Chem., 1930, 22, 836–839).—The relative value of volatility, viscosity, sulphonatable value, and oxidation reaction are now sufficiently recognised as to be incorporated in practically standardised specifications for petroleum oils suitable for use on both dormant and active plants. The high standard of refining at first considered necessary has led to the production of oils so bland that much of their toxicity to insects has been lost. This has been overcome by improved methods of refining, incorporation of other chemicals, and by the use of more toxic petroleum fractions, especially those containing cyclic organic sulphur compounds, used either straight or in the form

of an emulsion in which the percentage of emulsifier is kept as low as possible. Petroleum oils for orchard spraying should also possess high powers of penetration, low surface tension, and high solvent power for other and more active chemicals.

H. S. GARLICK.

Decomposition of acetylene. ANON. **Oxidation of toluene.** SHENDEROVICH and LIVSHITS.—See III.

See also A., Sept., 1108, **Adsorption of xanthates on carbon suspensions** (PETERSEN). 1127, **Propagation of combustion in hydrocarbon mixtures** (AUBERT and DUCHÈNE). 1132, **Catalytic reduction of carbon monoxide** (KODAMA). 1152, **Determination of ignition temperature of powder substances** (HARRISON). 1153, **Fractionating head [for tar]** (MAINZ).

PATENTS.

Combustion of pulverised fuel, more particularly pulverised coal. J. P. GOOSSENS (B.P. 331,555, 4.4.29).—The pulverised fuel is injected into the furnace, from a star-shaped orifice, in the form of thin, intersecting sheets, and the amounts of both primary air and carrier air are kept so low that only a gasification of the core of pulverised coal takes place before the point at which the secondary air encounters the jet. The secondary air is injected into the fuel flame at an acute angle in such a way as to produce turbulence and ensure as complete a combustion of the fuel as possible. A number of the fuel burners may be combined together in a casing to form the furnace front.

A. B. MANNING.

Rotary furnaces more particularly adapted for low-temperature distillation. KOHLENVEREDLUNG A.-G., and C. GEISSEN (B.P. 331,673, 17.6.29).—A rotary retort is filled with chequer brickwork forming a series of adjacent parallel passages extending the length of the retort. Through these the raw material and/or the heating gases are passed uninterruptedly to maintain the retort in continuous operation. A number of the passages may act as distilling chambers and the remainder as heating passages, the latter opening into antechambers at each end of the retort. In another modification all the passages communicate with means for admitting and discharging both the raw material and a heating and scavenging gas.

A. B. MANNING.

Carbonisation of material at low temperatures. C. PAMART (B.P. 331,497, 2.3.29. Addn. to B.P. 268,613; B., 1927, 468).—Powdered bituminous fuels, especially those of high agglutinating properties, are preheated, then partially oxidised, and finally subjected to low-temperature carbonisation in a retort similar to that described in the prior patent. The preheating is carried out in a similar, slightly inclined, rotary retort, arranged above the carbonising retort in the same heating enclosure. The oxidation is carried out in a screw conveyor which carries the preheated fuel from the outlet of the upper retort back to the inlet of the lower retort. The oxidising apparatus is provided with means for the introduction along its length of an adequate supply of air.

A. B. MANNING.

Destructive distillation [of carbonaceous materials]. E. ROSER (B.P. 314,078, 19.6.29. Ger., 23.6.28).

—The fuel is packed into iron tubular containers which are then introduced into a retort in which the fuel is distilled. The retort consists preferably of a series of vertical distilling tubes within a suitable combustion chamber. Jacketed tubes attached to the distilling tubes serve to cool the carbonised material and at the same time to heat water or air.

A. B. MANNING.

Destructive hydrogenation of coal, oils, etc. IMPERIAL CHEM. INDUSTRIES, LTD., and (A) H. G. WATTS, (B) C. F. R. HARRISON (B.P. 331,509 and 331,548, 2.4.29). —(A) The lower paraffin hydrocarbons are removed together with the benzene from the gaseous products of destructive hydrogenation by subjecting them, under pressure, to cooling in such a manner that the gas is brought into intimate contact with the condensed benzene. The latter may be supplemented by benzene which has previously been used for scrubbing the gas and has been degasified by release of pressure. (B) The compressed hydrogen to be used in the destructive hydrogenation process is heated by making use of the reaction $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O} + 47,800$ g.-cal. Hydrogen containing 3–5% of carbon monoxide, a mixture readily obtainable from water-gas by known means, is suitable for the purpose. The gas is preheated and then passed over a nickel catalyst, the reaction preferably being carried out in the same high-pressure vessel as the hydrogenation.

A. B. MANNING.

Manufacture of liquid products from coal or carbonaceous material, and liquefaction of these materials. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 311,759, 4.4.29. Holl., 16.5.28. Cf. B.P. 309,859; B., 1930, 853).—The initial materials are heated with hydrogen under pressure, preferably in the presence of one or more catalysts, without intensive splitting giving rise to the formation of gaseous products or carbonaceous deposits; as soon as the desired reaction temperature is reached the heating is stopped and the products which are in a gaseous state are withdrawn. The residue may be submitted to further destructive hydrogenation.

A. B. MANNING.

Combined gas and steam producer. C. MARISCHKA (B.P. 309,048, 3.4.29. Austr., 3.4.28).—The water tubes of a boiler are packed tightly against one another to form the wall surrounding the fuel bed of a gas producer. Between this wall and the outer casing of the apparatus is an annular gas jacket. When the apparatus is used for the generation of ordinary producer gas the hot gases produced in the inner chamber are passed into the gas jacket through gaps formed between the upper ends of the water tubes. When the apparatus is used for the generation of water-gas the water tubes form a gas-tight partition between the two chambers, and hot combustion gases formed by burning the blow gases in a separate combustion chamber are passed through the gas jacket.

A. B. MANNING.

Production of mixed oil gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (B.P. 331,756, 10.9.29. U.S., 3.1.29).—A carburetted water-gas plant is operated with the following sequence of steps: (a) the fuel bed is air-blasted and the blow gases are burned with secondary air in the carburettor

and superheater; (b) oil is passed through the carburettor and superheater, in the same direction as was taken by the blow gases, and then through the fuel bed, wherein it is cracked; and (c) the fuel bed is steamed to make water-gas and to remove the deposited carbon.

A. B. MANNING.

Recovery of acetylene from gaseous mixtures. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,610, 20.4.29).—The gaseous mixtures are scrubbed with a suitable solvent in stages under successively higher pressures, in each of which the partial pressure of the acetylene is not above 2 atm.

A. B. MANNING.

Removal of acetylene from gases. G. F. HORSLEY, F. ROFFEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,654, 24.5. and 9.11.29).—Acetylene is recovered from gas mixtures by scrubbing them with esters or ethers of high b.p. and low viscosity, *e.g.*, the esters, ethers, and mixed ester-ethers of glycol, glycerol, etc.

A. B. MANNING.

Separation of gases or vapours. C. G. HARRIS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,787, 24.10.29).—A mixture of olefines is separated by treatment with an adsorbent which is already largely saturated with a vapour or gas of affinity for the adsorbent intermediate between those of the olefines which it is desired to separate. Thus ethylene and propylene may be separated by passing the mixture over activated carbon saturated with water vapour.

A. B. MANNING.

Apparatus for removing solid particles from gas, especially from hot gases issuing from gas generators or producers. HUMPHREYS & GLASGOW, LTD., Assees. of E. J. BRADY (B.P. 331,755, 10.9.29. U.S., 26.10.28).—For this purpose a conical separating chamber has tangential gas inlet and outlet passages, the latter of which is placed at a higher level than the former. The chamber may be interposed between the producer and carburettor of a water-gas plant.

A. B. MANNING.

Apparatus for indicating presence of inflammable vapours or gases. H. T. RINGROSE (B.P. 331,711, 24.7.29).—In apparatus in which the combustion of the inflammable gas or vapour causes a diminution of pressure in a porous vessel, the movement of a diaphragm attached thereto is utilised to short-circuit a solenoid which normally retains a switch arm, in series with the lamp circuit, in the "make" position, the arrangement being such that when the switch arm is released by the short-circuiting of the solenoid it takes up a "break" position and causes the lamps to be extinguished.

A. B. MANNING.

Treatment of tars. G. T. MORGAN, F. S. SINNATT, and D. D. PRATT (B.P. 331,542, 4.2.29. Cf. B., 1929, 156).—Pitches, including the insoluble residues left on treating high- or low-temperature tars with light petroleum, are extracted with ether, benzene, or low-temperature tar spirit, and the solution is extracted successively with caustic soda and mineral acid. From the caustic soda extract "resinols" and "resinoic acids" are obtained by treatment with carbon dioxide followed by acidification. Neutralisation of the acid extract yields "resinamines," and evaporation of the solvent from the

residue leaves the "resinenes." The products may be purified by redissolving them in ether, benzene, etc., and reprecipitating them by the addition of petroleum.

A. B. MANNING.

Emulsification [of tar etc.]. C. G. FOX (B.P. 333,152—3, 4.2.29).—Emulsifying agents comprise (A) soap or soap-forming materials together with higher monohydric aliphatic alcohols present as esters in fats and waxes (*e.g.*, cetyl or ceryl alcohol), (B) the products obtained by treating with sulphuric or phosphoric acid liquid marine-animal waxes or fats containing material proportions of esters of monohydric aliphatic alcohols (*e.g.*, sperm oil).

L. A. COLES.

Manufacture of asphalt emulsions [using soap as emulsifying agent]. P. LECHLER (B.P. 333,496, 17.1.30. Ger., 2.2.29).—The bulk (about 75%) of the alkali necessary for soap formation is added in concentrated aqueous solution to a mixture of asphalt and a fatty acid, and the remainder is added subsequently in solutions of decreasing concentrations.

L. A. COLES.

Treatment of emulsions. W. T. REDDISH, Assr. to KONTOL Co. (U.S.P. 1,747,987, 18.2.30. Appl., 27.9.28).—Emulsions, *e.g.*, of petroleum and water, are broken by the use of "triethanolamine," especially in conjunction with a sulphonated mineral oil.

C. HOLLINS.

Cracking of oils. R. E. WILSON and R. J. DEARBORN (B.P. 318,991, 1.7.29. U.S., 15.9.28).—Oil is subjected to cracking temperatures and pressures in a cracking zone and the vapours are passed to a fractionating column maintained under superatmospheric pressure. Condensate is withdrawn from an intermediate point of the column and a portion returned to the cracking zone, the remainder passing to a second fractionating column maintained under materially lower pressure, into which a predetermined charge of fresh oil is introduced, the mixture of stripped condensate and fresh charge from the second fractionating column being returned to the cracking zone. The vapours from the second column are condensed and part of the condensate is forced into the first fractionating column near the top. The cracking zone includes a heating coil and a reaction chamber. The condensates from the two fractionating columns are returned to different points of the coil, whilst heavy unvaporised oil is withdrawn from the reaction chamber and passed to a low-pressure expansion zone formed by the lower section of the second fractionating column; released vapours are passed to the head of the column.

H. S. GARLICK.

Cracking of liquid hydrocarbons. TRENT PROCESS CORP. (B.P. 313,159, 21.5.29. U.S., 8.6.28).—Cracking is carried out in a cylinder fitted with axial heating tubes. Hot combustion gases are passed from the axis of the apparatus through the tubes and discharged into an outer cylinder. Oil is fed into the inner cylinder, which is rotated, causing the oil to be spread in thin films on the heating tubes and submitting the vapour as well as the liquid to cracking temperatures. The hot gases from the outer cylinder are used to preheat the cracking stock and also to temper the gases taken direct from the heating furnace before these are admitted to the heating

tubes. The outer cylinder is fitted with eyes so that the heating tubes are accessible for cleaning. T. A. SMITH.

Conversion of hydrocarbon oils. C. P. DUBBS and J. C. MORRELL, ASSTS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,744,109, 21.1.30. Appl., 29.5.22. Renewed 3.8.28).—An oil-water emulsion, of which oil constitutes at least 85%, is passed continuously through a heating coil, where it is raised to conversion temperature, and delivered to an enlarged zone where it is subjected to the action of catalytic material, *e.g.*, ferric oxide. Unvaporised portions are withdrawn, the vapours being separately removed and passed to a dephlegmator and condenser. H. S. GARLICK.

Converting high-boiling hydrocarbons into low-boiling hydrocarbons. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 333,335, 7.6.29).—Oil or its vapour is mixed directly with the products of combustion from an external burner in a mixing chamber, and travel through a small conduit into a larger one where reaction takes place. The mixture leaves the reaction chamber and enters another mixing chamber smaller than the reaction chamber, where it is again heated by combustion products from a second burner. The mixed gases are discharged into a second reaction chamber. The process is repeated as often as is necessary to obtain a predetermined degree of cracking, after which the hot gases and vapours enter the bottom of a fractionating column and travel counter-current to a stream of charging stock fed to the top of the column. Vapours leaving the top of the column pass to a condenser and uncondensed gases are passed to a compression or absorption plant. H. S. GARLICK.

Apparatus for refining used oil. F. O. KRIEGER (B.P. 333,271, 10.4.29).—The apparatus consists of a still and a receiver, both connected by a jacketed condensing pipe, and a device for maintaining a vacuum in the still, operated by the condensing medium passing through the jacket. Volatile vapours driven off from the used oil in the still are condensed and collected, whilst the oil remaining in the still is discharged beneath a plate supporting a clarifying substance contained in a chamber beneath the still. Clarified oil is removed through a tap in the top of the chamber. H. S. GARLICK.

Refining of heavy mineral oils by means of liquefied sulphurous acid. W. W. TRIGGS. From ALLGEM. GES. F. CHEM. IND. (B.P. 333,687, 21.6.29).—Heavy oil and liquid sulphur dioxide are added in continuous streams to a mixing vessel fitted with an agitating device. Mixture from this vessel is continuously removed to a horizontal unstirred vessel in which separation takes place, and from which the refined oil and acid solution are continuously withdrawn. The separation is facilitated by the provision, in the settling vessel, of inclined longitudinal partitions, or by admitting the mixture through a perforated tube which extends the whole length of the separating vessel. The perforations in the tube are directed upwards and the stream of oil and acid from them is directed downwards by means of a screen. T. A. SMITH.

Refining of oil, refining agent therefor, and its production. R. O. BOYKIN (U.S.P. 1,744,610, 21.1.30.

Appl., 30.4.24).—Decolorising clays for use in the refining of, *e.g.*, lubricating oil are prepared by mixing the particles of a suitable clay with a binding material to form a plastic mass which is moulded or forced through dies, to produce strings of uniform cross-section, and dried. H. S. GARLICK.

Manufacture of motor fuel. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,224, 24.4.29).—The liquid products of the destructive hydrogenation of mineral coal, *e.g.*, anthracite, or of the crude distillates therefrom, b.p. 100–200°, are blended with 5–50%, preferably 10–15%, of motor fuels having a tendency to knock (other than products obtained by destructive hydrogenation of carbonaceous materials) in amount sufficient to suppress knocking. Other anti-knock agents are added if desired. [Stat. ref.] H. S. GARLICK.

Synthetic liquid fuels. COMP. GÉN. DES PRODUITS DE SYNTHÈSE (B.P. 316,945, 2.8.29. Fr., 6.8.28).—Carbonaceous gases analogous to those obtained from a gas producer are treated at 80° and under atmospheric pressure with a catalyst which promotes polymerisation to a liquid fuel. The catalyst is produced from monazite sand, which is roasted, extracted with hydrochloric acid, the solution neutralised and evaporated, and the residue dissolved in sulphuric acid and evaporated until white fumes are evolved; cobalt sulphate solution is added to the residue, which is dissolved in water and electrolysed, and the electrolytic deposit subjected to the action of X-rays. H. S. GARLICK.

Coke ovens. E. COPPÉE (U.S.P. 1,775,191—2, 9.9.30. Appl., [A] 15.6.26, [B] 26.5.27. Belg., [A] 22.6.25, [B] 4.9.26).—See B.P. 253,887 and 273,630; B., 1927, 721.

Carbonising of coal. W. RUNGE, ASST. to INTERNAT. COAL CARBONIZATION CO. (U.S.P. 1,775,323, 9.9.30. Appl., 17.6.25).—See B.P. 264,169; B., 1927, 547.

Separation of constituents of coke-oven gases and the like. J. I. BRONN and G. FISCHER (U.S.P. 1,774,633, 2.9.30. Appl., 18.6.28. Ger., 27.6.27).—See B.P. 318,319; B., 1929, 969.

Composition for the purification of gases. A. JOSEPH, ASST. to COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (U.S.P. 1,775,366, 9.9.30. Appl., 1.3.29. Fr., 13.4.28).—See B.P. 309,585; B., 1930, 406.

Coal and other mineral sorting apparatus. W. H. BARKER and W. BENNETT (B.P. 334,392, 18.9.29).

Gas burners. BURTON FOUNDRY CO., LTD., and D. F. CAMPBELL (B.P. 334,307, 17.6.29). H. GUHL (B.P. 334,070, 31.8.28. Ger., 19.3.29).

Industrial gas-heating systems. PREMIX GAS PLANTS, LTD., and A. DOCKING (B.P. 333,924, 23.4.29).

Crushers for coal etc. (B.P. 333,642). **Pulverising mills** (B.P. 317,081). **Heating decomposable liquids** (B.P. 333,241). **Fractionation** (U.S.P. 1,744,134 and 1,744,421). **Purification of liquids** (B.P. 314,016). **Dispersions** (B.P. 334,100). **Separation of gases** (B.P. 308,792). **Treatment of vapours etc.** (U.S.P. 1,756,693).—See I. Carbon dioxide recorder (U.S.P. 1,751,265).—See VII. Synthetic resins (U.S.P. 1,744,135).—See XIII.

III.—ORGANIC INTERMEDIATES.

Dependence of the temperature of explosive decomposition of acetylene on pressure and gas-stream velocity. ANON. (Jahresber. VII Chem.-Tech. Reichsanst., 1928, 63—71; Chem. Zentr., 1930, i, 2036—2037).—Explosive decomposition of acetylene does not occur at 500° under pressures up to 3 kg. per cm.²; it was first observed at 510°, with a pressure of 2.05 kg. per cm.², and a gas velocity of 0.4 litre per min. into the explosion tube. With increasing velocity the limits of temperature and pressure are raised; the latter falls again at high temperatures and velocities. For motionless gas the pressure limit (extrapolated) for 540—900° is 1.37 kg. per cm.²; the pressure necessary to produce explosion by adiabatic compression is 170 kg. per cm.² A. A. ELDRIDGE.

Calorimetric determination of acetic anhydride. J. R. COCHRANE and C. A. SMYTHE (J. Soc. Chem. Ind. Victoria, 1930, 30, 351—353).—Acetic anhydride in acetic acid is determined by adding a measured quantity of concentrated sulphuric acid to a quantity of the mixture in a Dewar flask and observing the rise of temperature, which is proportional to the anhydride content. H. I. DOWNES.

Detection of isopropyl alcohol. J. WÜHRER (Pharm. Ztg., 1930, 75, 845—846).—*iso*Propyl alcohol (10% or more) in pharmaceutical preparations may usually be detected by its taste and odour. In absence of acetone it may be oxidised by chromic and sulphuric acids and the acetone detected by means of nitroprusside in ammoniacal solution. Of the direct colour tests for *isopropyl* alcohol involving treatment of a distillate of the drug with sulphuric acid and an aromatic aldehyde, a modification of that due to Reif (B., 1928, 686) is preferred. Traces of higher alcohols and ethereal oils which give a similar reaction may be removed by shaking the distillate with charcoal. H. E. F. NOTTON.

Oxidation of toluene to benzaldehyde with pyrolusite. F. S. SHENDEROVICH and S. S. LIVSHITS (J. Chem. Ind., Russia, 1929, 6, 1439—1440).—The optimal concentration of sulphuric acid is 60%. Rise of temperature favours the formation of tar, whilst below 35° the reaction is slow and more benzoic acid is formed. An excess of pyrolusite may cause the formation of tar during distillation with steam. Prolongation of the reaction tends to increase the relative quantities of benzoic acid and of tar. CHEMICAL ABSTRACTS.

Nitration of naphthalene to 1-nitronaphthalene. T. G. ALEKSANDROV and L. K. SHTAMM (J. Chem. Ind., Russia, 1929, 6, 1275—1278, 1522—1524).—The optimal excess of nitric acid is 3%, the yield being 98.96%. The quality of the naphthalene is not important provided that the m.p. is not lower than 79.3°. Utilisation of spent sulphuric acid depresses the m.p. of the product by 0.2°. The presence of a small quantity of nitrous acid is advantageous. Washing the reaction product with 1% sodium carbonate solution removes 2:4-dinitro- α -naphthol (1%). CHEMICAL ABSTRACTS.

Benzol recovery. GOLDSCHMIDT.—See II. **Optical properties of semicarbazones of aldehydes and ketones.** WILSON and KEENAN.—See XIX.

See also A., Sept., 1185, Anthracene derivatives (BARNETT and others). 1186, Friedel-Crafts reaction in the diphenyl series (GROGGINS).

PATENTS.

Manufacture of acetaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,635, 22.4.29).—Acetylene and steam are led at 350—400° over an alumina catalyst activated by addition of dehydrogenation catalysts, e.g., nickel oxide, copper oxide or sulphide, zinc oxide or sulphide, cobalt selenide, zinc phosphate, cadmium tungstate, a mixture of oxides of zinc and tungsten, of cadmium and molybdenum, etc.

C. HOLLINS.

Manufacture of oxygenated organic compounds. L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, Assees. of SOC. CHIM. DE LA GRANDE PAROISSE AZOTE & PROD. CHIM. (B.P. 306,512, 21.2.29. Fr., 22.2.28).—To a stoichiometric mixture of hydrogen and oxides of carbon excess of one of the gases, or an inert gas (or both), is added, and after carrying out the catalytic reaction and removing the products the whole exit gas is used for dilution of a fresh lot of stoichiometric mixture. If the initial diluent gas is nitrogen this is supplied by passing the stoichiometric mixture through an air burner; if hydrogen, the mixture is passed with steam over a suitable catalyst.

C. HOLLINS.

Manufacture of nitrogenous condensation products from acetylene and ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,258, 18.3. and 2.11.29. Cf. B.P. 295,276; B., 1930, 50).—In the process of the prior patent the yield of nitriles is increased at the expense of the pyridines by reducing the gas velocity to about 10 litres per hr. per litre of catalyst at 400°. The catalyst may be a compound of a metal of groups II to VII or a mixture, other than hydrated silicates alone.

C. HOLLINS.

Manufacture of condensation products containing nitrogen [from acetylene etc. and ammonia or amines]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,623, 22.2.29).—Uniform products, containing 75% of the theoretical yield of pyridines and quinolines, are obtained by diluting the reacting gases with steam. The velocity should be at least 10 litres per hr. per litre of catalyst, the temperature 300—500°, and the pressure less than 10 atm.

C. HOLLINS.

Manufacture of [sec. and tert. aliphatic] amines. I. G. FARBENIND. A.-G. (B.P. 332,868, 27.11.29. Ger., 27.11.28).—Primary aliphatic amines are led in vapour form over hydrogenation or dehydrogenation catalysts (copper, cobalt, nickel, platinum) below 300°. Ethylamine, passed over reduced copper on pumice at 260—270° at a rate of 70—75 g. per hr. per litre of catalyst, gives a mixture of 70% of di- and 28% of tri-ethylamine, the conversion being 70—75%. Butylamine similarly yields di- and tri-butylamines.

C. HOLLINS.

Manufacture of amino-alcohols. A. SKITA and F. KEIL (B.P. 313,617, 14.6.29. Ger., 15.6.28).—The catalytic reduction of diketones or hydroxylated ketones with hydrogen in presence of ammonia or amines leads to amino-alcohols; one or both ketonic groups may be

replaced by an aldehyde group. The following amino-alcohols are prepared by reduction in presence of colloidal platinum and gum arabic: β -cyclohexylamino-*n*-propyl alcohol, b.p. 116—117°/13 mm., from hydroxyacetone; δ -cyclohexylaminopentan- β -ol, b.p. 123—125°/13 mm., from acetylacetone; cyclohexylarabinamine (hydrochloride, m.p. 135—137°), from arabinose; cyclohexylmannamine (hydrochloride, m.p. 165—167°); β -methylamino- α - β -diphenylethyl alcohol, m.p. 135°, b.p. 196—199°/20 mm. (hydrochloride, m.p. 250°), from benzil; δ -dimethylaminopentan- β -ol, b.p. 61—63°/12 mm. (hydrochloride, m.p. 115—116°) and δ -(β -hydroxyethylamino)pentan- β -ol, b.p. 143—144°/12 mm., from acetylacetone; γ -hydroxy-*n*-butylaniline, m.p. 59—60°, b.p. 158—160°/8 mm., from aldol; β -methylamino- α - β -dianisylethyl alcohol (hydrochloride, m.p. 210—212°).

C. HOLLINS.

Manufacture of toluene and propylene from cymene. K. SCHÖLLKOPF (U.S.P. 1,747,604, 18.2.30. Appl., 28.2.29. Ger., 12.3.28).—*p*-Cymene vapour is passed over activated bleaching earths (tonsel) at 300—450°, whereby it is decomposed into toluene and propylene. The conversion is about 60%.

C. HOLLINS.

Production of cyclohexenealdehydes, and cyclohexenecarboxylic acids, which contain at least one dialkylated ring-carbon atom. SOC. ANON. M. NAEF & CIE. (B.P. 309,911, 11.4.29. Switz., 17.4.28).—Butadienes carrying a terminal *gem*-dimethyl grouping are heated for 1—2 days with acraldehyde or acrylic acid or homologues of these under pressure at 150—200°; a mixture of two isomeric aldehyde- or carboxy-cyclohexenes is obtained. The products give perfumes by condensation with acetone. $\beta\delta$ -Dimethyl- $\alpha\gamma$ -pentadiene gives with acraldehyde 4 (and 5)-aldehyde-1:3:3-trimethyl- Δ^1 -cyclohexenes, b.p. 78—79°/12 mm. (mixed semicarbazones, m.p. 197—198°); with acrylic acid the corresponding acids, b.p. 135—140°/12 mm.; with crotonaldehyde 4 (and 5)-aldehyde-1:3:3:5 (and 4)-tetramethylcyclohexenes, b.p. 82—84°/12 mm.; from β -methyl- $\alpha\gamma$ -pentadiene and acraldehyde or crotonaldehyde are obtained 4 (and 5)-aldehyde-3:3-dimethyl- or -3:3:5 (and 4)-trimethyl-cyclohexenes, the mixed isomerides having b.p. 74—75°/12 mm. and 79—81°/12 mm., respectively; similar tetra- and penta-methyl compounds, b.p. 83—85°/12 mm. and 86—88°/12 mm., respectively, result from $\beta\epsilon$ -dimethyl- $\beta\delta$ -hexadiene and acraldehyde or crotonaldehyde.

C. HOLLINS.

Manufacture of ethers. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 332,756, 1.8.29).—An aliphatic alcohol (other than methyl) is heated below 300° with a dehydration catalyst, preferably anhydrous aluminium sulphate, alumina, or thoria. The production of *n*- and *iso*-propyl, *n*- and *iso*-butyl, and ethyl ethers is described. The yields are 8—45%.

C. HOLLINS.

Manufacture of vinyl ethers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,605, 22.3.29).—A vinyl halide is heated under pressure with an alkaline alkoxide or aryloxide in presence of a diluent; or the vinyl halide may be circulated through the heated alkaline alkoxide etc. without pressure. The following vinyl ethers are described: methyl, b.p. 7°; ethyl, b.p. 36°;

butyl, b.p. 92—93°; *o*-tolyl, b.p. 168—170°; *m*-tolyl, b.p. 175—177°; phenyl, b.p. 157—158°; β -naphthyl, m.p. 33°, b.p. 264°. *iso*Propenyl ether, b.p. 62—63°, and β -butenyl ethyl ether, CHMe:CMc:OEt, b.p. 70—72°, are similarly obtained.

C. HOLLINS.

Apparatus for manufacture of esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,267, 18.3.29).—The alcohol (*e.g.*, glycerol) and acid are caused to flow in the liquid state down a tower packed with coke or other large-surface packing under reduced pressure (10—20 mm.). The upper part of the tower is maintained at 160°, the lower parts at 190° and 220—230°, and preferably the lower-boiling reactant is in excess. For the esterification of olive oil acids with glycerol the acids may be introduced at the top of the tower and the glycerol in the second zone.

C. HOLLINS.

Manufacture of diazonium salts of complex metallic fluoric acids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,227, 16.4.29).—Stable soluble diazonium metallofluorides are obtained by interaction of diazo solutions with hydrofluoro-titanic, -aluminic, -stannic, -antimonous, -antimonic, or -zincic acids. They are isolated by strong cooling from concentrated solutions.

C. HOLLINS.

Preparation of sulphur compounds of pyridine. G. KOCHENDOERFER, Assr. to DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (U.S.P. 1,753,658, 8.4.30. Appl., 29.11.27. Ger., 3.12.26).—Halogen or diazo substituents in the pyridine nucleus are replaced by thiol groups by the action of potassium hydrogen sulphide etc. The preparation of 2-thiol-5-cyanopyridine, m.p. 245°, 5-nitro-2-thiopyridine, m.p. 171—172° (decomp.), and the disulphide, m.p. 156°, 5-amino-2-thiopyridine, m.p. 245° (decomp.), and 5-iodo-2-thiopyridine, m.p. 216°, from the chloro- or bromo-compounds, and of 2-hydroxy-5-thiopyridine from diazotised 5-amino-2-hydroxypyridine, is described.

C. HOLLINS.

Manufacture of colloidal solutions and pastes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,557, 22.4.29).—Solid materials are dispersed by incorporation with polymerised butadienes in organic solvents. Examples are: zinc oxide in a 5—15% solution of polymerised butadiene in cyclohexane; zinc white in the polymeride and natural rubber; the barium salt of 6-chloro-*m*-toluidine-4-sulphonic acid \rightarrow β -naphthol in the polymeride in benzene; Helio-Bordeaux BL in the polymeride, with turpentine to give a lacquer; sodium dispersed in the polymeride in an atmosphere of nitrogen. Polymerides of suitably varying viscosities are used, those of low viscosity giving lacquers or enamels which produce hard and resistant coatings.

C. HOLLINS.

Manufacture of cyclic ketones. SCHERING-KAHLBAUM A.-G. (B.P. 310,055, 20.4.29. Ger., 21.4.28).—A mixture of a phenol (1 mol.) and a hexahydrophenol (2 mols.) is heated at 200° in presence of a hydrogenation catalyst (nickel), whereby the phenol is hydrogenated to a cyclohexanone. The production of cyclohexanone from phenol and cyclohexanol, and of cyclohexanone and camphor from phenol and borneol, is described.

C. HOLLINS.

Manufacture of esters and amides of 2:3-aminonaphthoic acid. I. G. FARBENIND. A.-G. (B.P. 315,666, 15.7.29. Ger., 14.7.28).— $\beta\beta$ -Naphthisatoic anhydride, made from 2:3-aminonaphthoic acid by action of carbonyl chloride or chloroformic esters, reacts with alcohols or phenols to give alkyl or aryl esters, or with ammonia or amines (other than purely aliphatic secondary amines) to give amides, of 2:3-aminonaphthoic acid. The amide, m.p. 234–236°, anilide, m.p. 237°, methylanilide, m.p. 165°, benzylamide, m.p. 210°, piperidide, m.p. 157–158°, and methyl, m.p. 104–105°, ethyl (m.p. 117–118°), isopropyl (m.p. 85°), butyl (m.p. 100–101°), isobutyl (m.p. 99–100°), benzyl (m.p. 105–106°), β -hydroxyethyl (m.p. 102–103°), and phenyl (m.p. 135°) esters are described. C. HOLLINS.

Manufacture of isatins. I. G. FARBENIND. A.-G. (B.P. 308,740, 27.3.29. Ger., 27.3.28).—The Stollé synthesis is extended to the use of aryloxamyl halides derived from primary arylamines other than naphthylamines (cf. B.P. 286,358; B., 1928, 327), the condensing agent being aluminium chloride in a diluent such as nitrobenzene, chlorobenzene, etc., at 60–70°. Isatins are thus prepared from *m*-5-xylydine (m.p. 240°), *m*-toluidine, *m*-4-xylydine (m.p. 242°), *p*-xylydine (m.p. 267°), ψ -cumidine (m.p. 275°), *m*-chloroaniline (m.p. 260°), *p*-aminoacetophenone (m.p. 350°), *ar*-tetrahydro- α -naphthylamine (m.p. 232°) and β -naphthylamine (m.p. 178°), *p*-toluidine (m.p. 180°), 4-chloro-*m*-toluidine (m.p. 246°), *o*-4-xylydine, *o*-toluidine (m.p. 267°), 6-chloro-*o*-toluidine (m.p. 245°), and cresidine (m.p. 235–236°). C. HOLLINS.

Manufacture of 5:8-dihalogeno-1:2-benzanthraquinones. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,192, 15.3.29).—Non-sulphonating condensing agents, *e.g.*, phosphorus pentoxide, aluminium chloride, zinc chloride, etc., are used for the cyclisation of 3:6-dichloro-2- α -naphthoylbenzoic acid, preferably in nitrobenzene, to 5:8-dichloro-1:2-benzanthraquinone, m.p. 259–260°. C. HOLLINS.

Concentrating aqueous chloroacetaldehyde solutions. O. ERNST and H. LANGE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,774,507, 2.9.30. Appl., 9.10.28. Ger., 28.10.27).—See B.P. 299,722; B., 1929, 887.

Manufacture of condensation products of the benzanthrone series. G. KALISCHER and H. SCHEYER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,755,879, 22.4.30. Appl., 9.7.27. Ger., 4.9.26).—See B.P. 300,331; B., 1929, 350.

Distillation of glycerin (U.S.P. 1,743,488).—See I. **Acetylene from mixed gases** (B.P. 331,610 and 331,654). **Separation of gases** (B.P. 331,787).—See II. **Apparatus for producing photo-chemical radiations** (B.P. 309,497).—See XI. **Fatty acid esters** (B.P. 334,022).—See XII. **Acetone and butyl alcohol by fermentation** (B.P. 306,138 and U.S.P. 1,744,958).—See XVIII.

IV.—DYESTUFFS.

Azo dyes with a 2:6-dimethoxynaphthalene ring. V. V. SHARVIN and N. V. TUTURIN (J. Chem. Ind., Russia, 1929, 6, 1524–1525).—Monoazo dyes of 2:6-dimethoxy- α -naphthylamine possess no affinity

for cotton, but dye wool well; the disazo dyes dye both. With disazo dyes of 2:6-dimethoxy-1:5-naphthylenediamine as compared with those of 1:5-naphthylenediamine, absorbed light is shifted towards the red end of the spectrum. The dyes are not fast to light.

CHEMICAL ABSTRACTS.

See also A., Sept., 1157, **Vegetable dyes from crocetin and xanthophyll** (KARRER and others).

PATENTS.

Manufacture of [wool] dyes of the anthraquinone series. IMPERIAL CHEM. INDUSTRIES, LTD., A. SHEPHERDSON, and W. W. TATUM (B.P. 331,952, 22.4.29).—In the condensation of 4-halogeno-1-aminoanthraquinone-2-sulphonic acids with a *p*-diamine, improved yield and purity result when copper oxide is used in place of copper or copper salts. C. HOLLINS.

Manufacture of compounds from vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,842, 9.3.29).—Vat dyes (or their vatable intermediates) are reduced to leuco-compounds and etherified by treatment with alkali aryloxides; preferably reduction and etherification are combined in one operation. Excess of phenol, or other organic medium, may be used as diluent. The aryl ethers regenerate the vat dyes by hydrolysis or acid oxidation. Pyranthrone gives a diphenyl ether, m.p. about 380°, and a monophenyl ether which is a greenish-brown vat dye. Diphenyl ethers from flavanthrone, dibenzanthrone, and 2:2'-dimethoxydibenzanthrone (m.p. about 305°) are also described. C. HOLLINS.

Manufacture of vat dyes of the dibenzpyrenequinone series. I. G. FARBENIND. A.-G. (B.P. 332,579, 23.3.29. Addn. to B.P. 308,617; B., 1930, 897).—Of the halogenated dibenzpyrenequinones used in the process of the prior patent, dibrominated 1:2:6:7-dibenzpyrene-3:8-quinone gives uniformly useful vat dyes, *e.g.*, with 1-aminoanthraquinone (corinth; heated with aluminium chloride, brown), 1-amino-2-aldehydoanthraquinone (red-brown), or 1-amino-5-benzamidoanthraquinone (corinth). C. HOLLINS.

Manufacture of azo dyes [for viscose silk]. IMPERIAL CHEM. INDUSTRIES, LTD., R. BRIGHTMAN, and W. L. B. WELLACOTT (B.P. 331,839, 5.3.29).—A 3:3'-diaminodiphenylcarbamide, free from hydroxyl, carbonyl, and sulphonic groups, is tetrazotised and coupled with 1 mol. of a phenol- or naphthol-carboxylic or sulphonic acid and 1 mol. of γ -acid, an *N*-substituted γ -acid, or a 1:8-dihydroxynaphthalenesulphonic acid. The same dyes are obtained from suitable *m*-nitroanilines or *m*-aminoacetanilides by separately coupling with the prescribed components, reducing or hydrolysing, and phosgenating an equimolecular mixture of the resulting monoazo dyes. Examples are: 3:3'-diaminodiphenylcarbamide with salicylic acid and γ -acid or phenyl- γ -acid (brown); *m*-nitroaniline \rightarrow salicylic acid, reduced, and phosgenated with the reduction product from *m*-nitroaniline \rightarrow chromotropic acid (bluish-red).

C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,208, 15.1.29).—2:3-Hydroxynaphthoic or

acetoacetic arylamides, or hydroxynaphthacarbazoles, are coupled in substance or on the fibre with a diazotised *o*-aminobenzophenone. Examples are: *o*-aminobenzophenone \rightarrow acetoacetic tolidide (greenish-yellow), 2:3-hydroxynaphthoic α -naphthylamide (red) or anilide (red); 2-amino-4'-methylbenzophenone \rightarrow acetoacetic tolidide (yellow); 4-chloro-2-aminobenzophenone \rightarrow 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide (scarlet). The dyes are fast to bucking with 0.35% sodium hydroxide for 4 hrs. at 1.5 atm. [Stat. ref.]

C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 332,319, 3.5.30).—A diazo, tetrazo, or diazoazo compound is coupled in substance or on the fibre with a 2:3-hydroxynaphthoyl-*p*-aminodiphenylamine. The dianisidine couplings especially give, when coppered, blue shades fast to light. Other examples are: *p*-aminodiphenylamine compound with diazotised 6-chloro-*o*-toluidine (brown-red), or *p*-nitroaniline \rightarrow 2:5-dimethoxyaniline (black); 4-amino-2'-methoxydiphenylamine compound with diazotised 5-nitro-*o*-anisidine (red-brown) or *o*-chloro-*p*-nitroaniline (brown); 3':4'-dichloro-4-aminodiphenylamine compound with diazotised 5-nitro-*o*-toluidine (bluish-red); 4'-nitro-4-aminodiphenylamine compound with diazotised 2:5-dichloroaniline (red-brown), α -aminoanthraquinone (red-brown), etc.

C. HOLLINS.

Manufacture of [dis]azo dyes [for viscose silk] and their application. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 332,954, 29.4.29).—A diamine of the type, $X[NH \cdot C_6H_4 \cdot NH_2(p)]_2$, where X represents a chain of 2 or more methylene, or methylene and carbonyl, groups, is tetrazotised and coupled with coupling components chosen so that the resulting dye contains at least 2 carbonyl or sulphonic groups. The alternative route from a nitroamine or monoacylated diamine of similar type is also described. Examples are: *pp'*-diamino-*NN'*-diphenylethylenediamine with salicylic acid and phenyl- γ -acid (brown), or with 2S-acid and β -naphthol (blue); *N*-(*p*-nitrophenyl)glycyl-*p*-phenylenediamine \rightarrow N.W. acid, reduced, \rightarrow S-acid (blue); *N*-(5-chloro-4-amino-2-methoxyphenyl)glycyl-5-nitro-*o*-anisidine, m.p. 205–206°, \rightarrow F-acid, reduced, \rightarrow H-acid (blue).

C. HOLLINS.

Production of diazo solutions. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 332,630, 27.3.29).—The solubility of diazonium fluoroborates is improved by addition of salts of metals of group I or III which, by double decomposition, give a more soluble diazonium salt; if necessary, the solution of the mixture may be filtered. Suitable salts are potassium chloride, aluminium chloride, alum, sodium chloride, ammonium chloride, potassium sulphate. Sodium sulphate etc. may be present as diluent.

C. HOLLINS.

Manufacture of water-soluble derivatives of aromatic diazo compounds. I. G. FARBENIND. A.-G. (B.P. 309,610, 13.4.29. Ger., 14.4.28).—Aromatic diazo compounds react with the aldehyde-bisulphite derivatives of ammonia or amines to yield soluble, stable diazoamino-compounds, $Ar \cdot N \cdot N \cdot NR \cdot CHR' \cdot O \cdot SO_2Na$, which regenerate diazo salts by the action of mineral

acids. Diazotised 4-chloro- or 5-nitro-*o*-toluidine is coupled with the formaldehyde-bisulphite compound of methylamine, ammonia, or dimethylamine. The products are for use in dyeing and for plant protection.

C. HOLLINS.

Manufacture of [metal compounds of trisazo] dyes. SOC. CHEM. IND. IN BASLE (B.P. 307,705, 11.3.29. Switz., 10.3.28).—The azo dye formed by acid coupling of diazotised J-acid with J-acid is coupled with 2 mols. (the same or different) of an *o*-amino-phenol or -naphthol, and the resulting trisazo dye is treated with chromium or chromium and copper solutions. Examples of *o*-aminophenol components are: 4-chloro-*o*-aminophenol (2 mols.), chromed, or chromed and coppered (grey on cotton and viscose); picramic acid (2 mols.), chromed (green-grey); 4-chloro-*o*-aminophenol and picramic acid, chromed, or chromed and coppered (grey). [Stat. ref.]

C. HOLLINS.

Manufacture of dyes of the triphenylmethane series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,560, 22.4.29).—The amines obtained by the action of an α - or β -phenylethyl halide on primary or secondary arylamines are condensed with aromatic aldehydes, ketones, or hydrols, or with formaldehyde and a tertiary arylamine, the products being oxidised, if necessary, to give triphenylmethane dyes. The phenylethylarylamines may be sulphonated before condensation. Examples are: Michler's ketone with α -phenyldiethylaniline, $CHPhMe \cdot NEtPh$, b.p. 174–178°/20 mm. (violet); *o*-chlorobenzaldehyde with α -phenylethyl-*o*-toluidinesulphonic acid, oxidised (violet); formaldehyde with sulphonated β -phenyldiethylaniline and diethylaniline, oxidised (bluish-violet).

C. HOLLINS.

Manufacture of basic dyes [of the rhodamine series]. IMPERIAL CHEM. INDUSTRIES, LTD., and M. WYLER (B.P. 333,016, 27.6.29).—2-Amino-*m*-4-xyleneol or its acetyl derivative (the acetyl group being removed during condensation) is condensed with 4-mono- or -di-alkylamino-2-hydroxybenzoyl-*o*-benzoic acid, and the free carbonyl group in the product is esterified to give a bluish-red basic rhodamine.

C. HOLLINS.

Liquid dyeing, colouring, and tinting composition. A. DAVID, Assr. to DIP-IT, INC. (U.S.P. 1,747,861, 18.2.30. Appl., 25.3.22).—An organic dye is suspended or dissolved in glycerin without the addition of soap or other thickeners or volatile solvents. The product is intended for use in collapsible tubes etc.

C. HOLLINS.

[Thioindigo] dyes. K. SCHIRMACHER and K. EISHOLD, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,755,667, 22.4.30. Appl., 22.5.28. Ger., 30.5.27).—See B.P. 291,361; B., 1929, 973.

Manufacture of azo dyes. L. LASKA and A. ZITSCHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,746,659, 11.2.30. Appl., 5.10.27. Ger., 15.10.26).—See B.P. 302,773; B., 1929, 165.

[Solubilised *o*-hydroxy-]azo dyes. H. SCHWEITZER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,775,605, 9.9.30. Appl., 16.12.27. Ger., 13.12.26).—See B.P. 282,107; B., 1929, 552.

Manufacture of [acid azo] dyes containing chromium. F. STRAUB, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,775,477, 9.9.30. Appl., 17.9.28. Switz., 23.9.27).—See B.P. 297,478; B., 1929, 890.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of the quality of wood for pulp and cellulose. G. G. KLEM (Papier-Fabr., 1930, 28, 489—494, 501—508, 521—524).—From the examination of a large number of spruce trees and logs it is concluded that the degree of taper may be used as a measure of the quality of the wood, since, within wide limits, the density of the wood and the length of the fibres increase with decreasing magnitude of the decrease in diameter per unit length, whilst the number of knots decreases.

R. K. CALLOW.

Chemical investigation of anaerobically decomposed wood. W. FUCHS (Brennstoff-Chem., 1930, 11, 352—354).—Determinations of the lignin and cellulose contents of bitumen-free specimens of such woods show that the lignin content has increased at the expense of the cellulose (cf. B., 1930, 540). The lignin-cellulose ratio changes during the decomposition from 0.5 to 3.5—9.

H. BURTON.

Peanut-hull cellulose. D. F. J. LYNCH and M. J. Goss (Ind. Eng. Chem., 1930, 22, 903—907).—Peanut hulls have a high pentosan content which may be readily hydrolysed to xylose. The yield, however, is only 7.6%, as compared with 26.5% for oat husks etc. As a source of cellulose the ash content was found to be high (4.3—4.6%), but this could be reduced to 1.65—1.9% by sifting, the cellulose content of the dry material being 43.5—45.0%. The best conditions as to time, temperature, pressure, and concentration for the extraction of the fibre by the soda, the sulphate, and the neutral sulphite methods were determined. By the sulphite method at concentrations above 20% yields of 42% of readily bleachable pulp were obtained. By the other methods the yields were 2% lower and the pulp was harder to bleach. The chlorine required for the bleaching is about 20% of the dry weight of pulp, and the process entails a loss of about 10% of pulp. The best conditions secure a yield of 36—38% of white pulp containing 75—78% of α -cellulose. By treating the pulp with dilute hydrochloric acid (0.5%) and dilute caustic soda (0.25%), alternately, with washing between each treatment, the ash content can be lowered to 0.3%. β - and γ -Celluloses can be removed by treatment with caustic soda (5—7% with heating or 8—9% cold) so as to leave, after washing and drying, a pulp containing 93% of α -cellulose.

H. J. DOWDEN.

Action of neutral salts and dilute acids on sulphite pulp. C. KULLGREN (Svensk Kem. Tidskr., 1930, 42, 179—193).—Sulphite pulp in contact with *N*-sodium chloride imparts a distinct acidity to the latter. The moist fibre from this treatment, after washing, absorbs from *N*-hydrochloric acid an amount of acid roughly equal to that developed in the former process. After washing, the whole cycle of operations may be repeated with the same sample of pulp; the development of acidity and absorption of acid are identical in amount with those previously found. This

phenomenon is explained as an ionic exchange between the liquid and the solid ligninsulphonic acid phases. In various samples of pulp of widely-differing sulphur content, 1 equivalent of acid formed \equiv 1.2—1.5 atoms of sulphur. The amount of acid absorbed from the hydrochloric acid solution is always less than that formed during the treatment with sodium chloride solution. This is due to hydrolysis and partial neutralisation of the salt by the carbon dioxide of the wash-water. Replacement of hydrochloric acid and sodium chloride by nitric acid and sodium nitrate leads to identical results. The ash content of the fibre varies in a manner to be expected from the ionic exchange. The material, after treatment with sodium chloride, may be dried at 100—105° without alteration in properties. When, however, the pretreatment has been with hydrochloric acid, and the free acid removed by washing with water, the material on drying becomes grey or black, and the individual fibres lose their strength. A comparison is made between the pulp produced by solutions containing 5% SO_2 and 5% $\text{SO}_2 + 4\% \text{Na}_2\text{SO}_4$ as reagents. The latter gave a fibre of good appearance at 135°, whilst the former, even at 110°, yielded a dark-coloured product and weakened fibres. In addition, the ash content of the product of the second process is notably higher than that of the first, the sulphur and sodium being present in about equal atomic proportions. In this process sodium chloride may not successfully replace sodium sulphate. The calcium found in certain sulphite pulps is present to a large extent as a salt of ligninsulphonic acid.

T. H. MORTON.

Behaviour of softening agents towards nitrocellulose. H. WOLFF and B. ROSEN (Farben-Ztg., 1930, 35, 2473—2474).—The swelling and solvation effects of softeners on nitrocelluloses of three different manufactures (Wasag, Hercules, Eilenburg), each in three degrees of viscosity, is investigated. The final effect is reached in 1 hr. with butyl phthalate, or in 7 days with tritoyl phosphate or sipalin, and appears to be dependent on the origin of the nitrocellulose and not on its viscosity, i.e., different nitrocelluloses show variations not reflected in viscosity measurements.

C. HOLLINS.

Sedimentation of causticiser sludge [in alkali recovery] in the sulphate-cellulose process. B. WIESLER (Papier-Fabr., 1930, 28, 559—560).—The slow sedimentation which occurs after sodium carbonate has been causticised by the addition of lime is principally due to the presence of iron sulphide. Laboratory and plant experiments show that iron sulphide settles rapidly in large floccules from a hot liquid, but when the cold liquid is stirred a fine suspension is obtained which settles slowly. Sedimentation is therefore best allowed to take place at as high a temperature as possible.

R. K. CALLOW.

Structure of celluloid and gelatinising agents of nitrocellulose as swelling agents. I. Change in optical anisotropy of camphor-celluloid containing varying amounts of camphor on stretching. J. C. DERKSEN, J. R. KATZ, K. HESS, and C. TROGUS (Z. physikal. Chem., 1930, 149, 371—381).—The specific double refraction of celluloid which has been stretched so as to retain a permanent elongation has been measured.

With celluloid containing 5–30% of camphor the double refraction is positive and increases continuously with increase in the permanent elongation, with 40% of camphor present it is first positive and then becomes negative as the elongation increases, and with 50% of camphor it is negative throughout. In many cases the value tends to a limit as the extension is increased, and if the limiting value is plotted against the camphor content the graph consists of two approximately straight lines intersecting at a point corresponding with 37% of camphor, *i.e.*, an equimolecular mixture of the two constituents. In explanation of these observations it is assumed that stretching causes orientation of the micelles in the celluloid, which becomes doubly refracting in consequence. Apparently there are present at least two kinds of elongated micelles, probably those of cellulose dinitrate and those of a compound of cellulose dinitrate and camphor in equimolecular proportions. The former are assumed to have a positive double refraction and to undergo orientation more rapidly than the latter, which have a negative double refraction. Evidence in support of this theory has been obtained from X-ray diagrams.

R. CUTHILL.

See also A., Sept., 1115, **Dependence of viscosity of cellulose solutions on temperature** (BERL and UMSTÄTTER). 1123, **Interaction of sulphurous acid and aldehydes [and the sulphite process]** (HOOVER and others). 1168, **Swelling and acetylation of cellulose** (BERNOUILLI and others). 1169, **Cellulose sulphuric esters** (GEBAUER-FÜLNEGG and DINGLER).

PATENTS.

Cellulosic compositions. BRIT. CELANESE, LTD. (B.P. 312,688, 10.5.29. U.S., 31.5.28.).—Plastic or celluloid-like compositions or masses, moulding powders, films, dopes, lacquers, artificial fibres, etc. having a basis of cellulose acetate are manufactured by incorporating, at any convenient stage and with or without the use of volatile solvents, one or more esters of aromatic sulphonic acids of the formula $R \cdot SO_2 \cdot OR'$, where R is an aryl radical and R' an alkyl or aryl hydrocarbon group containing at least two carbon atoms. F. R. ENNOS.

Alkylation of cellulose. E. I. DU PONT DE NEMOURS & Co. (B.P. 311,697, 8.3.29. U.S., 14.5.28.).—After grinding, and before alkylation, the cellulose is mixed with a slurry of alkali in an inert or non-etherifiable liquid (benzene). F. R. ENNOS.

Manufacture of acetylcellulose in the form of bands or threads. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 313,974, 20.6.29. Ger., 20.6.28.).—The primary solution of cellulose acetate, obtained as described in B.P. 291,001 and 325,209 (B., 1929, 750; 1930, 414), is squirted into a precipitating bath. H. ROYAL-DAWSON.

Manufacture of artificial silk. RÖHM & HAAS A.-G., and R. O. HERZOG (B.P. 311,784, 16.5.29. Ger., 18.5.28.).—One or more cellulose compounds (acetylcellulose) and one or more completely or partly polymerised products of unsaturated organic compounds, (esters of acrylic acid) are separately dissolved in non-miscible solvents, and the mixture of solutions so

obtained is spun into threads, polymerisation of the organic compound being completed where necessary.

F. R. ENNOS.

Manufacture of artificial filaments and the like. BRIT. CELANESE, LTD. (B.P. 318,631, 5.9.29. U.S., 8.9.28.).—Solutions of cellulose or of cellulose derivatives are extruded through orifices of compact or squat shape, having indentations in the form of re-entrant angles, into a precipitating medium or evaporative atmosphere, or through a cluster of circular holes which are so close together that the filaments after extrusion coalesce to form a single filament. F. R. ENNOS.

Production of artificial leather [from cellulose esters or ethers]. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS (B.P. 317,824, 9.8.29. Ger., 23.8.28. Addn. to B.P. 301,759; B., 1930, 319).—The basic fibrous material or the tips of its fibres are impregnated superficially with a thin layer of the cellulose ester or with a cellulose ester solvent only, the plastic cellulose ester mass being applied subsequently, in the latter case before all the solvent has evaporated. The upper layer of material may also be previously impregnated with a liquid softening agent, *e.g.*, ethyl phthalate, or with a gelatinising agent. F. R. ENNOS.

Manufacture of [sized] paper. R. HADDAN. FROM RAFFOLD INTERNAT. CORP. (B.P. 333,620, 17.5.29).—The paper is made from fibrous material which is mixed with a saponaceous paraffin emulsion containing rosin size or rosin and montan wax and with a carbonate filler (calcium carbonate, with or without magnesium hydroxide), both of which are mutually flocculated; precipitation of the paraffin emulsion may also be brought about by addition of alum. F. R. ENNOS.

Extraction of fat from raw wool. A. ENGELHARDT, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,775,590, 9.9.30. Appl., 12.9.27. Ger., 21.9.26).—See B.P. 300,941; B., 1929, 167.

Production of stiff fabrics which withstand washing. H. MÜLLER, Assr. to A.-G. CILANDER (U.S.P. 1,775,319, 9.9.30. Appl., 25.2.26. Ger., 21.1.26).—See B.P. 264,783; B., 1927, 406.

Manufacture of high-grade viscose products. A. JÄGER, Assr. to O. KOHORN & Co. (U.S.P. 1,774,712, 2.9.30. Appl., 24.10.28. Ger., 26.11.27).—See B.P. 301,305; B., 1929, 893.

Scraper blade for detaching threads of cellulosic material from the winding-up drum of a spinning machine. M. DASSONVILLE (B.P. 334,086, 26.9.29. Fr., 31.1.29. Addn. to B.P. 330,753).

Cleaning the nozzles in artificial silk spinning machines. O. VON KOHORN and H. SCHUPP (B.P. 334,109, 21.10.29. Ger., 15.11.28).

Manufacture of [cellulose ester] films [for tipping cigarettes]. C. MÜNCH (B.P. 334,271, 4.4.29).

Manufacture of abrasive paper. CARBORUNDUM Co., LTD., Assees. of H. C. MARTIN (B.P. 316,308, 18.7.29. U.S., 28.7.28).

Troughs for mixing etc. (B.P. 333,993).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Evaluation of textile impregnating agents. M. RIQUELME (Anal. Fís. Quím., 1930, 28, 784—791).—Determination of the capillary constants of an impregnating agent by the usual methods is not essential; it is more satisfactory to determine the rate of flow through a small orifice. H. F. GILLBE.

PATENTS.

Wetting agents. H. T. BÖRME A.-G. (B.P. 313,160, 23.5.29. Ger., 9.6.28).—Sulphonated fatty acids produced from hydroxylated or unsaturated acids are esterified, or the original acids are sulphonated and esterified in one operation by means of alkyl hydrogen sulphates or alcohol and sulphuric acid. The esters are used as wetting agents in textile operations.

C. HOLLINS.

Immunisation of vegetable fibres to direct dyes. SOC. CHEM. IND. IN BASLE (B.P. 315,435, 13.7.29. Switz., 13.7.28).—Cotton is first impregnated with crude copper sulphate solution, then dried, and acetylated with acetic anhydride at 100°. Immunisation is complete in less than 1 hr.

C. HOLLINS.

Dyeing with vat colours. F. L. REMELIN, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,750,942, 18.3.30. Appl., 26.6.28).—A leuco-vat dye solution is padded on the fibre, which is steamed by evaporation of the water in the padded fibre, e.g., by running the wet goods directly on hot rollers before oxidation.

C. HOLLINS.

Development of ester salts of leuco-compounds of vat dyes. I. G. FARBENIND. A.-G. (B.P. 310,803, 29.4.29. Ger., 30.4.28).—The dyeings are steamed in presence of a chloride, ester, or anhydride of a strong organic acid, especially of a sulphonic acid, e.g., α -naphthalenesulphonyl chloride.

C. HOLLINS.

Production of colour effects on artificial materials made from or containing cellulose esters. BRIT. CELANESE, LTD. (B.P. 314,446, 27.6.29. U.S., 27.6.28).—Cellulose ester material is superficially hydrolysed and dyed with dischargeable dyes, a white or colour discharge paste being then applied locally, e.g., by printing.

C. HOLLINS.

Dyeing of regenerated cellulose materials. H. JORDAN, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,755,119, 15.4.30. Appl., 11.9.28).—Level blue to violet shades on viscose silk are obtained with secondary disazo dyes of the type 4-acetamido-Cleve acid \rightarrow non-phenolic middle component \rightarrow J-acid or an *N*-substituted J-acid, the acetyl group being finally removed by hydrolysis. In the examples J-acid or phenyl-J-acid is the end component, the middle component being Cleve acid, *m*-toluidine, cresidine, α -naphthylamine, or 2-ethoxy-Cleve acid.

C. HOLLINS.

Dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 331,822 and 332,545, [A] 5.4.29, [B] 19.4.29).—Even shades are obtained on viscose silk by application of (A) disazo dyes prepared by coupling a tetrazotised 4:4'-diaminodiphenylcarbamide-mono- or -di-sulphonic

acid with 1 mol. of a carboxylated or sulphonated phenol, naphthol, naphthylamine, or *N*-substituted naphthylamine, and 1 mol. of the same component or of an aminonaphtholsulphonic acid or an *N*-derivative thereof. The dyes may be also produced by phosgenation of suitable mixtures of monoazo dyes. Amongst the examples are: 4:4'-diaminodiphenylcarbamide-3:3'-disulphonic acid with salicylic acid and γ -acid (bluish-red), or phenyl- γ -acid (claret); 2-chloro-4:4'-diamino-5-methoxydiphenylcarbamide-3'-sulphonic acid with *N.W.*-acid and 2*S*-acid (violet). (B) Disazo dyes of the type: non-nitrated amine of the benzene series \rightarrow (alkaline) aminonaphtholsulphonic acid \rightarrow coupling component, may also be used. E.g., *p*-chloroaniline \rightarrow γ -acid \rightarrow γ -acid (violet-brown) or 4-nitro-*m*-phenylenediamine (red); *m*-aminobenzoic acid \rightarrow γ -acid \rightarrow salicylic acid (maroon) or β -naphthol (red-orange); etc.

C. HOLLINS.

[Coloured] discharge printing on dyed fabrics. SOC. CHEM. IND. IN BASLE (B.P. 315,756, 26.4.29. Switz., 17.7.28. Addn. to B.P. 303,179; B., 1930, 506).—Fabric is printed with a discharge paste containing in addition a 4-aroxy- α -naphthol, steamed, and treated with a diazo solution before washing out the reduction products in the usual alkaline stripping bath. E.g., fabric grounded with indigo is printed with a discharge paste containing phenyl 4-hydroxy- α -naphthyl ketone, steamed, developed with diazotised 4-chloro-2-amino-diphenyl ether, and washed.

C. HOLLINS.

Manufacture of improved artificial silk, in particular cellulose acetate silk. I. G. FARBENIND. A.-G. (B.P. 309,558, 12.4.29. Ger., 12.4.28).—Artificial silk in the finished state is treated with acetic acid (about 40%) and stretched to several times its length, the acid being subsequently removed gradually by washing with acid in increasing dilution and finally with water, while maintaining or increasing the tension.

F. R. ENNOS.

Weighting of artificial silk. R. CLAVEL (B.P. 333,724, 30.7.29. Ger., 16.3.29. Addn. to B.P. 277,602; B., 1928, 154).—Satisfactory spotless weightings result when, in modification of the process of the prior patent, an acid fixing bath is employed, whereby washing of the textile material treated with a metal salt is avoided.

A. J. HALL.

Manufacture and treatment [delustring] of materials made or containing organic derivatives of cellulose. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 333,504, 5.2.29).—Delustred cellulose acetate threads are prepared by dry- or wet-spinning solutions of the acetate containing wetting-out agents and afterwards removing the wetting-out agent by treatment with aqueous liquors or steam. Suitable wetting-out agents, such as soaps, sulphonated fatty or aromatic acids, etc., are indicated.

A. J. HALL.

Method of washing [yarns, fabrics, etc.]. A. RAYNER (B.P. 333,177, 3.5.29).—The material is impregnated with a solution containing 1–2% of a soap-forming (fatty) acid and then with an alkaline solution to form a soap *in situ*.

L. A. COLES.

Preparation of leather cloth etc. IMPERIAL CHEM. INDUSTRIES, LTD., and G. E. SCHARFF (B.P. 332,602, 23.1.29).—Leather cloth is sprayed or spread with a self-cracking enamel comprising a cellulose derivative, volatile solvents, and about 3—30 times as much pigment and/or filler as cellulose compound, plasticisers being absent or present in small amount. On drying, the film cracks and may be covered by an impregnating transparent varnish containing plasticiser.

C. HOLLINS.

Dyeing of pelts, hairs, feathers, etc. P. VIRCK, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,775,074, 2.9.30. Appl., 6.12.27. Ger., 14.9.26).—See B.P. 290,126; B., 1928, 521.

Production of fast dyeings and printings. P. VIRCK, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,774,621, 2.9.30. Appl., 6.12.27. Ger., 8.10.26).—See B.P. 306,632; B., 1929, 353.

Discharging of dyed cellulose acetate materials. A. FISCHER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,747,540, 18.2.30. Appl., 16.10.26. Ger., 17.6.26).—See G.P. 461,753; B., 1930, 762.

Apparatus for treating hanks of yarn with liquids. G. S. LORD (B.P. 334,171, 26.3.29).

Apparatus for fluid treatment of wound textile fibres. O. J. OBERMAIER, and OBERMAIER & Co. (B.P. 318,236, 9.7.29. Ger., 31.8.28).

Wet treatment of skein yarns, particularly of artificial silk. E. BLASCHKE (B.P. 334,382, 3.9.29. Ger., 9.8.29).

[Apparatus for] mercerising, bleaching, or like processes. J. A. BOLTON and S. S. HAMMERSLEY (B.P. 333,888, 20.4.29).

Drying and finishing machine for fabrics in long lengths. MOORE FABRIC CO. (B.P. 334,439, 31.10.29. U.S., 19.2.29).

Steaming of fabrics printed with vat dyes. I. G. FARBENIND. A.-G. (B.P. 333,873, 13.4.29. Ger., 3.1.29).

Disazo dyes on viscose silk (B.P. 332,954).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Cheaper ammonia with off-peak electric power. P. McMICAHEL (Chem. Met. Eng., 1939, 37, 484—487).—It is claimed that if off-peak electric power is purchasable at 0.5 cent per unit, the cost of electrolytic hydrogen and oxygen will be about 32 cents per 1000 cub. ft. If a credit is obtained for the oxygen produced both electrolytically and in the production of nitrogen equal to the above production cost, then the over-all cost of synthetic ammonia, using such electrolytic hydrogen, is calculated to be \$60 per ton of NH_3 . This is for an annual output of 26,250 tons NH_3 . It is possible that it might be more economical to produce nitrogen by the combustion of hydrogen in air rather than by liquefaction, the capital cost of this method being much less.

C. IRWIN.

Dissociation of carbon dioxide under the influence of the electric discharge under reduced pressure.

F. FISCHER, H. KÜSTER, and K. PETERS (Brennstoff-Chem., 1930, 11, 300—304; cf. B., 1930, 594).—The degree of dissociation of carbon dioxide ($2\text{CO}_2 \rightleftharpoons 2\text{CO} + \text{O}_2$) caused by subjecting a current of the gas to an electric discharge under pressures of 30—100 mm. Hg has been determined. With the dry gas the maximum degree of dissociation observed was 43%, but with the moist gas it was only 9%, the difference being due to re-combination of the primary dissociation products in the presence of water vapour. The maximum dissociation products occurred at medium pressures (40—60 mm. Hg) and at a rate of passage of 100—200 litres/hr.; it increased with increasing rate of electrical energy input. The theoretical equilibrium temperatures corresponding with the observed degrees of dissociation lie about 1000° higher than the temperatures measured in the discharge tube by means of a thermocouple. The dissociation is therefore only partly thermal in effect, and partly electrical.

A. B. MANNING.

Alkali recovery in the sulphate-cellulose process. WIESLER.—See V. Iron oxides and hydroxides from rusting of iron. SCHIKORR.—See X. Determining cream of tartar and tartaric acid in baking powders. HARTMANN.—See XIX. Oxygen for gas masks. HLOCH.—See XXIII.

See also A., Sept., 1132, **Platinum-free catalyst for production of nitric acid** (ADADUROV and WEIN-SCHENKER). 1135, **Ozone production in silent electric discharge** (BRINER and SUSZ). 1136, **Preparation of pure hydrogen electrolytically** (HULUBEI). 1139, **Synthesis of ammonia** (DUPARC and others). 1143, **Determination of iodides in presence of other halides** (GORBATSCHEV and KASATKINA).

PATENTS.

Manufacture of phosphoric acid and products containing phosphoric acid. AKTIEB. KEMISKA PATENTER, Assees. of KONSTGÖDNINGSFABR. AKTIEB. I LANDSKRONA (B.P. 314,977, 4.4.29. Ger., 7.7.28).—Crude phosphates are treated under pressure in closed vessels with sulphuric acid diluted with phosphoric acid containing at least 15% P_2O_5 , the phosphates preferably being worked up with the phosphoric acid, which may contain a little sulphuric acid, to a paste which is run into the sulphuric acid. The temperature of the mixture is raised during at least a part of the process to $(120-p)^\circ$ ($p = \% \text{P}_2\text{O}_5$ in the phosphoric acid used), and the conditions are so regulated that the calcium sulphate formed is deposited as anhydrite or as a mixture of it with semihydrate (cf. B.P. 314,976; B., 1930, 861).

L. A. COLES.

Separation of alkalis from siliceous minerals. P. E. BILLINGHURST (B.P. 333,076, 9.9.29).—The finely-powdered silicate, e.g., felspar or spodumene, is blown through a Ω -tube heated at 1500—1800° by means of a bath of molten metal in an induction furnace, and the vapour of alkali oxides so obtained is condensed in water.

A. R. POWELL.

Manufacture of alkali carbonates. INTERNAT. INDUSTRIAL & CHEM. CO., LTD. (B.P. 333,834, 18.11.29. Fr., 21.11.28).—A solution containing sodium silicate

and hydroxide with barium sulphate in suspension, prepared by the double decomposition of sodium sulphate and barium orthosilicate (Ba_2SiO_4), is carbonated in accordance with the equation: $\text{Na}_2\text{SiO}_3 + 2\text{NaOH} + 2\text{BaSO}_4 + 2\text{CO}_2 = \text{SiO}_2 + 2\text{Na}_2\text{CO}_3 + 2\text{BaSO}_4 + \text{H}_2\text{O}$; after filtration, the solution is evaporated to yield sodium carbonate and the insoluble residue is calcined in the presence or absence of carbon to recover barium orthosilicate, the sulphur dioxide liberated being used, if desired, to prepare sodium sulphate from the chloride.

L. A. COLES.

Decomposition of raw phosphates. A. MESSERSCHMITT (B.P. 333,805, 21.10.29. Ger., 23.10.28. Addn. to B.P. 300,961; B., 1930, 57).—The liberation in the prior process of free sulphur, which can be recovered from the waste gases, is attained by using sufficient carbon for the complete reduction of the alkali sulphate and such quantities of acid components (silica) that the product contains little or no free alkali or alkaline-earth bases.

L. A. COLES.

Decomposition of crude phosphate. CHEMIE-VERFAHREN-GES.M.B.H. (B.P. 333,797, 11.10.29. Ger., 28.11.28).—Recovery of potassium sulphate from mixed potassium calcium sulphates precipitated in the treatment of crude phosphates with potassium sulphate and, e.g., nitric acid is facilitated by agitating the precipitated material with a dilute aqueous solution of the acid used in the decomposition as an intermediate step between two washings with water.

L. A. COLES.

Production of non-caustic calcium cyanamide which is free from dust. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 333,353, 1.7.29 and 13.3.30).—Free lime present in crude calcium cyanamide is neutralised by the separate or successive treatment of the material with solid phosphoric acid and ammonium nitrate in the absence of water and at a temperature not above 150° .

L. A. COLES.

Preparation of anhydrous magnesium chloride practically free from oxide. I. G. FARBENIND. A.-G. (B.P. 333,741, 16.8.29. Ger., 4.10.28).—A mixture of magnesite with wood or peat charcoal, both in lumps the size of nuts, supported on a pervious bed of lumps of electrode carbon, is treated, e.g., in a shaft furnace, with an ascending stream of chlorine introduced at the bottom of the mixture. The material is heated initially to reaction temperature with the aid of electrodes situated below the bed of carbon and dipping into the mixture, respectively, and the temperature is subsequently maintained by the heat of reaction.

L. A. COLES.

Preparation of base-exchanging filter materials. S. A. KRÜGER (B.P. 333,844, 5.12.29. Swed., 5.1.29).—Reactive, non-friable products are obtained by calcining base-exchanging clays etc. under non-oxidising conditions, e.g., in the presence of neutral or reducing gases or in closed chambers.

L. A. COLES.

Treatment of filtering materials [e.g., clays]. J. V. APABLASA, ASSR. to O. J. SALISBURY (U.S.P. 1,742,433, 7.1.30. Appl., 21.12.23).—Clays etc. for filtering and bleaching purposes are treated while in a dry and finely-divided state with a solution containing the reaction products of sulphuric acid, sodium silicate,

sodium bisulphite, and water, the quantity of solution applied being insufficient to cause fluidity of the material.

B. M. VENABLES.

Compound of cobalt cyanate with a tertiary base. T. L. DAVIS, ASSR. to W. O. SNELLING (U.S.P. 1,750,160, 11.3.30. Appl., 16.4.28).—An aqueous solution of cobalt chloride (1 mol.) is mixed with an aqueous solution of potassium cyanate (2 mols.), and to the mixture is added a tertiary base (pyridine and quinoline). The precipitate is extracted with chloroform and allowed to crystallise. Such compounds are useful in the preparation of temperature-recording solutions.

C. HOLLINS.

Carbon dioxide recorder. A. B. CUNNINGHAM, ASSR. to REPUBLIC FLOW METERS Co. (U.S.P. 1,751,265, 18.3.30. Appl., 16.9.21).—The apparatus comprises an aspirator device for periodically withdrawing a fixed quantity of gas from a gas stream, means for passing this gas through potassium hydroxide solution and measuring the volume of unabsorbed gas, and means for recording this volume on a time chart.

A. R. POWELL.

Manufacture of sodium sulphide drops. F. MEYER, ASSR. to CHEM. FABR. KUNHEIM & Co. (U.S.P. 1,774,560, 2.9.30. Appl., 25.3.25. Ger., 1.5.24).—See B.P. 233,316; B., 1926, 439.

Preparation of metal bromides. J. H. VAN DER MEULEN (U.S.P. 1,775,598, 9.9.30. Appl., 29.12.27. Ger., 25.2.27).—See B.P. 285,915; B., 1928, 815.

Production of aluminium oxide. W. W. TRIGGS. FROM NORTON Co. (B.P. 332,902, 27.4.29).—See U.S.P. 1,719,131; B., 1930, 665.

Crystalliser (B.P. 333,598). Separation of gases (B.P. 308,792 and 333,907).—See I. Arsenical fume (U.S.P. 1,755,985).—See X. Electrolysis of water (U.S.P. 1,757,235).—See XI. Fertilisers (B.P. 333,477).—See XVI. Antiseptic liquid (B.P. 332,520).—See XX.

VIII.—GLASS; CERAMICS.

Chemical and physico-chemical reactions in the grinding and polishing of glass. F. W. PRESTON (J. Soc. Glass Tech., 1930, 14, 127—132 T).—In the grinding of emery before use, large quantities of hydrogen were generated by the action of the water on the fine particles of steel ground away from the drum, and the gas interfered seriously with the subsequent grading process. A similar reaction occurred when grinding glass with iron tools. In the polishing operation the glass removed reacted with the ferrous sulphate in the rouge to produce sodium sulphate, iron hydroxide, and gelatinous silica, the last-named providing the principal bond in "rouge cake," analysis of which is given. (Cf. following abstract.)

M. PARKIN.

Nature of "rough cake" [from polishing of glass]. W. M. HAMPTON (J. Soc. Glass Tech., 1930, 14, 133—137 T).—An analysis of rouge cake gave (%): Na_2SiO_3 1.18, Na_2SO_4 1.03, CaSO_4 14.56, Ca(OH)_2 0.92, SiO_2 (hydrated) 10.08, FeO 1.54, Fe_2O_3 5.45, $\text{Fe}_2(\text{OH})_6$ 25.70, SiO_2 6.96, Al_2O_3 0.10, CaO 0.30, Na_2O 0.16, organic matter (felt) 1.44, hygroscopic water 31.15. (Cf. preceding abstract.)

M. PARKIN.

Thermal endurance of glass. C. E. GOULD and W. M. HAMPTON (*J. Soc. Glass Tech.*, 1930, 14, 188—204 r).—The temperature difference, θ , necessary to cause fracture by heating paraffin wax in a beaker to a given temperature and then plunging into cold water, repeating with 5° rises in temperature until fracture took place, was related to the (maximum) wall thickness, $2b$, of the beaker at the fracture by the equation $\theta \sqrt{2b} = \text{a constant} = B$. An alternative test, using glass rods, was found useful where only small amounts of the glass in question was available, but was not to be recommended. It is shown mathematically that when the thickness is great, the temperature drop to cause fracture becomes independent of the thickness; an expression has also been derived enabling the thermal endurance figure to be calculated from the composition of the glass. M. PARKIN.

Changes in thermal expansion characteristics during the burning of a silica brick. J. A. SUGDEN and J. W. COBB (*Trans. Ceram. Soc.*, 1930, 29, 217—230).—Test pieces burned in a works' kiln were withdrawn at intervals and cooled quickly in order to measure the thermal expansion. Results show it to be impossible to calculate reliable figures for the quartz, tridymite, and cristobalite composition from the expansion curve. The good qualities of a well-fired silica material lie in some property other than that of thermal expansion; presumably they are due to accompanying changes in the bond, and in the structural relation of the bond to the crystalline aggregate. R. J. CARTLIDGE.

Plasticity of clay. I. Mechanical methods of measurement. S. R. HIND. **II. Effect of non-plastic additions on mechanical properties of a plastic clay.** S. R. HIND and E. P. DEGG (*Trans. Ceram. Soc.*, 1930, 29, 177—207, 208—216).—I. "Plasticity" is defined, and the literature on the mechanical properties of plastic clay is reviewed. Experiments on clay subjected to axial loading, intermittent loading in tension, and a steadily increasing load were conducted, using a machine which is described. The strains produced in plastic distortion result in shear at about 45° to the direction of applied force. Data for china clays, ball clays, Stourbridge clay, and a ball clay-rape oil mixture are given. A study of the stress-strain curves yields a softness factor peculiar to each test specimen, and a further simple relation was found to exist for all the stress-strain diagrams, dependent on the water content, extension under load, constant for hand-working, and increase in softness due to increase in water content.

II. Experiments are described in which mixtures containing up to 60% of grog were used, and the effects of grain size with 30% additions of non-plastics were also noted. The workability and minimum water content for plasticity decrease regularly with increasing grog content; the former tends to be proportional to the clay content when using fine grog, but is much less when coarse material is used. R. J. CARTLIDGE.

Experiments with Indian sillimanite. J. H. CHESTERS (*Trans. Ceram. Soc.*, 1930, 29, 305—309).—Four series of experiments are described in which the sillimanite used was ground in a ball mill with ball clay, ball clay and felspar, calcined alumina and raw

cyanite, and the mixtures produced were fired at different temperatures. Values for the drying shrinkage, burning shrinkage, and resistance to permeability of these mixtures are tabulated. R. J. CARTLIDGE.

Manufacture of felspathic or hard porcelain from British raw materials. B. MOORE and J. W. MELLOR (*Trans. Ceram. Soc.*, 1930, 29, 258—274).—In the opinion of the authors, the felspar required for hard porcelain can be obtained by the use of Cornish stone. A study was made of adapting a body to suit working conditions, using British materials. The bodies developed are comparable with earthenware, so that they may be thrown and turned, jolleyed, pressed, or cast. After firing in the hard-enamel or easy-majolica kiln the biscuit product is very porous and requires careful handling to obtain an even coat of glaze. The glazed ware must be fired in a china biscuit oven. Ternary diagrams showing the relations between composition and certain properties of the bodies are given. Sixty-six ternary mixtures were fired at 1000°, 1100°, 1200°, and 1230°, respectively, and the results are shown graphically. R. J. CARTLIDGE.

Improvements in preparing and moulding refractories, especially for manufacturing large blocks. H. ACKERMANN and H. KNUTH (*Trans. Ceram. Soc.*, 1930, 29, 280—295).—By mixing clay slip with a suitable amount of grog a body of low plasticity may be obtained which, when moulded by strong mechanical action, produces a block of exact shape. A comparison of bulk sp. gr., porosity, and crushing strength shows the superiority of the S.u.G. products. This "S.u.G." process has been covered by patents (cf. Scheidhauer & Giessing A.-G., B., 1927, 254, 443, etc.). R. J. CARTLIDGE.

Differences between the interior and exterior portions of fireclay products consequent on the firing operation. A. T. GREEN (*Trans. Ceram. Soc.*, 1930, 29, 296—304).—The variation in sp. gr. and porosity of the interior and exterior portions of samples of Stourbridge and Scottish fireclay materials, taken during vitrification, has been studied. Such variations, depending on the control of firing, may or may not have detrimental influences on the finished product. R. J. CARTLIDGE.

Chemical analysis of refractory materials. III. H. J. VAN ROYEN and H. GREWE (*Arch. Eisenhüttenw.*, 1930—1, 4, 17—21; *Stahl u. Eisen*, 1930, 50, 1229—1230).—A method of analysis for chromite refractories is presented. Experiments were first carried out on synthetic solutions of potash alum, pure iron oxide, potassium dichromate, pure calcium carbonate, and magnesium chloride. Magnesia was precipitated by the cold process, this being preferred to Treadwell's method. Alumina in the alum solution was determined by the ammonia and phosphate method. Aluminium, iron, and chromium were separated by ammonia from the alkaline earths, the chromium was determined by Philip's method, the iron by the Knecht-Hibbert method, and the aluminium by difference. Before being fused with sodium carbonate, the chrome-brick sample must be ground to fine powder. F. SALT.

Preliminary investigation of the magnesia-zircon series. W. J. REES and J. H. CHESTERS (Trans. Ceram. Soc., 1930, 29, 309—316).—The shrinkage which occurs when articles made of magnesia are fired at high temperatures, giving rise to cracking of the article, can be reduced when zircon is added to the magnesia; a mineral the density of which is lower than the mean of its constituents is formed. It is suggested that this mineral is probably a *magnesium zirconium silicate*. R. J. CARTLIDGE.

Lead for stained glass. BROWN.—See X.

See also A., Sept., 1114, **Clearing of clay turbidities** (FOERSTER). 1121, **System sodium oxide-silica** (KRACEK). 1122, **System sodium oxide-silica-zirconia** (D'ANS and LÖFFLER). 1153, **Crucibles for silicate fusions** (SALMANG).

PATENTS.

[Glass-annealing] furnaces. W. A. MORTON (B.P. 333,596, 17.4.29).—A furnace especially suitable for annealing glass is provided with a longitudinal metallic conveyor in the muffle chamber which is heated by radiation from a combustion chamber beneath one end and flues extending therefrom. The radiation from the floor of the muffle chamber to the goods is controlled by adjustable louvres and, at the other end, the cooling of the chamber is regulated by removing more or less insulation from the roof and walls. B. M. VENABLES.

Manufacture of vitreous material. P. B. CROSSLLEY (B.P. 333,240, 8.2.29).—Mixtures the constituents of which have all been disintegrated at least in part to a colloidal condition and comprising mica and asbestos, a relatively infusible material (e.g., porcelain, soda-lime or crystal glass), and a relatively fusible material (e.g., fluxes, glazes, vitreous enamels, etc., such as lead borates), with the addition, if desired, of about 10% of refractory material (zirconia), are moistened with water or dilute caustic soda solution, moulded cold under compression, and subsequently vitrified at the usual pressure, or, alternatively, they are vitrified and subsequently moulded while hot. L. A. COLES.

Manufacture of abrasive articles. CARBORUNDUM Co., LTD., Assees. of N. P. ROBIE (B.P. 333,409, 23.8.29. U.S., 27.10.28).—The abrasive grains are mixed successively with a liquid reactive resin (e.g., "Bakelite AR0014") and with sufficient of a dry, powdered, reactive resin (e.g., "Redmanol") to yield a workable paste, which is moulded under pressure and heated to harden the bond; inorganic or organic fillers may be added to the paste. L. A. COLES.

Bonding of refractories. S. J. LUBOWSKY, Assr. to METAL & THERMIT CORP. (U.S.P. 1,774,607, 2.9.30. Appl., 5.12.25).—See B.P. 232,680; B., 1925, 632.

Machines for working glass. SOC. ANON. D'ETUDES ET DE CONSTRUCTIONS D'APPAREILS MÉCANIQUES POUR LA VERRERIE (B.P. 334,056, 21.8.29. Fr., 12.10.28).

Methods and apparatus for working fused silica. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. A. MILLAR (B.P. 334,470, 17.12.29. U.S., 20.12.28).

Drying of tiles, bricks, or other ceramic products. A. MONNIER (B.P. 333,921, 20.4.29).

Apparatus for drying, heating, etc. (B.P. 332,951).—See I. **Linings for furnaces** (B.P. 333,582).—See XI.

IX.—BUILDING MATERIALS.

Plaster [of Paris] and mould-making. A. HEATH (Trans. Ceram. Soc., 1930, 29, 231—238).—A brief outline is given of the manufacture of plaster and of the factors affecting the hardness of moulds made therefrom. R. J. CARTLIDGE.

Grinding of cement clinker. A. C. DAVIS (Cement, 1930, 3, 1184—1195).—Types of grinding mill are described and illustrated.

PATENTS.

Preservation of natural and artificial building materials. D. DE ROS (B.P. 333,535, 13.5.29).—A hot solution of gum arabic is sprayed on to the surface of the stone under sufficient pressure to cause it to penetrate to the depth of the weathered zone. On drying, the deleterious salts in the stone are drawn to the surface by capillarity and may be removed by brushing.

A. R. POWELL.

Burning cement in rotary kilns. O. LELLEP (U.S.P. 1,775,313, 9.9.30. Appl., 22.3.28. Ger., 2.4.27).—See B.P. 288,192; B., 1929, 395.

Bituminous emulsions (B.P. 333,152—3, 333,303, and 333,496).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Composition of the bath in Siemens-Martin furnaces at different depths. S. SCHLEICHER (Stahl u. Eisen, 1930, 50, 1049—1060).—Samples of metal have been taken from various depths in the open-hearth furnace during the several stages of the steel-making process and analysed for carbon, manganese, sulphur, and phosphorus. Except in the case of the two last-named elements there is relatively little variation in the composition with the depth during the refining operation, as the reaction takes place with great speed throughout the mass of molten metal. The size of the iron ore added to burn out the carbon has practically no effect on the rate of carbon removal, and, provided that sufficient carbon is present, the whole of the oxygen content of the ore can be utilised. Although desulphurisation with fluorspar is a much more prolonged operation, the sulphur content of the metal during this stage of refining varies only slightly from top to bottom of the bath. During the deoxidation with ferromanganese the composition of the slag shows wide variations, especially in the magnesia content; this is due to the relatively high viscosity caused by the presence of manganese oxide. A. R. POWELL.

Rapid test of the tendency to rust of iron and steel. ANON. (Jahresber. VII Chem.-Tech. Reichsanst., 1928, 216; Chem. Zentr., 1930, i, 2003).—The quantity of sulphuric acid necessary to de-passivate the metal after treatment with chromic acid is determined. Addition of a small quantity of copper sulphate is advantageous. A. A. ELDRIDGE.

Estimation of the reaction possibilities in steel production by the aid of physico-chemical principles. H. SCHENCK (Stahl u. Eisen, 1930, 50, 953—966).—The course of the chemical reactions which take place during the manufacture of steel in open-hearth furnaces or basic converters is limited by a "maximum efficiency value," which corresponds with the equilibrium stage of the reaction, and hence determines the course of the reaction. The relations between this value, the temperature, and the composition of the slag are elucidated for the reactions involving manganese, phosphorus, and sulphur, and it is shown that the closeness with which the "actual efficiency value" approaches the theoretical maximum is dependent solely on the conditions existing in the furnace. Expressions are also derived showing the relationship between the carbon content of the bath, the ferrous oxide content of the slag, and the temperature. A. R. POWELL.

Dynamic strengthening and capacity for overloading of steels. A. THUM and W. WISS (Mitt. Materialprüf., 1930, 111—112).—By subjecting steel to 5,000,000 reversals of a load just below the fatigue strength and then repeating the treatment several times after increasing the load by 1 kg./mm.² every time, the fatigue limit may be increased by as much as 20%. If the load is increased after fewer reversals the material acquires a greater strength at the cost of a shorter effective life. Previous static cold-work also increases the fatigue strength and the capacity of the metal to withstand overloads in dynamic tests. A. R. POWELL.

Iron oxides and hydroxides produced in the rusting of iron. G. SCHKORR (Mitt. Materialprüf., 1930, 118—119).—Iron rust consists generally of a mixture of two forms of ferric hydroxide, FeO·OH. The α -form, which remains non-magnetic on heating above 200°, results from the direct oxidation of ferrous ions, and the γ -form, which becomes magnetic with the loss of water at 200°, is produced by the oxidation of intermediate green and black compounds containing both ferrous and ferric oxides. The yellow turbidity often seen in tap water from iron pipes consists of α -hydroxide, whereas the brown to black adherent coating of rust on iron is mainly γ -hydroxide. Besides these compounds iron rust may contain black ferrous ferrite, Fe₃O₄.aq, white ferrous hydroxide (in the absence of oxygen), green ferrous ferrite as an intermediate product in the formation of the black compound, red ferric oxide (above 70° in the presence of excess of oxygen), and amorphous brown ferric hydroxide (under accelerated rusting conditions). The mechanism of rusting may thus be explained: in air-free water a black film is formed, Fe+2H₂O=Fe(OH)₂+2H; in the absence of air α -FeO·OH sol reacts with iron to form Fe₃O₄ or a green sol, FeO·OH+H=Fe(OH)₂; the green sol reacts with the FeO·OH to produce the green ferrite Fe(OH)₂.FeO·OH, or with an excess to form Fe₃O₄, and finally the latter is oxidised to γ -FeO·OH. A. R. POWELL.

Alterations in the properties of hæmatite pig iron cast from the first melting. E. PRIVOVARSKY (Stahl u. Eisen, 1930, 50, 966—968).—The alterations which take place in the mechanical properties of pig iron during tapping from the blast-furnace into an acid mixer from which finished castings are poured

have been determined. The composition and properties of samples cast in sand and in chill moulds directly from the blast furnace during the charging of the mixer and during the emptying of the mixer are tabulated, showing the variations over a period of 7 days. Sand castings have the better properties when taken directly from the blast furnace, but chill castings are superior to sand castings poured from the mixer. In both cases an appreciable improvement in properties takes place during the passage of the metal through the mixer.

A. R. POWELL.

Ageing of metals. A. KRÜGER (Arch. Eisenhüttenw., 1929—1930, 3, 721—730; Stahl u. Eisen, 1930, 50, 768).—The notched-bar impact strength of boiler-plate and low-silicon steels after artificial ageing varies between 10 and 22% of that of the untreated metal. Quenching and annealing at 400—600° renders these steels less sensitive to ageing, whereas severe cold-rolling increases their sensitivity to ageing. The values of the impact strength of these steels after various heat treatments and of zinc and brass have been determined between —80° and 300°. The changes in micro-structure during ageing of these alloys have been followed, and the results are discussed on the basis of the modern theories of age-hardening. A. R. POWELL.

Artificial ageing of duralumin and super-duralumin. K. L. MEISSNER (Inst. Metals, Sept., 1930. Advance copy. 28 pp.).—Artificial ageing of duralumin (4.2% Cu, 0.5% Mg, 0.3% Si, 0.3% Fe, 0.25—0.6% Mn, remainder Al) which has previously been age-hardened at the ordinary temperature for 5 days causes a decrease in the Brinell hardness, yield point, and tensile strength at 100—125°, after which the values of these properties rise to maxima at 150—170°. The yield point decreases from 25 kg./mm.² to 22 kg./mm.² after ageing at 100°, but increases to 36—38 kg./mm.² after ageing at 160—170°, whereas the tensile strength varies only between 38 and 42 kg./mm.² during this treatment, but the ductility and flexibility are considerably reduced by ageing at 160—170°. Metal of high yield point, tensile strength, and ductility is obtained only by annealing at 100—125° material which has been cold-rolled and age-hardened at the ordinary temperature. Ageing above 175° reduces the hardness, yield point, and tensile strength of the alloys, but increases the ductility slightly and the flexibility considerably. Artificial ageing applied directly to the quenched material produces similar results in all cases. Artificial ageing of super-duralumin (4.4% Cu, 0.54% Mg, 0.79% Si, 0.25% Fe, and 0.61% Mn) proceeds in the same way as with duralumin, but the increase in yield point with ageing above 125° is much more marked and the values for the other tensile properties are distinctly higher for any given heat treatment. The causes of these changes during ageing are discussed at some length, but no satisfactory explanation to account for them can yet be put forward.

A. R. POWELL.

Protecting light aluminium alloys from corrosion. G. V. AKIMOV (Vestn. Metalloprom., 1929, 9, 81—88).—The fact that duralumin sheets riveted to zinc sheets were not corroded by sea-water is discussed. Determinations of the P.D. of an aluminium-zinc couple

in salt water showed that the zinc was the anode; the *P.D.* increased rapidly at first and then gradually diminished to a constant value, the aluminium acquiring a stable, protective, brown film. The protective influence of zinc extends over a considerable distance.

CHEMICAL ABSTRACTS.

Protecting aluminium and its alloys from corrosion. H. J. HÜESTER (Amer. Machinist, Eur. Ed., 1929, 70, 331).—The metal (containing up to 4% Cu) is given an oxide coating by electrolysis in chromic acid solution (3%).

CHEMICAL ABSTRACTS.

Resistance of metals and alloys to attack by salt solutions. ANON. (Jahresber. VII Chem.-Tech. Reichsanst., 1928, 205—211; Chem. Zentr., 1930, i, 2003).—The degree to which nickel, copper, iron, and alloys of these metals are attacked by solid salts depends essentially on the hygroscopicity of the salts; the order of diminishing corrosive action is: magnesium chloride, carnallite, "Hartsalz," sodium chloride, potassium chloride, magnesium sulphate. The type of corrosion is described. The loss of weight was greatest with the ferrous metals. In the corrosion of metals and alloys by aqueous solutions of salts, exclusion of air diminishes the corrosion by 50%; the loss of weight increases with increasing quantities of solution per cm.² of surface, when the temperature is raised, or when oxygen is passed through the liquid.

A. A. ELDRIDGE.

Open-air corrosion of copper. II. Mineralogical relationships of corrosion products. W. H. J. VERNON and L. WHITBY (Inst. Metals, Sept., 1930, Advance copy. 8 pp.; cf. B., 1929, 855).—Analysis of corrosion products of various ages on copper exposed to town, country, and sea air indicates that, after prolonged exposure, the constituents present tend to assume the composition of the corresponding minerals, basic copper sulphate becoming brochantite, the basic chloride atacamite, and the basic carbonate malachite. In the younger deposits in towns cuprous sulphide (chalcocite) often occurs; this then oxidises to blue cupric sulphide (covellite). When lead is present in or near the copper the double basic sulphate caledonite occurs in the corrosion products. After shorter periods the products are less basic than the corresponding minerals.

A. R. POWELL.

Rapid determination of copper in poor minerals for tariff purposes. M. LORA and L. SILVÁN (Anal. Fis. Quím., 1930, 28, 718—723).—For the rapid assay of copper in ores containing up to 2.5% of the metal the electrolytic method may with advantage be replaced by Heine's colorimetric procedure, which permits a rapid classification of ores into the customary tariff groups. For ores containing 0.1—1.5% Cu the error does not exceed $\pm 0.05\%$, but the method is not applicable to richer ores.

H. F. GILLBE.

Effects of silicon on the properties of brass. II. H. W. GOULD and K. W. RAY (Metals and Alloys, 1930, 1, 502—507; cf. B., 1930, 911).—Maximal hardness is reached at 9% Si for 85:15 brass or 7% Si for 65:35 and 60:40 brass; other maxima were observed for brittleness, tensile strength, impact breaking strength, and elongation. The working properties of silicon brasses are described.

CHEMICAL ABSTRACTS.

Effect of phosphorus on the strength of Admiralty gun-metal. H. C. DEWS (Inst. Metals, Sept., 1930, Advance copy. 12 pp.).—Phosphorus up to 0.04% has little effect on the tensile strength and density of gun-metal (87.5% Cu, 10% Sn, 2% Zn, 0.5% Pb), but increases the hardness slightly. With more than 0.05% P copper phosphide can be detected in the microstructure of sand-cast alloys, and a sharp reduction takes place in the tensile strength and a slight reduction in the hardness. The density remains unchanged with up to 0.06% P, but larger proportions (up to 0.13% P) cause a slight decrease. The optimum casting temperature varies between 1060° and 1140° according to the phosphorus content.

A. R. POWELL.

Silicon-copper alloys and silicon-manganese-copper alloys. E. VOCE (Inst. Metals, Sept., 1930, 31 pp.).—The mechanical properties and resistance to corrosion of copper alloys with up to 6% Si and with 1—5% Si and 1—5% Mn have been investigated; in the case of the ternary alloys the values of the mechanical properties are reproduced in a number of graphs on the ternary diagram. The tensile strength of copper is considerably increased, but the conductivity is diminished by the addition of 1% Si; this alloy may readily be drawn into wire. The alloy with 4.5% Si is readily hot-rolled into sheet having a tensile strength of 39 tons/in.² and an elongation of 13.5% in the direction of rolling. The alloy with 3% Si can be cold-rolled to 50% reduction and then has a strength of 49 tons/in.² with an elongation of 10%; after annealing, these values become 30 tons/in.² and 60—70%, respectively, and the sheet is suitable for spinning. Of the ternary alloys examined, that with 3.5% Si and 1% Mn has the best mechanical properties, its tensile strength being 22 tons/in.² with an elongation of 25%. These alloys work better hot than cold; those with a high silicon and manganese content can be made to acquire a strength of 50 tons/in.² by hard rolling, and other alloys of the series show an elongation of over 70% in the annealed state. Silicon-copper alloys are slightly more resistant to corrosion than the ternary alloys containing manganese. At temperatures up to 750° the rate of oxidation of silicon-copper alloys is less than that of copper and decreases with increasing silicon content; the presence of manganese has little effect on the rate of oxidation, but renders the oxide less adherent.

A. R. POWELL.

New silicon-zinc-copper alloy. E. VADERS (Inst. Metals, Sept., 1930, Advance copy. 17 pp.).—The solid solubility of silicon in silicon-zinc-copper alloys decreases with increasing zinc content from 6% Si with 0% Zn to about 1.5% Si with 30% Zn. Alloys containing more silicon show the presence of a second constituent which appears blue after etching a microsection with ammonium persulphate. Within the solid-solution range silicon increases considerably the hardness, tensile strength, and ductility of sand- and chill-cast zinc-copper alloys; thus the 4:15:81 silicon-zinc-copper alloy after extrusion or drawing and annealing at 750° has a tensile strength of 60—61 kg./mm.², an elongation of 27%, and a Brinell hardness of 145. The same alloy after extrusion but before annealing gives values of 64.5 kg./mm.², 30.5%, and 147, respectively, and after drawing from 15 to 14 mm. 88 kg./mm.², 15%, and 202, respectively.

Addition of up to 2% Sn or Ni decreases the strength and ductility slightly, but increases the hardness. Alloys with 80–90% Cu and up to 4% Si are very fluid when molten, and yield dense and sharp castings in sand moulds even when the cross-section does not exceed 2 mm. Bells made of an alloy containing 81–82% Cu, 4–5% Si, 14% Zn, and 0.5% Sn have the same tone characteristics as similar bells made of tin bronze, but their d is 10% less. Pressure castings of the same alloy without tin are denser, stronger, and more easily worked than those made from α -brass. A. R. POWELL.

Refining of lead by chlorination. A. S. SHACHMURADOV (Min. Svir. Tzvet. Met., 1929, 4, 323–326).—A process employing direct chlorination at 440–460° is described; the yield is 78%, and the duration of the process 10–12 hrs. CHEMICAL ABSTRACTS.

Heat-treatment, ball hardness, and allotropy of lead. F. HARGREAVES (Inst. Metals, Sept., 1930. Advance copy. 19 pp.).—The Brinell hardness of lead has been measured immediately after quenching from temperatures up to 310° and after subsequent ageing at the ordinary temperature. The hardness-quenching temperature curve of very pure lead (99.999%) shows two marked critical points at 187° and 228°, and it is suggested that lead undergoes allotropic changes at these points. The presence of 0.005% Sn inhibits these changes, but small amounts of bismuth and cadmium are without effect. Marked changes take place in the hardness of the metal within a few minutes of quenching and may continue for a period of several days, the ultimate value reached being well below the value immediately after quenching. Addition of small quantities of impurities rapidly increases the hardness of lead, but the quenched metal is always harder than metal in a state of equilibrium at the ordinary temperature.

A. R. POWELL.

Structure of lead as related to stained glass. S. L. BROWN (J. Brit. Soc. Master Glass Painters, 1927–1928, 2, 123).—An alloy composed of undesilvered lead and 1.5% Sn is least liable to form lead carbonate.

CHEMICAL ABSTRACTS.

Electrodeposition of zinc on aluminium from sulphate solutions. H. C. COCKS (Trans. Faraday Soc., 1930, 26, 517–526).—Zinc deposited from solutions of the sulphate on aluminium improves in texture and becomes finer in grain with decrease in the p_H of the solution or increase in the current density, and at the same time the final static potentials become more positive. Certain addition agents also have a favourable action on the deposit, the effectiveness increasing in the order glucose, β -naphthol, gum arabic, gum arabic + β -naphthol. These substances have the further effect of increasing the throwing power of an unbuffered solution of p_H 3–4. The deposition potential becomes more negative for a given current density as the p_H is reduced and increase of current density at a fixed p_H has a similar effect, although the variations are only small in absence of addition agents. Sodium acetate added as a buffer to a solution containing β -naphthol with or without gum arabic causes the deposition potential to change much less with variation in current density. For the technical electrodeposition of zinc

on aluminium a N -solution of zinc sulphate 0.25*N* in respect of sodium acetate and containing 1 g. of gum arabic per litre, with or without 0.1 g. of β -naphthol per litre, is recommended. R. CUTHILL.

Electrodeposition of nickel. II. Effect of current density and temperature. III. Effect of small quantities of iron and aluminium. J. B. O'SULLIVAN (Trans. Faraday Soc., 1930, 26, 533–539, 540–543; cf. B., 1930, 287).—II. In the deposition of nickel from buffered solutions of the sulphate, variations in the structure of the surface of the deposit with changes in temperature and current density have been traced to variations in the throwing power of the solutions; raising the temperature has the same effect as reducing the current density. The deposition potential graphed against the logarithm of the current density gives a straight line in all cases, but since the slope of the line and deposition potential at unit current density change from one solution to another it is concluded that the constants of the Freundlich isotherm for the adsorption of hydrogen at the cathode-liquid interface (cf. Sand, A., 1930, 297) depend on the p_H and the nature of the buffer present.

III. Small amounts of iron salts have no noticeable effect on the deposits of nickel obtained by electrolysis of solutions of the sulphate, whereas aluminium salts, unless present in extremely small quantities, tend to blacken the deposit. The probable explanation is that iron is completely deposited as metal before it can form a colloid, whilst aluminium accumulates in the cathode film and when the p_H becomes sufficiently high it forms a colloid, part of which is deposited simultaneously with the nickel, thus affecting the structure of the deposit. This further supports the view (B., 1930, 287) that the fineness of grain of nickel deposited in absence of foreign metals is due to the co-precipitation of a little colloidal hydroxide along with the metal. R. CUTHILL.

[Electro]deposition of nickel at high current densities. N. R. LABAN (J. Electroplaters' Dep. Tech. Soc., 1930, 5, 129–134).—Methods satisfactory in mass-production are described, and optimal conditions are recorded. CHEMICAL ABSTRACTS.

Bright plating on small lead parts. J. UNDERWOOD (Monthly Rev. Amer. Electroplaters' Soc., 1929, 16, 44–46).—For copper-plating, sodium cyanide (3 oz.) and Rochelle salt (0.25 oz.) are dissolved in water (1 gal.), copper cyanide being added to the warm solution to saturation; a solution of lead acetate (2 oz.) and sodium hydroxide (2 oz.) is added (1 pint per gal.), and the bath is operated at 70°. For nickel-plating, the bath should contain nickel sulphate (32 oz.), magnesium sulphate (26 oz.), and ammonium chloride (2.1 oz. per gal.), and a small quantity of cadmium chloride. CHEMICAL ABSTRACTS.

See also A., Sept., 1106, **X-Ray analysis of copper-manganese alloys** (PERSSON). **Aluminium-silicon-copper alloys** (URASOV and others). 1112, **Dispersion of nickel in dry acetone** (FOLLEY and HENRY). 1113, **Preparation of tungsten and chromium hydrosols** (LOTTERMOSER and RIEDEL). 1128, **Influence of soluble fluorides on corrosion of iron** (CHAPMAN). 1134,

Tantalum cathode for electrodeposition of copper (MEARS and PINE). **Solvents for electrodeposition of metals** (YNTEMA and AUDRIETH). **Electrolytic transfer of platinum metals** (ATKINSON). **Influence of chromic acid on electrodeposition of nickel** (MACNAUGHTAN and HAMMOND). 1136, **Preparation of alkali metals by reduction with zirconium** (DE BOER and others). 1137, **Preparation of manganese-free magnesium** (ORENT and RASK).

PATENTS.

Furnaces for the heat treatment of metallic or other bodies. WELLMAN SMITH OWEN ENG. CORP., LTD., and E. W. PLUMLEY (B.P. 334,006, 2.7.29).—The furnaces are of the long horizontal kiln type and contain a conveyor device comprising a horizontal shaft carrying a series of fingers which rotate between fixed fingers attached to the walls. The shaft is adapted to move forward when the fingers are pointing upwards, and back again when the fingers point downwards.

A. R. POWELL.

Reduction of iron ores. TRENT PROCESS CORP. (B.P. 313,597, 17.5.29. U.S., 16.6.28).—A mixture of iron ore and a solid carbonaceous fuel is passed through an externally heated cylindrical furnace and the hot reduced iron is quenched in crude petroleum, the resulting vapours being passed backwards through the reduction furnace in which they are cracked; the deposited carbon assists the reduction of the ore.

A. R. POWELL.

Manufacture of stainless iron. W. M. FARNSWORTH, ASSR. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,744,374, 21.1.30. Appl., 29.6.27).—Scrap iron containing about 12% Cr is melted under reducing conditions in an electric furnace lined with magnesia, then treated with ferric oxide (iron ore) at 1700° until the metal contains less than 0.12% C. The substantial quantity of chromium which passes into the slag in this operation is reduced back again into the iron by the addition of lime and ferrosilicon to the charge.

A. R. POWELL.

Dissolving and removing metallic fouling from the surface of iron and steel objects. G. HALBE (B.P. 333,963, 31.5.29).—Particles of copper, nickel, lead, antimony, or their alloys may be removed from the interior of iron or steel tubes, e.g., rifle bores, by treatment with an aqueous or alcoholic solution of mercuric chloride.

A. R. POWELL.

Welding iron or steel alloys containing copper. F. KRUPP A.-G. (B.P. 316,614, 26.7.29. Ger., 1.8.28).—A welding rod consisting of iron with 0.01–0.3% C, 0.5–14% Mn, and 0.4–2% Cu is used. A. R. POWELL.

Producing a titanium or ferrotitanium regulus. W. and H. MATHESIUS (B.P. 333,816, 31.10.29. Ger., 1.11.28).—Ilmenite or titania mixed with a chromate, tungstate, permanganate, or the like is reduced aluminothermically to produce an alloy with a high titanium content and containing also one or more other metals of value as additions to a steel.

A. R. POWELL.

Copper-nickel-iron alloys. W. P. DIGBY (B.P. 333,774, 30.5.29; cf. B.P. 322,216; B., 1930, 106).—

The alloys comprise 25–45% Cu, 5–15% Ni, and the remainder iron, up to 20% of the total of which may be replaced by chromium. A small amount of manganese is added just prior to casting, followed, if desired, by magnesium as a deoxidiser.

A. R. POWELL.

[Centrifugal] gold separator. J. V. RAWLINGS, ASSR. to E. M. BEUS (U.S.P. 1,750,860, 18.3.30. Appl., 8.6.29).—The apparatus comprises three bowls rotating as one unit about a central shaft; the innermost bowl is provided with horizontal riffles and a flared upper edge containing a series of holes through which the pulp overflows into the second bowl, which is wider and shallower than the first and is provided with tubes at the lower part communicating with the lowermost bowl. This bowl is hemispherical in shape, and has a series of vertical riffles in the lower part and horizontal riffles in the upper part. The apparatus thus forms a three-stage concentrator, gold accumulating in the apex of the first bowl and other heavy minerals in the riffles of the two succeeding bowls.

A. R. POWELL.

Granulated product and its manufacture [from arsenical fume]. H. C. GARDNER (U.S.P. 1,755,985, 22.4.30. Appl., 22.8.27).—Dust resulting from the reduction of arsenical ores is granulated by mixing it with dilute sulphuric acid, or with sulphuric acid and then water, at the same time agitating and drying the mixture.

B. M. VENABLES.

Production of rhenium. Extraction of rhenium concentrations. SIEMENS & HALSKE A.-G. (B.P. 317,035 and 332,627, 27.2.29. Ger., [A] 9.8.28, [B] 22.11.28).—(A) Molybdenite is dissolved in nitric acid and the solution treated with ammonium nitrate and phosphate to remove the greater part of the molybdenum as ammonium phosphomolybdate. The filtrate is treated with hydrogen sulphide, the mixed molybdenum rhenium sulphide redissolved in nitric acid, and the phosphate treatment repeated. This procedure, after several repetitions, affords a sulphide precipitate from which a rhenium concentrate may be obtained by fractional sublimation in air. (B) The solution of the mineral in hydrochloric acid is treated with hydrogen sulphide, and the precipitate further treated as above.

A. R. POWELL.

Electrodes or welding rods for use in the process of electrically welding or depositing metals. J. E. POLLAK. From R. D. THOMPSON and G. C. MILLS (B.P. 333,985, 11.6.29).—The rod is coated with a paint composed of aluminium or an aluminium alloy dispersed in amyl acetate, with or without an outer coating of flux.

A. R. POWELL.

Electric smelting of zinc ore. E. C. GASKILL, ASSR. to ST. JOSEPH LEAD CO. (U.S.P. 1,743,886, 14.1.30. Appl., 17.11.26).—Roasted zinc ore is heated at the end of the roasting operation to a temperature at which it sinters to porous granules. A mixture of these with a carbonaceous fuel is passed through a two-stage preheater into a vertical, rectangular, electrical resistance furnace on two opposite sides of which are carbon electrodes between which the ore mixture forms a resistor column in which the heat required for reduction and volatilisation of the zinc is generated.

A. R. POWELL.

Production of metal sheets by electrolytic means. E. KELSEN (B.P. 314,434, 26.6.29. Austr., 27.6.28).—Electrolysis is effected in a shallow circular tank on the bottom of which is placed the anode material; the cathode comprises a rotating circular disc forming the lid of the tank and operating in a water-seal.

A. R. POWELL.

Deoxidation of molten metals or alloys. G. MASING, ASSR. to METAL & THERMIT CORP. (U.S.P. 2,774,837, 2.9.30. Appl., 1.3.28. Ger., 11.4.27).—See B.P. 288,543; B., 1928, 820.

Pigments for anticorrosive paint etc. (B.P. 318,639).—See XIII. **Rubber coated with metal** (B.P. 311,766).—See XIV.

XI.—ELECTROTECHNICS.

Method of mapping equipotential lines and its application to electrical precipitator problems. A. W. SIMON and L. C. KRON (Rev. Sci. Instr., 1930, 1, 527—536).

Reproducing coke sections. ZIPPERER and LORENZ. **Transformer oils.** YAMADA.—See II. **Ammonia and electric power.** McMICHAEL. **Dissociation of carbon dioxide electrically.** FISCHER and others.—See VII. **Steel for dynamo etc. sheets.** KUSSMANN and others. **Protecting aluminium.** AKIMOV; HUESTER. **Zinc on aluminium.** COCKS. **Electrodeposition of nickel.** O'SULLIVAN; LABAN. **Plating on lead.** UNDERWOOD.—See X.

See also A., Sept., 1112, **Dispersion of nickel in dry acetone** (FOLLEY and HENRY). 1121, **Iron-nitrogen system; magnetic investigation** (LEHRER). 1134, **Tantalum cathode for electrodeposition of copper** (MEARS and PINE). **Solvents for electrodeposition of metals** (YNTEMA and AUDRIETH). **Electrolytic transfer of platinum metals** (ATKINSON). **Influence of chromic acid on electrodeposition of nickel** (MACNAUGHTAN and HAMMOND). 1135, **Ozone production in silent electric discharge** (BRINER and SUSZ). 1136, **Preparation of pure hydrogen electrolytically** (HULUBEI). 1151, **Triode valve for determination of p_H** (HARRISON). 1152, **Application of photoelectric cell to chemical analysis and control** (PARTRIDGE).

PATENTS.

Electric induction furnace [without iron core]. HIRSCH KUPFER- U. MESSINGWERKE A.-G. (B.P. 333,455, 15.10.29. Ger., 15.10.28).—A deep groove, of width less than the depth of penetration of the eddy currents in the melting material in the crucible, is arranged in the bottom of the crucible near its walls. J. S. G. THOMAS.

[Refractory linings for] electrical induction furnaces. N. R. DAVIS, G. A. WOOD, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 333,582, 15.5.29).—Lining material is graded so as to be most easily sintered near the rim and least easily where most rapid erosion is likely to occur. J. S. G. THOMAS.

[Dental] electric furnace. A. J. ASCH (U.S.P. 1,757,895, 6.5.30. Appl., 5.1.28).—A heating element consisting of a rectangular strip of platinum foil is

arranged between an inner and an outer muffle, and its free end portions, projecting outside the wall of the outer muffle, are provided with removable circuit-connectors. J. S. G. THOMAS.

Electric purification of gases containing hot vapours. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 316,626, 31.7.29. Ger., 1.8.28).—Hot gases are cooled before entering the purifying chamber, by admixture of colder gas, e.g., air, in proportion controlled by the current flowing in the electro-filter. J. S. G. THOMAS.

Ensuring stability of operation in the electrical cleaning of gases from revolving furnaces. LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 333,427, 23.9.29).—Gas enters the electrical cleaning apparatus at above 150°, with a linear velocity about half that necessary at lower temperatures. The potential of the electric field is increased progressively in successive stages. J. S. G. THOMAS.

Apparatus for removing dust from electrodes [in Cottrell-type precipitators]. A. L. LABBE, ASSR. to AMER. SMELTING & REFINING Co. (U.S.P. 1,758,404, 13.5.30. Appl., 2.9.25).—A device in which one of a number of vibrators, actuated by air pressure or electrically to deliver rapid blows to the bars or frames supporting the electrodes, is described. J. S. G. THOMAS.

Compound for impregnation of cable insulation. HERCULES POWDER Co., Assees. of G. F. KENNEDY (B.P. 333,196, 5.3.29. U.S., 24.12.28).—A mixture of 5—50% of abietic acid, substantially pure and free from resenes, with hydrocarbon oils is claimed. J. S. G. THOMAS.

Electron-discharge apparatus. H. C. THOMPSON, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,756,889, 29.4.30. Appl., 14.12.27).—The emitting surface is formed by coating an electrode with a metallic compound having alkaline properties, e.g., lithium borate, either alone or mixed with amyl acetate (zapon) varnish, and vaporising the compound *in vacuo*. J. S. G. THOMAS.

[Cathode for] electron-discharge device. L. K. MARSHALL, ASSR. to OLD COLONY TRUST Co. (U.S.P. 1,758,710, 13.5.30. Appl., 31.1.29).—A film of caesium is deposited upon a metallic member coated with palladium which has absorbed hydrogen, and a discharge is passed between this electrode and an anode in helium. J. S. G. THOMAS.

Manufacture of a glowing cathode for electron-discharge device. S. LOEWE (B.P. 307,930, 15.3.29. Ger., 16.3.28).—The surface of a sheath of easily oxidisable metal, e.g., nickel, chromium, or iron, surrounding a core of non-oxidisable metal, e.g., platinum, is oxidised by heating, and coated with electron-emitting material. J. S. G. THOMAS.

Cathodes for thermionic valves. Thermionic cathodes. B. LOEWE (B.P. 306,960, 27.2.29, and B.P. 309,140, 4.4.29. Ger., [A] 28.2.28, [B] 5.4.28).—(A) A powdered mixture of substances, e.g., barium oxide and silicon or ferrosilicon, preferably in the form of compressed tablets, is arranged and heated in the discharge device so that material of high electronic emission, e.g., barium, produced by reaction between the components of the mixture, is deposited on the cathode.

If desired, one of the products of the reaction may be added to the mixture. (B) Ferrosilicon, aluminium, magnesium, or other reaction component is added to barium hydroxide or strontium hydroxide, freed from water of crystallisation by immersion in molten hydrocarbons, *e.g.*, paraffin wax, heated, if desired, under high or low pressure. [Stat. ref. to (A).] J. S. G. THOMAS.

Manufacture of [metallic clean-up for] vacuum device. H. C. RENTSCHLER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,757,976, 13.5.30. Appl., 27.9.24).—Misch metal is attached to an iron electrode which can be heated to vaporise the misch metal. J. S. G. THOMAS.

Apparatus for producing photochemical radiations. H. SPINDLER (B.P. 309,497, 10.4.29. Fr., 11.4.28).—Mercury vapour, heated by a mercury-vapour arc discharge to a temperature corresponding to a pressure of several hundred atmospheres, is exposed to an electric current much greater than that customarily used in mercury-vapour lamps. The device is applicable to the synthesis of formaldehyde. J. S. G. THOMAS.

Electrolytic apparatus [for electrolysis of water etc.]. F. G. CLARK and J. N. SMITH, Assrs. to F. G. CLARK (U.S.P. 1,757,235, 6.5.30. Appl., 7.4.21. Renewed 8.2.27).—An annular anode and cathode are arranged in spaced relation to a horizontal, annular, slot-like passage between them, through which electrolyte flows. Means are provided for dividing the electrolyte into anolyte and catholyte, and for freeing both from oxygen and hydrogen. J. S. G. THOMAS.

Production or influencing of diaphragm for electro-[end]osmotic purposes. SIEMENS & HALSKE A.-G. (B.P. 312,068, 10.5.29. Ger., 18.5.28).—To prevent migration of liquid in such diaphragms, a substance such as "chromoxide" may be added to them, or they may be coated with material, *e.g.*, chrome-gelatin, so that the natural potential of the diaphragms is less than 0.03 volt, measured in 0.05% potassium chloride solution.

J. S. G. THOMAS.

Production of a good conducting electrical connexion between a metal compound layer and a coating of a ductile metal applied thereon [e.g., in dry copper oxide rectifiers]. SIEMENS-SCHUCKERT-WERKE A.-G. (B.P. 334,140, 25.11.29. Ger., 5.12.28).—To produce strong permanent joints between the leads and oxide-coated copper plates the oxide layer is coated with a thin film of fine graphite, then sprayed with a ductile metal, *e.g.*, zinc, and finally the joint is covered with a paste comprising red lead or manganese dioxide and a japan varnish.

A. R. POWELL.

Dipping solution for electric condensers. P. NEUMANN, Assr. to TELEPHON-APPARAT FABR. E. ZWEITUSCH & Co. (U.S.P. 1,775,136, 9.9.30. Appl., 13.7.28. Ger., 29.10.27).—See B.P. 299,697; B., 1929, 527.

Electrical apparatus for heating liquids. F. FOY (B.P. 333,869, 19.2.29).

Electron-discharge devices. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. W. HULL (B.P. 309,442, 25.2.29. U.S., 10.4.28).

Electron-discharge tubes. MARCONI'S WIRELESS TELEGRAPH Co., LTD., Assees. of W. VAN B. ROBERTS (B.P. 320,022, 30.9.29. U.S., 28.9.28).

[Gas-filled] electric discharge devices. H. J. SPANNER and U. DOERING (B.P. 311,282, 7.5.29. Ger., 8.5.28).

Vacuum electric tube devices. E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 334,256 and 334,298, [A] 31.5. and 2.8.29, [B] 10.6.29).

X-Ray tubes [with rotating anticathode]. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 318,995, 26.7.29. Holl., 14.9.28).

Photo-electric cell. E. G. O., A. E. M., and K. E. H. PRESSLER (O. PRESSLER, THÜRINGER VAKUUMRÖHREN-FABR. U. FABRIK WISSENSCHAFTLICHER APPARATE) (B.P. 316,937, 24.7.29. Ger., 6.8.28).

Salinometers (B.P. 333,727). **Chemical synthesis** (B.P. 314,999).—See I. **Indicator for inflammable gases** (B.P. 331,711). **Liquid fuel** (B.P. 316,945).—See II. **Electrodes or welding rods** (B.P. 333,985). **Smelting of zinc ore** (U.S.P. 1,743,886). **Metal sheets** (B.P. 314,434).—See X. **Antiseptic liquid** (B.P. 332,520).—See XX.

XII.—FATS; OILS; WAXES.

Various causes of turbidity in soap solutions. R. BÜRSTENBINDER (Chem. Umschau, 1930, 37, 232—233).—The turbidity of soap solutions may in many cases be referred to other causes than hydrolysis, which may even be greater in a clear solution than in a cloudy one. Turbid solutions resulting from the presence of lime soaps can be cleared by the addition of 1—3% of sugar or 0.5—1% of potassium chloride.

E. LEWKOWITSCHE.

Apparent iodine value of tung oil as a criterion of its purity. J. VAN LOON (Chem. Umschau, 1930, 37, 229—232).—A curve has been constructed showing the variation of apparent iodine value (Wijs, 2½ hrs.) with the excess of reagent employed: this curve appears to be characteristic for pure tung oils, and may be used for identification by comparing the observed iodine value of an unknown sample with the figure obtained from the curve for the same concentration. A sharp bend in the curve at iodine value 160 corresponds to two thirds saturation of the elæostearic acid present.

E. LEWKOWITSCHE.

Use of the thiocyanogen value. IV. **Analysis of oils containing linolenic acid.** **Composition of soya-bean oil.** W. KIMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 325 B).—The specimen of soya-bean oil (thiocyanogen value 79.7; other properties given) contained 0.54% of unsaponifiable matter and 95.7% of fatty acids (thiocyanogen value 83.1, iodine value 139); solid fatty acids were determined by the lead salt-alcohol method, and the following composition was calculated: saturated fatty acids 11.8%, oleic acid 25.9%, linoleic acid 58.8%, linolenic acid 3.8%.

C. W. SHOPPEE.

Pure and adulterated butters. HOTON.—See XIX. **Effect of stabilisers in lard.** HUSA.—See XX.

See also A., Sept., 1162, **Iron soaps** (SALVATERRA). 1203, **Fatty oil of bull frog** (TASAKI and YAMAMOTO). **Unsaponifiable matter in egg-yolk oil** (FRANKEL and MATHIS).

PATENTS.

Saponification of glycerides of fatty acids. DEUTS. GOLD-U. SILBER-SCHNEIDENSTALT FORM. ROESSLER (B.P. 308,603, 21.3.29. Ger., 23.3.28).—Fats are saponified at about 100° by the combined action of (aqueous) alkaline carbonates and lipolytic agents such as sulphonated "castor bases," "Idrapid" reagent, etc. E. LEWKOWITSCH.

Cleavage of fatty acid esters. H. T. BÖHME A.-G. (B.P. 334,022, 19.7.29. Ger., 17.10.28).—The products obtained by the sulphonation of mixtures of alcohols corresponding to the higher fatty acids (*e.g.*, oleyl alcohol) and aromatic hydrocarbons (*e.g.*, naphthalene) are used as catalysts in fat hydrolysis. E. LEWKOWITSCH.

Production of emulsions of oxidised drying oils, or varnishes or lacquers containing such oils. P. C. VAN DER WILLIGEN (B.P. 333,690, 25.6.29. Holl., 6.11.28).—Emulsions of drying oils, or varnishes, are oxidised at moderate temperatures (30–70°) by blowing with air etc. (*cf.* B.P. 301,901; B., 1930, 420), while the p_H of the emulsion is maintained at not below 8 by the addition of alkalis or buffer substances, *e.g.*, sodium carbonate, soap; under these conditions flocculation of the linoxyn formed is avoided. E. LEWKOWITSCH.

Distillation of glycerin (U.S.P. 1,743,488).—See I. **Emulsification of tar etc.** (B.P. 333,152–3).—See II. **Esters** (B.P. 332,267).—See III. **Wetting agents** (B.P. 313,160).—See VI. **Linnoxyn** (B.P. 332,257).—See XIII. **Preventing oxidation etc. of organic compounds** (B.P. 332,764).—See XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Viscosity of paints. H. WOLFF (Farben-Ztg., 1930, 35, 2175–2176, 2228–2229; *cf.* B., 1929, 825, 947).—Using the Wolff-Hoepke turbo-viscosimeter, the relation between drop weight used and time of fall over a given distance was studied for paints of various oil contents. For water and linseed oil the product of weight and time is constant within limits, but for paints based on basic lead sulphate, zinc oxide, lithopone, and micaceous iron oxide the product decreases with decreasing time of fall. The ratio of the products for times of fall of 20 and 10 sec. is practically independent of oil content for usable paints based on a given pigment in linseed oil. The value is highest for zinc oxide and least for micaceous iron oxide. It is also higher for lithopone-linseed oil than for lithopone-stand oil, the latter value agreeing with that for stand oil alone. The bearing of these results on plasticity is discussed with reference to the viscosity equation previously developed (*loc. cit.*). S. S. WOOLF.

Analysis of titanium white. J. PFANHAUSER and S. TOMASZEWSKI (Przemysl Chem., 1930, 14, 353–355).—Titanium white is fused with potassium hydrogen sulphate, and the extract of the melt is left 2–3 hrs. with about 0.5 g. of zinc and excess of hydrochloric acid. Ferric ammonium sulphate is then added to the solution, and ferrous iron produced by oxidation of titanous acid is titrated with potassium permanganate solution in the presence of Reinhardt's reagent. In the presence of a

large excess of zinc a correction, amounting to 0.83 c.c. of 0.1N-permanganate per g. of zinc, should be deducted from the number of c.c. of permanganate used.

R. TRUSZKOWSKI.

See also A., Sept., 1115, **Measurement of particle size in ultra-violet light** (HASLAM and HALL).

PATENTS.

Manufacture of [rubberised] paint. W. C. GEER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,744,881, 28.1.30. Appl., 6.6.27).—Compositions containing an artificial isomeride of rubber, *e.g.*, one prepared by heating rubber with phenolsulphonic acid for 6 hrs. at 140°, to which solvents, pigments, fillers, flowing agents, preservatives, or antioxidants, *e.g.*, condensation products of aldehydes with nitrogenous bases, may be added, are claimed. S. S. WOOLF.

[Cellulose ester] coating. E. C. HAINES, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,744,699, 21.1.30. Appl., 22.1.26).—From 0.1 to 20% by wt. of finely-divided nitrocellulose or appropriate cellulose ester is incorporated with an oil or varnish type of undercoat to which a nitrocellulose or cellulose ester lacquer, normally substantially non-adherent thereto, is to be applied. S. S. WOOLF.

Production of pigments, particularly for compositions used as anticorrosives and for painting ships' bottoms. F. RAHTJEN and M. RAGG (B.P. 318,639, 7.9.29. Ger., 8.9.28).—Raw materials containing oxides or other compounds of lead are heated in the absence of air at 380–400° with reducing agents, neutral substances (*e.g.*, ilmenite, zinc oxide, silicates, borates) being added to prevent the molten lead from forming macroscopic balls. As reducing agents there may be used organic materials of high m.p. or b.p., *e.g.*, pitch, soot, resins, tall oil, glycerin, etc., or inorganic materials, *e.g.*, zinc, aluminium, calcium, galena, antimony sulphide, etc., used in excess for the purpose of forming lead alloys. S. S. WOOLF.

Manufacture of [synthetic] resins. J. C. MORRELL and G. EGLOFF, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,744,135, 21.1.30. Appl., 3.1.28).—Chlorine or other halogen gas is passed through a mixture of polymerised hydrocarbons, *e.g.*, those obtained from petroleum distillates, and phenols at raised temperatures (150°) until the product is suitably viscous. S. S. WOOLF.

Manufacture of soluble and fusible synthetic resin. W. H. MOSS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,743,680, 14.1.30. Appl., 3.9.27).—An alkali dihydrogen phosphate is used as catalyst for phenol-formaldehyde condensations, being added as such or formed *in situ* by carrying out the condensation in the presence of phosphoric acid and adding a suitable amount of alkali prior to the formation of an insoluble and infusible resin. Resistance to darkening on exposure to light and freedom from tendency to change into the insoluble form are claimed. S. S. WOOLF.

Production of shaped articles from urea or its derivatives and solid polymerised formaldehyde. BAKELITE GES.M.B.H. (B.P. 318,883, 318,959, 333,296, and 333,298 [A–D] 4.2.29. Ger., [A, D] 11.9.28, [B, C]

13.9.28. Addns. [B] to B.P. 305,211 and [C, D] to [A].—(A) The condensation described in B.P. 305,211 (B., 1930, 828) is carried out in a melt of organic compounds, *e.g.*, natural or non-alkaline artificial resins (other than urea-phenol-formaldehyde resins), which are not capable of being hardened or only capable of being hardened with greater difficulty than urea-formaldehyde condensation products. (B) The parent process (B.P. 305,211) is carried out with the addition of acid, neutral, or basic catalysts. (C) The process described in (A) is carried out in the presence of acid, neutral, or basic catalysts. (D) The process described in (A) is carried out in the melt of solid hydrocarbons or solid chlorinated, nitrated, or hydrogenated hydrocarbons.

S. S. WOOLF.

Preparation of linoxyn or similar materials. V. SCHOLZ, and ATLAS AGO CHEM. FABR. A.-G. (B.P. 332,257, 13.3.29).—Linseed oil, tung oil, castor oil, or mixtures of such oils or the fatty acids thereof, to which catalysts, *e.g.*, litharge, have been added, are blown at a suitable temperature with air or similar gas containing free oxygen until a viscous, yellow-brown, blown linoxyn or corresponding product capable of no further oxidation by blowing is obtained. This is then kneaded with air under controlled conditions, giving rise to light-coloured granular products. The addition of 0.5–2% of bases, *e.g.*, aniline or urea, in the blowing or kneading processes stabilises the products. These may be incorporated with resins, nitrocellulose, celluloid, saponification agents, etc.

S. S. WOOLF.

Coating, plastic, impregnating, adhesive, and like compositions. V. SCHOLZ, and ATLAS AGO CHEM. FABR. A.-G. (B.P. 332,266, 13.3.29. Cf. preceding abstract).—Solid linoxyn is dissolved in alcohol or other suitable organic solvent under pressure and heat, the solvent is evaporated, and the oil so obtained is mixed with, *e.g.*, swollen nitrocellulose, the solvent of the latter being subsequently removed. Alternatively, the solid linoxyn may be kneaded with the cellulose gel and after removal of solvent the plastic or pulverulent mass is converted by pressure and heat into a transparent product, "linoloid," soluble in ethyl acetate, acetone, etc. Rubber solutions with or without the addition of amino-compounds, pigments, fillers, etc. may be incorporated.

S. S. WOOLF.

Heat treatment of plastic and other material. R. E. COLEMAN, Assr. to ECONOMY FUSE & MANUF. CO. (U.S.P. 1,727,964, 10.9.29. Appl., 13.4.23).—Materials to be heat-cured, *e.g.*, electrical insulating materials, baking japans, etc., comprising a base and a "flowability-conferring agent" which includes a volatile oil, are heated in a flowing, inert, dry atmosphere, *e.g.*, dry steam or carbon dioxide, with which, if desired, oxygen-containing gas may be admixed. The rate of flow of gas is such as to restrict the polymerisation of the "flowability-conferring agent."

S. S. WOOLF.

Manufacture of artificial masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,166 and 333,304, [A] 25.4.29, [B] 14.5.29).—(A) One or more vinyl esters in the presence of at least 5% by wt. of acid condensing agents are condensed with mono- or poly-hydric phenols or mixtures thereof, inert solvents, diluents, fillers, or

colouring materials are added if desired, and the product is hardened, *e.g.*, by the use of sulphur. Polymerised vinyl esters may be employed, preferably by dissolving them in the monomeric vinyl ester, or the esters may be polymerised to a certain extent, with or without catalysts, before the condensation with phenols in the presence of acid catalysts is undertaken. (B) Solid derivatives of cellulose are worked mechanically with 3–10 times their weight of one or more fillers in the presence of softening agents and resins, and, if desired, of colouring material, water, and up to 10% by wt. of the solid constituents used of an easily volatile organic solvent (which is evaporated during the operation), until a homogeneous mass is obtained which is suitable for priming purposes after dilution with organic solvents.

S. S. WOOLF.

Production of iron oxide pigments. J. LAUX, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,774,930–2, 2.9.30. Appl., [A] 7.9.26, [B, C] 8.9.27. Ger., [A–C] 20.8.25).—See B.P. 263,376; B., 1927, 156.

Apparatus for spraying of paint or other liquids. T. D. JONES and S. WAGENHEIM (B.P. 333,908, 15.5.29).

Grinding paints, inks, etc. (B.P. 333,436).—See I. Colloidal solutions and pastes (B.P. 332,557).—See III. Azo pigments (B.P. 332,208 and 332,319).—See IV. Abrasive articles (B.P. 333,409).—See VIII. Varnishes or lacquers (B.P. 333,690).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Use of madder lake in accelerated rubber mixtures. R. DITMAR and O. FUHRMANN (Caoutchouc et Gutta-Percha, 1930, 27, 15,172–15,173).—The lake obtained by precipitation of alizarin on alumina is not stable towards vulcanisation with sulphur chloride. It withstands heat-vulcanisation satisfactorily in the presence of certain organic accelerators, *e.g.*, some dialkyl-dithiocarbamates and mercaptobenzthiazole combinations, but others cause discoloration.

D. F. TWISS.

PATENTS.

Production of [thermoplastic] conversion products of rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,762, 6.8.29).—Rubber, in presence of a solvent if desired, is treated with a phosphoryl halide at 15–40°. The solvent is distilled off and the phosphorus halide is removed by washing with dilute ammonia, *e.g.*, on a hot roller-mill, leaving a thermoplastic which softens at 100–110°.

C. HOLLINS.

Accelerator for vulcanisation of rubber. A. CAMBRON, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,754,010, 8.4.30. Appl., 20.1.26).—Aldehyde-arylamine accelerators which are hard and do not tend in powder form to agglomerate are obtained, without the use of excess aldehyde, by condensing the reagents in substantial absence of water in presence of acidic substances, *e.g.*, zinc chloride, hydrochloric or sulphuric acid, aluminium chloride, chlorodinitrobenzene, etc. Aniline or xyldine is thus condensed with acetaldehyde, butaldehyde, or crotonaldehyde.

C. HOLLINS.

Acceleration of vulcanisation. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,885, 11.4.29).—

Nitroaryl 2-benzthiazyl disulphides, obtainable from 2-thiolbenzthiazole and nitrochlorothiolbenzenes ($\text{NO}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{SCl}$), are vulcanisation accelerators having in marked degree the property of delayed action.

C. HOLLINS.

Preventing oxidation or retarding ageing of organic compounds [rubber, oils, soaps]. GOOD-YEAR TIRE & RUBBER CO., Assees. of J. TEPPEMA (B.P. 332,764, 6.8.29. U.S., 26.11.28).—Dihydroxydiaryl-methanes, *e.g.*, methylenedi- β -naphthol, are added to the rubber etc.

C. HOLLINS.

Inhibiting or reducing the perishing of artificial and natural varieties of rubber. I. G. FARBENIND. A.-G. (B.P. 318,865, 9.9.29. Ger., 10.9.28).—Indanephens (cf. B.P. 297,075; B., 1928, 845) or their salts or substitution products, *e.g.*, resorcyindane, are applied as anti-perishing agents in rubber mixtures, an advantageous proportion being 1–3 pts. per 100 pts. of rubber. The indanephens are relatively non-poisonous, and rubber containing them does not discolour in sunlight.

D. F. TWISS.

Coating rubber articles with metal, particularly artificial sets of teeth or the like. M. OW-ESCHINGEN (B.P. 311,766, 6.5.29. Austr., 16.5.28).—Rubber articles, particularly artificial dentures, are dipped in a solution of a reducing agent, *e.g.*, quinol, pyrocatechol, or pyrogallol, in a solvent, *e.g.*, ether-benzene, capable of swelling the rubber; the articles are then dried, the reducing agent is retained on and in the surface layer, thereby causing the deposition of metal on the rubber on subsequent immersion in a solution of a reducible metal such as silver or gold. A succession of such treatments may be necessary to obtain an adherent metallic layer which can be strengthened by electrodeposition.

D. F. TWISS.

Manufacture of rubber-like material. F. W. HULTMAN (B.P. 333,901, 18.2.29).—See U.S.P. 1,704,194; B., 1929, 368.

Preservation of rubber. J. TEPPEMA, Assr. to GOOD-YEAR TIRE & RUBBER CO. (U.S.P. 1,746,371, 11.2.30. Appl., 9.3.29).—See B.P. 281,616; B., 1929, 66.

Manufacture of [sponge-rubber] articles from dispersions of organic materials. DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, E. A. MURPHY, D. F. TWISS, and W. G. GORHAM (B.P. 333,952, 28.5.29).

Colloidal solutions and pastes (B.P. 332,557).—See III. Rubberised paint (U.S.P. 1,744,881).—See XIII.

XV.—LEATHER; GLUE.

Air-permeability of leather. R. S. EDWARDS (J. Soc. Leather Trades' Chem., 1930, 14, 392–409).—The air-permeability of different leathers was measured in two ways (methods *A* and *B*). In *A* the specimen was clamped to the end of a glass tube placed in series with a capillary tube of known dimensions and air drawn through them successively by means of a suction pump. The pressure at each end of the capillary was measured by means of tubes connected to vertical tubes dipping into a mercury trough. The volume of gas permeating the leather was calculated from Meyer's

formula for the flow of gas through the capillary tube and the permeability was calculated therefrom. In method *B* a convenient volume of gas was evacuated from an enclosure which was connected to a mercury column and also to a tube fitted with a tap, and at its end an ordinary rubber plunger. The latter was placed on the sample of leather, the tap opened, and the time noted for the pressure in the enclosure to increase to a certain value. Determinations by method *A* showed that patent leather was non-porous. The order of increasing permeability of other leathers was: stuffed vegetable-tanned upper leather, French sole leather, waterproofed English sole leather, heavy chrome-tanned upper leather, English sole leather, glacé kid, gorse calf, willow calf, box calf, vegetable-tanned calf (Russia). The permeability in the direction flesh to grain was about 2% greater than in the opposite direction. Comparison of the permeabilities of different parts of a chrome-tanned calfskin by method *B* gave widely varying results (20–110).

D. WOODROFFE.

See also A., Sept., 1118, **Swelling of gelatin in acid and salt solutions** (VON MORACZEWSKI and GRZYCKI). 1223, Kola tannins (CASPARIS and REBER).

PATENTS.

Leather-dressing process. O. L. STEVEN (U.S.P. 1,750,732, 18.3.30. Appl., 30.6.28. Ger., 22.7.27).—Synthetic tanning agents or ligninsulphonic acids are fixed in leather by soaking the leather in a solution of a salt of aniline, benzidine, or naphthylamine prior to finishing.

C. HOLLINS.

Production of artificial masses from casein. O. SCHMIDT, K. SEYDEL, and E. MEYER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,775,175, 9.9.30. Appl., 12.9.27. Ger., 27.8.26).—See B.P. 297,483; B., 1928, 869.

Manufacture [compacting and finishing] of [vegetable-tanned sole] leather. C. G. SHAW (B.P. 333,871, 19.2.29).

Artificial leather (B.P. 317,824).—See V. Preparation of leather cloth (B.P. 332,602).—See VI.

XVI.—AGRICULTURE.

Soil acidity. W. U. BEHRENS (Z. Pflanz. Düng., 1930, 18A, 5–44).—Existing methods for determining the degree of saturation of soils with bases are largely conventional, since the total adsorptive capacity of soils varies greatly with the concentration of the neutral salt solution with which it is treated. The value ($T-S$) is preferably determined by treating soil with 0.01N-sodium borate, the unadsorbed alkali being subsequently determined by titration. Although by this process the soil suspension is maintained at p_{H} 8.0–10.0, the adsorptive capacity of the soil is increased by about 3%. Relations between the p_{H} values of soil suspensions in water and in potassium chloride solution, the degree of salination, and exchange acidity are examined. With $V=0.5$, p_{H} (water) = 5.5, p_{H} (KCl) = 4.6, and exchange acidity = 0.2 c.c. of N-acid per 100 g. of soil. Treatment of neutral soils with potassium chloride solution causes an exchange of

potassium for aluminium, thereby producing a type of exchange acidity characterised by a decreased p_H and an increased titratable acidity in the solid phase. Hydrolytic acidity is determined from the amount of potassium hydroxide necessary to produce the same p_H in the soil suspension as is obtained by shaking soil with acetate solution, and values are compared with the amount of acetic acid liberated in this manner. In mineral soils the acid produced approaches the equivalent of the direct potash consumption, but in organic soils there is a deficit ascribed to the adsorption of acetic acid by the humus. Humus soils when shaken with neutral salt solutions liberate their maximum titratable acid very rapidly, and it is suggested that organic acids and aluminium salts exist normally in such soils in an adsorbed condition. Curves expressing relationships between the p_H and the buffer capacity of soils ($\Delta s / \Delta p_H$, where Δs = quantity of acid producing a change of Δp_H in a soil) show minima between p_H 5 and 6.5. In the acid area two components of the curve are differentiated, viz., buffering due to the liberation of "permutit acids" and that resulting from the dissolution of alumina from the silicate complex. Soils and added acid do not attain complete equilibrium within 3 days.

A. G. POLLARD.

Colorimetric determination of p_H values in alkaline soils. P. KAMERNAN (J. S. Afr. Chem. Inst., 1930, 13, 59—63).—Colorimetric methods for determining p_H values gave unsatisfactory results. Discrepancies are probably due to hydrolytic effects brought about by the increased water: soil ratio necessitated by the preparation of a filtered soil extract.

A. G. POLLARD.

Rapid colorimetric determination of citric-soluble phosphoric acid in soils. A. NĚMEC, J. LANIK, and A. KOPPOVA (Compt. rend., 1930, 191, 69—71).—The soil (10 g.) is stirred with 100 c.c. of a 1% solution of citric acid, and 5 c.c. of the solution, after filtering and centrifuging, are acidified and titrated with potassium permanganate solution at the b.p. Dilute hydrogen peroxide solution is added to destroy the excess of permanganate, and the solution is neutralised with ammonia, using dinitrophenol as indicator. Then 2 c.c. of ammonium molybdate solution and 0.3 c.c. of stannous chloride solution per mg. of P_2O_5 present are added, and the blue colour of the solution after dilution to 100 c.c. is compared with that given by a known amount of citric acid.

H. F. GILLBE.

Determination of the adsorption capacity [of soils]. S. N. ALESCHIN (Z. Pflanz. Düng., 1930, 18A, 44—48).—Modifications of the Bobko-Askinasi method are described. The soil is saturated with barium by shaking with barium chloride solution and then transferring to a filter and washing with more barium chloride solution until all calcium is removed. The soil is further washed with water until free from chloride. Adsorbed barium is determined by shaking the washed soil with excess of 0.05N-sulphuric acid (500 c.c. per 10 g. of soil used) and, after filtration, titrating the residual acid with soda. To ensure the soil being treated with exactly the same concentration of acid, the washed sample may be dried in an oven before treatment, or,

if used wet, the moisture must be allowed for. The washing out of the excess of barium chloride in the initial process may be replaced by precipitation with sulphuric acid.

A. G. POLLARD.

Determination of total carbonates in soils. A. N. PURI (Bull. Imp. Inst. Agric. Res., Pusa, 1930, No. 206, 7 pp.).—Ten g. of soil are stirred with 100 c.c. of water and excess (0.2—0.5 g.) of calcium sulphate is added. The mixture is heated to boiling, 10 c.c. of 0.1N-aluminium chloride are added, and the mixture is shaken. About 10 drops of a 1% alcoholic solution of bromothymol-blue are added and, after vigorous shaking, the suspension is allowed to settle. A yellow colour in the supernatant liquid indicates < 1% of carbonates in the soil; a green to blue colour, above 1% of carbonates. The colour is again noted after the further addition of 10 drops of 1% alcoholic solution of bromocresol-green. When no carbonate is present the colour is golden-yellow; with < 1%, green; and with > 1%, deep blue-green. The suspension is again heated to boiling and titrated with 0.5N-sulphuric acid, boiling being continued for 2 min. after each addition of acid. Complete decomposition of carbonates is indicated by a golden-yellow colour which persists after boiling and allowing the suspension to settle.

A. G. POLLARD.

Silage investigations at Bangalore. T. S. KRISHNAN (Mem. Dept. Agric. India, 1930, 10, 237—259).—Chemical changes occurring during the ensilage of *Sorghum vulgare* are examined. Protein changes resemble those occurring under European conditions. A high ratio of volatile base: amino-acid is possible without deterioration of the silage. Butyric acid was absent from all samples examined. During ensilage there is a considerable loss of phosphate and of some calcium in the drainage, together with smaller amounts of chloride, alkalis, and magnesium. The sulphate content of the silage increased, presumably as a result of the oxidation of protein-sulphur.

A. G. POLLARD.

Chemical method for determining fertiliser requirements [of soils] and the action of phosphatic fertilisers. A. NĚMEC (Z. Pflanz. Düng., 1930, 18A, 48—56).—Lack of agreement between field trials and laboratory tests of the phosphate requirement of soils is attributed to interaction between soil and fertiliser in the field, which is not adequately represented in laboratory tests. The efficiency of phosphate fertilisers is affected by the nature and proportions of soluble cations in the soil. In soils containing more than 50 mg. of citric-soluble iron per 100 g. of soil, phosphate fertilisers may not exert their full effect on crop yields. This effect is most marked in soils originally poor in phosphate. The author's method for determining phosphate requirements (B., 1929, 420, 938) is supplemented by a determination of iron soluble in 1% citric acid.

A. G. POLLARD.

The mixed fertiliser "Nitrophoska." W. WERNER (Z. Pflanz. Düng., 1930, 9B, 339—360).—Pot cultures with a number of crops show that in sandy soils of small adsorptive power, "Nitrophoska," used in conjunction with lime, may cause plant injury by the liberation of free ammonia, and actual losses of nitrogen may occur

where heavy dressings of lime are applied. In weakly acid sandy soils the water-soluble phosphate in Nitrophoska is transformed into a relatively unassimilable form (probably iron or aluminium phosphate). In such soils superphosphate was much the more efficient, but on very acid soils the superiority of superphosphate was not observed. On loams Nitrophoska gave better crop yields than superphosphate and, in general, may be recommended for use on weakly acid to neutral soils.

A. G. POLLARD.

Field manurial trials with Nitrophoska. F. HONCAMP and H. WIESSMANN (Z. Pflanz. Düng., 1930, 9B, 361—363).—In field trials with potatoes and roots Nitrophoska proved as efficient as did corresponding mixtures of the simple fertilisers.

A. G. POLLARD.

"Huminit." K. ECKL (Z. Pflanz. Düng., 1930, 9B, 378—382).—In fertiliser trials the addition of "Huminit" (cf. Densch., B., 1929, 532) to farmyard manure did not increase its crop-producing power.

A. G. POLLARD.

Modern pasture management and nitrogen manuring. MÖLLER (Z. Pflanz. Düng., 1930, 9B, 363—377).—Results of meat and milk trials on the intensive grazing system are recorded. The efficiency of nitrogenous fertilisers in this practice is largely dependent on weather conditions. Tables of costs and returns are given for a number of fertilisers.

A. G. POLLARD.

Effect of rye and vetch green manures on the microflora, nitrates, and hydrogen-ion concentration of two acid and neutralised soils. N. R. SMITH and H. HUMFELD (J. Agric. Res., 1930, 41, 97—123).—On both acid and neutral soils green manuring increased the number of micro-organisms developing on soil extract-agar plates. Maximum numbers were reached in 4—5 days and subsequently decreased rapidly as the leafy portions of the manure disappeared. On soils neutralised with chalk, but not on acid soils, a later increase in bacterial numbers occurred, this phenomena being characteristic of the first application of green manure only. The number of fungi appearing in acid and neutral soils was not affected by green manuring. Nitrate accumulation was increased by manuring. A slightly increased acidity coincided with nitrate production in unlimed soils, the effect being more marked in sandy than in clay soils.

A. G. POLLARD.

Control of the stalk borer in maize. L. B. RIPLEY and G. A. HEPBURN (Farming in S. Africa, 1929, 4, 353—354).—Cryolite (1 in 600, suspension) is an effective stomach poison; sodium chromium fluoride is less poisonous.

CHEMICAL ABSTRACTS.

Use of sodium chlorate in the control of Johnson grass. H. J. HARPER (J. Amer. Soc. Agron., 1930, 22, 417—422).—Sulphuric acid is not so effective as sodium chlorate. Soils treated with sodium chlorate have a lower nitrifying power than untreated soils.

CHEMICAL ABSTRACTS.

Determination of the quantity of oil retained by citrus foliage after spraying. L. L. ENGLISH (J. Agric. Res., 1930, 41, 131—133).—As soon as the oil spray on foliage has dried, 50 sample leaves are collected from various parts of the tree and a disc 10 cm.² in area is punched from each leaf. The discs are shaken with

50 c.c. of ether for 1 min. and the extract is filtered. A second extract is made and the combined solutions are evaporated to 20—25 c.c. and placed in a Babcock skim-milk bottle with 5 c.c. of approx. 0.5*N*-sulphuric acid. Residual ether is removed by heating to 50°, the temperature slowly raised to 80°, and sufficient hot acid added to bring the oil into the capillary. The bottle is centrifuged in a heated centrifuge and the oil volume measured. Repeated heating and centrifuging may be necessary to produce complete separation of the oil.

A. G. POLLARD.

Petroleum oil for spraying. DE ONG.—See II.

See also A., Sept., 1109, **Calcium hydroxide absorption by hydrated silica** (SHAW and MACINTYRE).

PATENTS.

Production of materials containing phosphorus and nitrogen suitable for use as fertilisers. HYDRO NITRO Soc. ANON. (B.P. 333,477, 19.11.29. Switz., 1.12.28).—Phosphorus pentoxide is heated under pressure in an autoclave with ammonium carbamate or with ammonia and carbonic acid, yielding a mixture of urea and ammonium phosphates.

L. A. COLES.

Derivatives of aromatic diazo compound (B.P. 309,610).—See IV. **Calcium cyanamide** (B.P. 333,353).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Colloids [cane wax] in granulated sugar. C. F. BARDORF (Ind. Eng. Chem., 1930, 22, 907).—The claim that certain granulated sugars contained colloids (highly dispersed cane wax) capable of coagulating the flavouring extract used in the preparation of beverages was examined. Macroscopical inspection of the sugar crystals, observation of a thick layer of a 50% solution, and a percolation test were made and the results compared with those given by a flocculation test. No conclusions could be drawn as the sugars of best quality from the refiner's point of view were often the most unsatisfactory as regards the coagulation. It is considered significant that the most severe flocculation was in all cases produced by sugars refined from Natal raws.

H. J. DOWDEN.

Effect of heat on acacia. GABEL.—See XX.

See also A., Sept., 1151, **Measurement of turbidity** (INGERSOLL). 1167, **Colorimetric determination of starch** (PALOHEIMO). **Separation of components of potato starch** (BALDWIN).

XVIII.—FERMENTATION INDUSTRIES.

Determination of [ethyl] alcohol [by chromic oxidation]. L. SEMICHON and M. FLANZY (Ann. Falsif., 1930, 23, 347—349; cf. B., 1929, 449).—The authors acknowledge the priority of Martin, but maintain that they have introduced important refinements, viz., separate oxidation and distillation operations, different concentration for the oxidising agent, and oxidation in the cold.

H. J. DOWDEN.

Chemico-bromotological studies on vinegars. U. PRATOLONGO [with, in part, M. P. ALLAN] (Giorn. Chim. Ind. Appl., 1930, 12, 335—340).—Italian legislation of October, 1925, forbids the use as food products of

synthetic or distilled vinegar, and the coloration of alcohol vinegar or of wine vinegar, even with colouring matters derived from the grape. Fermentation vinegar is distinguishable from artificial vinegar by the iodine value, which is about $38.6 \pm 10\%$ for the former and about 2 for the latter, and by the luminescence of the former when exposed to ultra-violet light. Marc vinegar differs from genuine wine vinegar in having a lower alcohol + acetic acid content, a lower extract, a lower ash, and a less pronounced alkalinity of the ash. Marc vinegars may be distinguished from diluted wine vinegars by their higher ash content, by the higher alkalinity of the ash, by the greater value of the ratio between ash and extract, and by the lower content of alcohol + acetic acid in relation to the fixed acidity. Methods for detecting artificial coloration of vinegars are discussed. Addition of strong mineral acid to vinegar may be detected by colorimetric determination of the p_H value, using thymol-blue as indicator. With alcohol vinegar, the p_H is diminished from 2.4 to 1.8 or 1.2 by the presence of 0.1 or 0.2% of sulphuric acid, and that of wine vinegar is similarly lowered from 2.4 to 2.2, or 2.0, or 1.8 by the presence of 0.1, 0.2, or 0.5% of the acid.

T. H. POPE.

PATENTS.

Continuous process for preparing *n*-butyl alcohol and acetone by fermentation. COMMERCIAL SOLVENTS CORP. (B.P. 306,138, 11.2.29. U.S., 17.2.28).—Sterile mash is introduced into the first of a number of fermenters which are connected in series and are preferably located on descending levels. After inoculation of the mash with a butyl alcohol-acetone bacillus, further additions of sterile mash to the first fermenter cause the partially fermented mash to flow from the first fermenter through the others until all are filled. The influx of fermenting mash is prevented in the last fermenter, and a definite proportion of the mash therein is removed. Fresh sterile mash equivalent in volume to that withdrawn is then added to the first fermenter.

C. RANKEN.

Production of acetone and butyl alcohol by fermentation. G. W. FREIBERG (U.S.P. 1,744,958, 28.1.30. Appl., 14.4.27).—After the separation and recovery of the germ portion of steeped maize grain, the residual wet carbohydrate-containing portion of the grain is mashed and subsequently fermented by micro-organisms.

C. RANKEN.

Manufacture of baking yeast. H. CLAASSEN (U.S.P. 1,774,546, 2.9.30. Appl., 6.10.27. Ger., 19.11.26).—See B.P. 280,861; B., 1928, 686.

XIX.—FOODS.

Determination of cream of tartar and tartaric acid in tartrate baking powders. B. G. HARTMANN (J. Assoc. Off. Agric. Chem., 1930, 13, 385—389).—To 2.5 g. of baking powder are added 100 c.c. of distilled water at 50°. After keeping for 30 min. with occasional shaking the solution is diluted to 250 c.c. and filtered. Two 100-c.c. portions of filtrate are each evaporated to 20 c.c. To one portion are added 3.5 c.c. of *N*-potassium hydroxide, 2 c.c. of glacial acetic acid, and 80 c.c. of 95% alcohol. The second portion is treated similarly

except that sodium hydroxide is substituted for the potassium compound. The two solutions are placed in a refrigerator for 1 hr., stirred vigorously, and allowed to remain in the refrigerator overnight. The precipitates are collected in Gooch crucibles, washed first with ice-cold 80% alcohol and finally with cold alcohol, treated with hot water, and titrated with 0.1*N*-alkali, using phenolphthalein as indicator. Then total tartaric acid (%) = $1.5A$; cream of tartar (%) = $1.88B$; tartaric acid (%) = $1.5(A-B)$, where A = titration of portion treated with potash and B that with soda. Free tartaric acid may be determined directly by extracting 1.25 g. of baking powder with 50 c.c. of chloroform in a 200-c.c. graduated flask for 5 min. 100 C.c. of alcohol saturated with cream of tartar (free from tartaric acid) are added. After 30 min. the liquid is diluted to 200 c.c. with alcohol, shaken, filtered, and 100 c.c. of the filtrate are titrated with 0.1*N*-alkali, using phenolphthalein. Tartaric acid (%) = $1.2 \times \text{alkali consumption}$.

A. G. POLLARD.

Limits of error of the Babcock test for cream. W. H. MARTIN, A. C. FAY, and K. M. RENNER (J. Agric. Res., 1930, 41, 147—159).—The results of single tests on 37—40% cream were not closer than ± 0.44 to $\pm 0.55\%$ of fat, nor the averages of duplicates than ± 0.31 to $\pm 0.39\%$ of fat.

A. G. POLLARD.

Crystallisation of lactose in sweetened condensed milk. B. SEGAL (J. S. Afr. Chem. Inst., 1930, 13, 33—40).—The texture of sweetened condensed milk is a function of the size of the lactose crystals. The cooling of condensed milk during manufacture should aim at the immediate production of a large number of crystal nuclei rather than the growth of crystals round a small number of centres.

A. G. POLLARD.

Pure butters and butters adulterated [with coconut oil]. L. HOTON (Ann. Falsif., 1930, 23, 324—337).—The difficulties of detecting adulteration of butter with coconut oil, even to the extent of 20%, are emphasised. The Polenske determination is considered to be of little value, and the variability of the results according to the conditions, character of the distillation, etc. is illustrated. Pure butters have given the same ratio of insoluble to soluble fatty acids as those containing 20% of coconut oil. The differences in the solubilities in dilute alcohol of the fatty acids in butter, coconut oil, and margarine are also valueless. The observation that, when heated, the fatty acids in butter increase in weight owing to oxidation, whilst those in coconut oil lose weight by volatilisation, has been shown to be incorrect. Using samples of pure butter and butter containing 20% of coconut oil, it was found that loss of weight occurred in both cases, accompanied by a rise in the refractive index (n). The method previously described (cf. B., 1929, 129) has been applied to some 60 samples of butter of different origin, and the values of n for the pure butter and that after addition of 10% of coconut oil are tabulated. With the pure butters the maximum and minimum values were 26 and 19, respectively, whilst with the adulterated samples the values were 1—3 units lower. An attempt has been made to correlate the refractive index of the insoluble fatty acids with the amounts of soluble and insoluble fatty acids present, but certain anomalous

cases were found. The test, however, affords additional evidence in the detection of coconut oil in butter.

H. J. DOWDEN.

Acid to sugar ratio in oranges. D. J. R. VAN WIJK (J. S. Afr. Chem. Inst., 1930, 13, 52—58).—Examination of three typical varieties of oranges shows that maturity as commonly judged from the ratio, acid: degrees Brix of the juice, is unsatisfactory. A better basis of comparison is the ratio acid: "calculated sugar" (where calculated sugar = degrees Brix — acid — 1.75). The value 1.75 represents the contribution to the density of the juice, of extraneous matter other than acid and sugar, and is fairly constant for all samples examined.

A. G. POLLARD.

Vitamins in dried fruits. II. Effect of drying and of sulphur dioxide on vitamin-A content of fruits. A. F. MORGAN and A. FIELD (J. Biol. Chem., 1930, 88, 9—25; cf. A., 1929, 960).—The maximum retention of vitamin-A during the drying of peaches, prunes, and apricots is obtained when the fruit is treated with sulphur dioxide and subsequently dehydrated at about 72°; in the absence of sulphur dioxide, however, sun-drying is less destructive to vitamin-A than dehydration at the higher temperature. Lye dipping of prunes had no significant effect on their vitamin-A content. The stability of the vitamin-A of the three fruits towards drying decreases in the order mentioned; nevertheless, the absolute vitamin-A content of dried apricots is higher than that of the fresh peaches, and is comparable with that of spinach, egg yolk, and butter.

C. R. HARRINGTON.

Identification of flavouring constituents of commercial flavours. I. Optical properties of the semicarbazones of certain aldehydes and ketones. J. B. WILSON and G. L. KEENAN (J. Assoc. Off. Agric. Chem., 1930, 13, 389—397).—Details are given for the preparation of the semicarbazones of typical aldehydes and ketones present in flavourings. Chemical and optical properties suitable for identification are recorded together with photomicrographs of characteristic semicarbazones.

A. G. POLLARD.

Frying oil in fish preserved in oil. G. HINARD (Ann. Falsif., 1930, 23, 344—347).—When canned fish, e.g., sardines, are cooked in a vegetable oil, e.g., arachis, which differs from that in which they are subsequently packed (olive oil), it is difficult to determine the extent to which the packing oil is contaminated by the cooking oil. Examination of a sample of sprats which had been cooked in a highly refined mineral oil and afterwards packed in olive oil showed that the filling oil contained about 5% and the fish itself about 1% of the cooking oil.

H. J. DOWDEN.

Colloids in granulated sugar. BARDORF.—See XVII. **Vinegars.** PRATOLONGO.—See XVIII.

PATENTS.

Refrigerating apparatus for foodstuffs and the like. V. C. FROM, C. D. ROWLEY, and A. W. LARSKY (B.P. 334,027, 26.7.29).

Treatment of foods [with solar ultra-violet rays]. SOLAR RES. CORP. (B.P. 310,802, 26.4.29. U.S., 30.4.28).

Vitamin compound (U.S.P. 1,753,531).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Effect of stabilisers in lard in relation to its use in ointment of potassium iodide, N.F.V. W. J. HUSA (J. Amer. Pharm. Assoc., 1930, 19, 825—828).—Deterioration, measured by the amount of free iodine liberated from potassium iodide, occurs much more rapidly in plain leaf lard than in lard benzoinated by the U.S.P. method. The presence of benzoic acid retards the rate of absorption of iodine at the ethylenic linkings. Although quinone reduces the rate of deterioration of lard, it is not suitable for use as a stabiliser in ointment of potassium iodide, N.F.V., on account of the grey-brown colour which develops; similarly, the presence of benzoic acid in the lard causes a grey colour to develop in the ointment made from it.

E. H. SHARPLES.

Effect of heat on acacia. L. F. GABEL (J. Amer. Pharm. Assoc., 1930, 19, 828—830).—Prolonged heating of powdered acacia increases the viscosity of the mucilage. Maximum viscosity is obtained by heating at 100° for 3 days. Continued heating of normal mucilage of acacia causes no increase in the viscosity. The formation of the thick mucilage from heated acacia is probably due to the production of metarabic acid because treatment of the powdered gum with alcoholic sulphuric acid has a similar effect.

E. H. SHARPLES.

Physiological potency of commercial ergot preparations. W. T. McCLOSKEY, M. R. THOMPSON, and N. G. BARBELLA (J. Amer. Pharm. Assoc., 1930, 19, 844—847).—Examination of 79 commercial samples of fluid extract of ergot, assayed by the U.S.P. cock's comb method, showed that the U.S.P. X. fluid extract is the most satisfactory, apart from those which contain a specified amount of ergotamine or ergotoxine in suitable solution. Fluid extract of ergot should be packed in well-filled, small-sized containers, the label on which should bear an expiry date.

E. H. SHARPLES.

Phytochemistry of centaury. B. GAAL (Ber. Ungar. pharm. Ges., 1929, 5, 58—72; Chem. Zentr., 1929, i, 1583—1584).—The drug gave 3.18% of green, unctuous extract with light petroleum. The acid value was 25.24—25.9, the saponif. value 101.63—101.54, unsaponifiable fatty acids 13.02%, water-insoluble fatty acids 47.15%, free acid 14.36%. Bromination of the fatty acids gave α -linolenic acid hexabromide and oleic acid dibromide. Reduction of the first yielded an acid with an odour of cod-liver oil, the iodine value differing from that of linolenic acid; reduction of the second gave oleic acid. The solid fatty acids gave four fractions with m.p. between 62° and 72°. The fractions contained cerotic, stearic, and palmitic acids. The products obtained from the unsaponifiable portion of the main extract are described.

L. S. THEOBALD.

Leaf oils of Washington conifers. I. Introduction. A. J. LEHMAN and E. V. LYNN (J. Amer. Pharm. Assoc., 1930, 19, 840—843).—Published results on the chemical and physical constants of 8 species of conifers grown in Washington are reviewed.

E. H. SHARPLES.

Extraction of orange oil. F. J. DE VILLIERS (J. S. Afr. Chem. Inst., 1930, 13, 48—52).—Steam-distillation of orange peel causes some decomposition of the essential oil unless carried out in high vacuum. Alcohol proved the best solvent for extraction, the peel being rasped

under alcohol and prolonged contact avoided. Addition of water to the extract causes the separation of a fine emulsion difficult to separate directly by centrifuging. The particles of the emulsion are negatively charged and separation is effected by the addition of electrolytes. The absorption of ions by the emulsion was in the order $\text{Fe}^{++} > \text{Ca}^{++} > \text{Na}^{+}$, but the most effective coagulant was the hydrogen ion. A. G. POLLARD.

Detection of isopropyl alcohol. WÜHRER.—See III.

See also A., Sept., 1108, **Solubility of pharmaceutical preparations in glycerol** (BIRZA). 1177, **Action of ammonia and dimethylamine on ethylene, allylbenzene, and phenylcyclohexene oxides, and their homologues** (LÉVY and SFRAS). 1194, **Yohimbe alkaloids** (HAHN and SCHUCH). 1220, **Purification of toxins and antitoxin** (LINDERSTRÖM-LANG and SCHMIDT). 1221, **Purification of carotene** (JAVILLIER and EMERIQUE). 1222, **Isolation of vitamin-B₁** (WILLIAMS and others).

PATENTS.

Manufacture of spinal anæsthetic solutions. H. A. METZ LABS., INC., Assees. of G. P. PITKIN and C. W. HOOPER (B.P. 311,382, 9.5.29. U.S., 10.5.28).—Gliadin (0.03–0.06%) in alcohol solution is added to a spinal anæsthetic solution, e.g., β -diethylaminoethyl *p*-aminobenzoate hydrochloride (5–10%). The gliadin is precipitated on contact with the spinal fluid as a semi-permeable membrane; the anæsthetic floats in globules surrounded by this membrane, and is absorbed by the intradural nerve trunks. Cereal mucilage, without alcohol, may be used in place of gliadin. C. HOLLINS.

Rapid preparation of an antiseptic liquid. G. PANSINI (B.P. 332,520, 17.1.29).—Sodium chloride solution is electrolysed at 40°, using a high anode current density to obtain a hypochlorite solution saturated with ozone. [Stat. ref.] A. R. POWELL.

Vitamin compound. R. K. PRINCE, Assr. to VITAMIN FOOD CO., INC. (U.S.P. 1,753,531, 8.4.30. Appl., 2.1.25. Renewed 8.10.28).—A preparation containing vitamins-A, -B, and -C, calcium lactate, tricalcium phosphate, sea-salt, and endocrine gland substance is physiologically more active than are the separate ingredients; e.g., a mixture is made of 1.5% of orchitic substance, 5.6% of dehydrated citrus juices, 0.20% of aqueous 1% sodium iodide, 11.7% of tricalcium phosphate, 16.8% of dehydrated autolysed yeast extract, and 64.2% of milk powder. C. HOLLINS.

Manufacture of hormones from urine. SCHERING-KAHLBAUM A.-G. (B.P. 310,056, 20.4.29. Ger., 21.4.28).—Ovarial hormone and that of the anterior lobe of the pituitary body are adsorbed from the concentrated urine of pregnant women by means of animal charcoal or silica gel. Extraction of the adsorbent material by water-insoluble solvent (chloroform, ether) yields the ovarian hormone, and the pituitary hormone is extracted from the residue with much water. The urine may be freed from ovarian hormone by a preliminary extraction with ether or chloroform. C. HOLLINS.

Preparation of physiologically active substances from the anterior lobes of the hypophysis. I. G. FARBERIND. A.-G. (B.P. 316,882, 1.8.29. Ger., 4.8.28. Addn. to B.P. 291,018; B., 1930, 302).—Improved

and more concentrated aqueous extracts are obtained by subjecting the glands, after extraction of oestrus-retarding constituents with an organic solvent, to autolysis for several days at 20–25°, especially with papain. The albumin degradation products may be precipitated by adding acetone to the aqueous extract, and the active substance, after removal of acetone, is extracted from the filtrate with ether. C. HOLLINS.

Products for destroying animals. F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 332,797, 2.10.29. Ger., 19.9.28).—Urethanes of the type, $\text{NR}_2 \cdot \text{CO} \cdot \text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{NR}'_2$, in which R is alkyl or hydrogen and R' is alkyl, are strong poisons, but are unstable in pastry or other alkaline-reacting bait. They are stabilised by the addition of acids or of buffer salts; e.g., the methylcarbamate ester of dimethyl-*m*-aminophenol methiodide is added to the bait with tartaric acid. C. HOLLINS.

Preparation of lobelia alkaloids. A. BOEHRINGER (B.P. 314,532, 24.6.29. Ger., 29.6.28).—Lobelanidine [2:6-di-(β -hydroxy- β -phenylethyl)methylaniline], which is therapeutically of little value, is converted by oxidation with sulphuric acid and permanganate into the keto-alcohol, lobeline, and the diketone, lobelanine, both of which are therapeutically useful. The diketone may be reduced to give a separable mixture of lobelanidine and lobeline. *dl*-Lobeline, m.p. 104–106°, may be resolved by means of tartaric acid. *l*-Lobeline tartrate, m.p. 70°, *l*-lobeline sodium tartrate, m.p. 180°, and *l*-lobeline, m.p. 130–131°, $[\alpha]_D^{25}$ —38.5°, are described. C. HOLLINS.

Preservation of the aroma of tobacco and tobacco goods. G. SENFTNER (B.P. 333,826, 7.11.29. Ger., 9.11.28).—The tobacco etc. is enclosed in materials which have been pretreated with photodynamically-active, light-accumulating substances such as ergosterol, cholesterol, eosin, methylene-blue. The preservative effect may be enhanced by exposing the treated wrapping materials to the action of chemically active rays, such as ultra-violet light. E. H. SHARPLES.

Manufacture of [therapeutic] stable super-saturated calcium glutaconate solutions. CHEM. FABR. VORM. SANDOZ (B.P. 332,840, 25.10.29. Ger., 29.10.28. Addn. to B.P. 314,460; B., 1929, 698).—Therapeutically active substances for injection are added to hot calcium glutaconate solutions, which are then sealed in ampullæ and stabilised by one or more heatings at 50–100°. Examples of added substances are sodium chloride or salicylate, other glutaconates, alkaloids, etc. C. HOLLINS.

Stable and sterilised solutions of salts of dialkyl-aminoarylphosphinous acids. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 332,926, 30.4.29).—After addition of an acid-binding agent (sodium carbonate), solutions of salts of *p*-dialkylaminoarylphosphinous acids may be sterilised at 100° without decomposition and separation of oil. C. HOLLINS.

Preparation of the sodium salt of *p*-oxyphenyl-arsinic [*p*-hydroxybenzenearsinic] acid. UNION CHIM. BELGE SOC. ANON. (B.P. 333,023, 5.7.29. Belg., 7.6.29).—*p*-Hydroxybenzenearsinic acid is precipitated as calcium salt, which is filtered off, washed with hot ammonia solution, and converted into sodium salt by boiling with sodium carbonate solution. C. HOLLINS.

Preparation of organo-metallic [arsenic and antimony] compounds. H. J. BARBER, and MAY & BAKER, LTD. (B.P. 331,869, 8.3.29).—An arsinic or stibinic acid is condensed with 4 mols. of an aliphatic or aromatic thiol compound, *e.g.*, thioglycollic acid: $R \cdot AsO_3H + 4R'SH \rightarrow R \cdot As(SR')_3 + R'S_3 + 3H_2O$. Carboxymethyl esters of dithioarsenous acids are thus obtained from 8-acetamido-3-hydroxybenz-1:4-oxazine-6-arsinic acid, anilinoacetamido-*p*-arsinic acid, and 3-acetamido-6-hydroxybenzenearsinic acid with thioglycollic acid. Corresponding products are prepared from 3-amino-4-hydroxybenzenearsinic acid and thioglycollamide or cysteine, arsanilic acid and ethyl glycollate, 8-acetamido-3-hydroxybenz-1:4-oxazine-6-arsinic acid and β -thioethyl alcohol, and stibanilic acid or 4-chlorobenzenestibinic acid and thioglycollamide. C. HOLLINS.

Manufacture of 1:8-cineole (eucalyptol). RHEINISCHE KAMPFER FABR. G.M.B.H. (B.P. 317,757, 8.8.29. Ger., 21.8.28).—The mixture (45% of terpenes, 22% each of 1:4- and 1:8-cineoles) obtained by dehydration of terpin hydrate, terpineol, etc. is cooled to -15° and treated with 60% sulphuric acid, whereby the 1:4-cineole is hydrated to terpineol or terpin hydrate. On steam-distillation an oil is obtained from which pure 1:8-cineole, m.p. $0-1^\circ$, may be separated by fractional distillation and freezing; the residue is worked up for terpineol. C. HOLLINS.

Preparation of solutions of medicaments. C. L. LAUTENSCHLÄGER, M. BOCKMÜHL, and R. SCHWABE, ASSRS. to WINTHROP CHEM. CO. (U.S.P. 1,752,305, 1.4.30. Appl., 5.4.26. Ger., 21.4.25).—See G.P. 446,290; B., 1928, 623.

Production of nutritive substances [for absorption through the skin]. K. STEJSKAL (U.S.P. 1,774,771, 2.9.30. Appl., 21.4.27. Austr., 17.5.26).—See B.P. 271,120; B., 1929, 113.

Stable and sterilised solution of salts of *p*-dialkyl-aminoarylphosphinous acids. L. BENDA, ASSR. to WINTHROP CHEM. CO., INC. (U.S.P. 1,758,425, 13.5.30. Appl., 4.1.29. Ger., 29.3.28).—See B.P. 332,926; preceding.

cycloHexene aldehydes and carboxylic acids (B.P. 309,911).—See III. **Inhalants** (B.P. 333,520).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Antifogging agents in [photographic] developers. A. P. H. TRIVELLI and E. C. JENSEN (J. Franklin Inst., 1930, 210, 287—309).—The antifogging action of 6-nitrobenziminazole as a typical agent when added to the developer instead of to the photographic emulsion is investigated. With quinol, pyrogallol, and particularly with *p*-aminophenol developers, 6-nitrobenziminazole is superior to potassium bromide or iodide.

[With G. SILBERSTEIN.] Determinations of resolving power showed that with negative film 6-nitrobenziminazole does not, in general, give as good results as those given by potassium bromide, but this may possibly be remedied by using lower concentrations.

J. R. I. HEPBURN.

Reproducing coke sections. ZIPPERER and LORENZ.—See II.

See also A., Sept., 1136, **Action of light on silver bromide** (CHATTERJI and MACMAHON).

PATENTS.

Photographic chemical process and material. [Production of diazotypes.] O. A. HALL, ASSR. to BROWN LINE PROCESS CO. (U.S.P. 1,752,174, 25.3.30. Appl., 17.5.27).—A solution of pyridine in dilute sulphuric acid is treated with sodium nitrite at 0° , and there are then added in succession a solution of 1:2:6-aminonaphtholsulphonic acid and tartaric acid, a solution of tartar emetic, and a solution of nickel sulphate, the mixture being kept in the dark. The mixture is used for sensitising paper, and after exposure a purple image, becoming brown in the air, is developed with ammonia gas. C. HOLLINS.

Development of [diazotype] photo-prints. M., G., H., and A. RENKER (DURENER FABR. PRÄPARIERTER PAPIERE RENKER & Co.) (B.P. 305,104, 30.1.29. Ger., 30.1.28).—Running of colour and warping of the paper in the diazotype development are minimised by flooding the sensitised side of the paper with a large quantity of the developing liquid (ammonia solution), which is immediately removed by blowing with warm air. [Stat. ref.] C. HOLLINS.

XXII.—EXPLOSIVES; MATCHES.

Explosive mixtures of benzene and petroleum. ANON.—See II. **Explosive decomposition of acetylene.** ANON.—See III.

See also A., Sept., 1152, **Determination of ignition temperature of powder substances** (HARRISON).

PATENTS.

Blasting detonators. HERCULES POWDER CO., ASSEES. of (A) E. M. SYMMES, (B) K. F. PAUL (B.P. 333,534 and 333,539, 13.5.29. U.S., 13.10.28).—(A) A mixture of diazodinitrophenol (20—80%) and lead azide (80—20%) is used for the priming charge of detonators, being pressed on top of a secondary charge of tetryl, trinitrotoluene, or picric acid. (B) Diazodinitrophenol is used alone, or in admixture with oxidising salts, such as perchlorates, in the primer charge, and may also form part of the secondary charge. W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of effluents by activated sludge; laboratory results with an effluent from Oviedo. L. VALDÉS (Anal. Fís. Quím., 1930, 28, 932—943).—A sample of effluent was mixed with 20% by vol. of activated sludge and aerated at the rate of 10 litres of air per litre for 6 hrs. The resulting water was clear and unpolluted, and contained organic matter corresponding with 8.3 mg. per litre of oxygen as compared with 120 mg. for the untreated water; the biochemical oxygen demand fell from 322 to 10 mg. per litre and the organic nitrogen content from 25 to 3 mg. per litre. The water was rendered completely sterile by treatment with 2 mg. of chlorine per litre. The activated sludge used contained 3.08% of solids, of which the composition was: inorganic compounds 48.3%, organic compounds

51.7%, total nitrogen 4.78%, and phosphoric acid 3.02%.
H. F. GILLBE.

Oxygen-production process of a new "chemical" gas mask. A. HLOCH (Z. angew. Chem., 1930, 43, 732—734).—The oxygen is produced at the rate of 2.5 litres/min. by the regulated decomposition of potassium chlorate which, together with a catalyst to induce decomposition at 300° and a substance to modify the rate thereof, is made into a rod-shaped briquette which is ignited at one end immediately before use. In the gas mask apparatus provision is made for absorption of the carbon dioxide and moisture expelled and for mixing the residual nitrogen with the oxygen produced in the generator. The new briquettes have the advantage over sodium peroxide that the heat generated is less and there is no period of induction before a regular and uniform stream of oxygen is evolved.

A. R. POWELL.

Economic aspects of water purification. E. GROSS (Gas- u. Wasserfach, 1930, 73, 601—606).—In erecting a water purification system the possibility of bringing a purer water from a distance instead of taking an impure water closer at hand should be considered; the critical distance within which such a procedure would be advantageous is calculated as a function of the cost of purification, quantity of water delivered, etc. If the water is acid or contains iron the purifying plant should be erected near the source in order to minimise corrosion or the formation of deposits within the conduits. If possible the plant should be erected at a suitable height above the points to be supplied so that the water need be elevated only once. Some modern developments in water purification which have resulted in decreased consumption of coagulants, or have otherwise increased the efficiency of the plant, e.g., the use of the Bamag-Meguïn band sieve for the preliminary filtration, the introduction of the Dorr mixer, of the Dorr clarifier, and of rapid filters, the exact control of chlorination, etc., are briefly discussed.

A. B. MANNING.

See also A., Sept., 1143, **Determination of oxygen in sea-water** (NICLOUX).

PATENTS.

Separation or settlement from sewage and other liquids, of matters held in suspension therein. J. A. COOMBS, and ACTIVATED SLUDGE, LTD. (B.P. 333,974, 5.6.29 and 3.3.30).—A design of sedimentation tank is claimed in which the influent is delivered through a hollow beam which supports in the centre of the tank a baffled inlet surrounded by a guard chamber in which eddies may be dissipated or a rotatory motion given to the incoming liquor by means of suitably placed baffles. The tanks, which may be either square or circular in plan, have a slightly convex bottom, and are fitted with revolving scrapers which sweep the deposited sludge into pockets situated either at the corners or around the outside edge beyond the orbit of the scraper arm. The effluent is discharged over weirs into a peripheral channel and the sludge is delivered by hydraulic head or air-lift into a similar channel surrounding the first. Perforated baffles, reaching nearly to the bottom of the tank and parallel with the overflow weirs, form a still pool around its outer edge, and so

permit the settlement of the lightest particles of suspended matter. Detritus etc. may be collected in a central pocket below the inlet and may be separately removed and dealt with.
C. JEPSON.

Preparation of fumigants, inhalants, and the like. T. W. F. CLARK (B.P. 333,520, 11.5.29).—By fireproofing the active agent, e.g., with sodium tungstate, its loss is minimised in cases where its efficient distribution depends on the heat produced by the combustion of some combustible material mixed therewith and with potassium nitrate.
C. JEPSON.

Manufacture of insecticides and the like. J. GUILLISSEN, and UNION CHIM. BELGE SOC. ANON. (B.P. 333,743, 19.8.29).—The active insecticidal principle of the pyrethrum (*Pyrethrum cinereafolium*), obtained by solvent extraction of the flowers and stalks, is dissolved in pyridine or ethyl lactate and the solution is used as an insecticide.
E. H. SHARPLES.

Preparations for exterminating rats, mice, and like vermin. C. BALL (B.P. 333,216 and 333,218, 6.5.29).—The preparations (A) comprise a suspension in an oily or fatty vehicle (e.g., "benzoated" lard) of $\frac{1}{3}$ —6% (preferably 2%) of zinc phosphide, with the addition, if desired, of about 10% of baked maize meal, fish meal, etc., or (B) are obtained by stirring together baked maize meal etc., heated oil or fat (e.g., lard), and about 2% of poison, preferably zinc phosphide.

L. A. COLES.

Distillation of water. G. E. HUGHES, A. II. WARING, J. E. BRAHAM, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 333,547, 14.2.29).—De-aerated and heated water is allowed to fall freely in films over the outside surfaces of a number of straight vertical tubes through which steam is passed from an exterior source. Some of the water is evaporated and the remainder recirculated by means of a pump. Water condensed on the inside of the tubes is passed to a flash boiler, and the steam released by the reduction in pressure together with that produced by evaporation in the first effect is led into the tubes of a second evaporator and the cycle is repeated. The steam issuing from the final evaporator is condensed, and the whole of the distilled water produced is collected and utilised. In a three-stage evaporator a known weight of steam produces about three times its weight of distilled water (cf. B.P. 279,526; B., 1928, 74).
C. JEPSON.

Distillation of water. A.-G. BROWN, BOVERI & Co. (B.P. 334,141, 26.11.29. Ger., 5.12.28).—A supply of distilled water for boiler-feed purposes is rendered sufficiently elastic to provide for times of peak load by using a number of feed-water heaters in series and means by which steam from the still may be condensed in any of them at will. In this way the condensation temperature of the hot vapour is reduced and the whole of its heat transferred to the feed water without any disturbing increase of temperature.
C. JEPSON.

Apparatus for removal of injurious gases from underground workings. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of F. GERLACH (B.P. 333,876, 15.5.28. Ger., 20.12.28).

Line filter (U.S.P. 1,755,780). **Straining liquids** (B.P. 333,850).—See I. **Filter materials** (B.P. 333,844).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 7 and 14, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat losses from plant surfaces. R. M. COLLES (Trans. Ceram. Soc., 1930, 29, 182—190; cf. B., 1929, 913).—Steel cylinders were heated at 230° or at as high a temperature as the surface coating would permit, i.e., at temperatures at which convection forms the major source of loss, and the rate of loss of heat was plotted against the temperature for various coatings and weather conditions. Since radiation loss varies as the difference between the fourth powers of the absolute temperatures of the object cooling and that of its surroundings, and convection loss is proportional to the 1.25th power of the difference in temperature, it is possible to separate the two losses. It is found that radiation is greatly affected by the nature of the surface and is unaffected by the weather, whilst convection is almost *vice versa*. The formulæ and graphs are convenient to use, on the assumption that the surface temperature can be first estimated and the increased convection loss caused by wind and decreased by shielding is demonstrated. The shape of the object is not very important unless it is so re-entrant that pockets of still air are formed or radiation to itself takes place. A wind of 10 m.p.h. increases the convection loss to 2½—3 times that in still air. B. M. VENABLES.

Application of Osborne Reynolds' theory of heat transfer to flow through a pipe. G. I. TAYLOR (Proc. Roy. Soc., 1930, A, 129, 25—30).—Mathematical. The work of Eagle and Ferguson (B., 1930, 843) is criticised on the ground that Reynolds' theory suffers from two possible sources of error, and that even if the theory is true, the experiments were carried out under conditions to which it is not applicable. Moreover, Eagle and Ferguson's method of measuring mean temperature at any section of their pipe is inadmissible. L. L. BIRCUMSHAW.

Factors affecting the problem of smoke prevention. W. E. GIBBS (J. Inst. Fuel, 1930, 3, 361—375).—Coal smoke is formed partly by condensation (carbon particles) and partly by mechanical dispersion (ash particles). Screen analyses of particles from chain-grate stokers and powdered-fuel firing are given. The latter are the smaller, and the residual particles passing through a centrifugal separator show 97% below 75 μ . The total amount may be 6 g./m.³ of solids in such a smoke, and whilst the larger particles may be removed by gravity or centrifugal force, they do not carry the smaller particles with them. The problem of smoke prevention is that of the flocculation of the smaller particles. It is shown that with turbulent motion in gas flues there is no possibility of gravity settlement of particles of less than

500 μ diam. Centrifugal separators readily collect particles down to 50 μ . The most efficient types combine a high radius of curvature with a short transverse path for the particles. Perforated plates with staggered perforations give this condition, but the power consumption is too high for power-station practice. The efficiency of all separating methods is greatly increased by wetting the particles. Spray-washing is much more effective than bubbling, but smokes are much more difficult to wash than gases as the particles move more slowly than gas molecules. The degree of dispersion of the spray and the "wettability" of the smoke particles are both important. The latter quality may be modified by lowering the surface tension of the water or by electrical charges. The electrostatic flocculation of smokes is also discussed. The cyclone type of separator is the most satisfactory apparatus known at present, but it would be desirable for it to be supplemented by some simple method of flocculating the finer particles. C. IRWIN.

Correlation of sieving analyses. H. HEYWOOD (J. Inst. Fuel, 1930, 3, 428—432).—Results of sieving analyses depend on variations in procedure, time of sieving, and variation in aperture with the diameter of the sieve wire. Curves are obtained giving conversion factors for use in the sieving of coal dust with increase in time of sieving, weight on sieve, and slight variations in aperture. The true apertures in microns of a number of standard sieves are given. No standard exists for silk sieves, in which the aperture depends on the strength of the strands, and very high tolerances are necessary for the finer wire sieves. C. IRWIN.

Electrical determination of the dew point of flue gases. H. F. JOHNSTONE (Univ. Ill. Eng. Exp. Sta. Circ., 1929, No. 20, 22 pp.).—A method based on the variation of the electrical conductivity of the surface of a dielectric when a film is condensed on it is described. Minimal concentrations of sulphur trioxide exert a profound effect on the dew point.

CHEMICAL ABSTRACTS.

Determination of the capacity of a large vessel. F. W. J. CLENDINNEN (J. Soc. Chem. Ind. Victoria, 1930, 30, 361).—The vessel is filled to the mark with water and the chloride content of this determined before and after the addition of a relatively small measured volume of saturated brine solution by Cavanagh's electrometric titration method (A., 1927, 1045; 1928, 607). The capacity is calculated from these data.

H. I. DOWNES.

Town's gas for heat-treatment furnaces. WALTER.—See II. **Wetting of powders.** HARKINS and DAHLSTROM.—See XIII.

* The remainder of this set of Abstracts will appear in next week's issue.

PATENTS.

Retort furnace. C. V. MCINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS CO. (U.S.P. 1,757,644, 6.5.30. Appl., 20.8.25).—The heat-transmitting wall is semi-cylindrical with deep circumferential corrugations and is made of easily renewable metallic sections. The coal or other material under treatment is conveyed through the trough by stirrers which oscillate through about 270°.

B. M. VENABLES.

Heating furnace [for viscous fluids, e.g., tar etc.]. H. A. DREFFEN (U.S.P. 1,757,707, 6.5.30. Appl., 30.3.28).—To avoid local overheating of the fluid in a tubular, direct-fired heater, the burning gases are caused to pass upwards through a free space between two nests of tubes, heating them only by radiation; the partially cooled gases are then passed in contact with the tubes.

B. M. VENABLES.

[Regulation of] dry kilns. M. B. HALL, Assr. to FOXBORO CO. (U.S.P. 1,757,629, 6.5.30. Appl., 7.9.27).—Wet- and dry-bulb thermometers containing a volatile liquid are inserted in the kiln, and the vapour pressure of the thermometric liquid directly actuates valves controlling, respectively, water sprays and heating steam. The "bulb" of the dry thermometer is in the form of a long tube extending along the full length of the kiln, so that the maximum temperature at any point will be the controlling one.

B. M. VENABLES.

Heat-insulating material. G. B. PEEBLES (U.S.P. 1,757,470, 6.5.30. Appl., 8.8.28).—Sheets for building-board or insulating purposes are constructed from the precipitated waste products of the ammonia-soda process, which are dried, mixed with magnesium and sodium silicate, and pressed at about 20,000 lb./in.²

B. M. VENABLES.

Grinding mill. G. F. PETTINOS (U.S.P. 1,758,010, 13.5.30. Appl., 18.8.28).—The mill is of the disintegrator type with rigid hammers and a vertical shaft, and with a number of grinding compartments, increasing in diameter downwards, which are separated from each other by annular partitions.

B. M. VENABLES.

Attrition mill. A. SCHUCHARDT (U.S.P. 1,758,539, 13.5.30. Appl., 2.4.29).—Means are described for feeding material through apertures close to the centre of one disc of a disc grinder in which each disc is rotated by a separate motor.

B. M. VENABLES.

Disintegration of [cellular] matter. W. KOEHLER (U.S.P. 1,746,731, 11.2.30. Appl., 21.1.29).—Cellular material of any kind, e.g., oils, fats, vegetable products, or living organisms, is broken down and the material sterilised by subjecting it to the very high pressure and radiant heat produced by an explosion, the pressure being then suddenly, but not necessarily immediately, reduced to a low figure by exhausting into a vacuum chamber. For the sterilisation of milk, hydrogen and oxygen in atomic proportions would be used; for fruit juices it is better to use carbon oxysulphide and oxygen in proportions to yield sulphur dioxide and carbon dioxide.

B. M. VENABLES.

Apparatus for separating comminuted matter. R. ANDERSON (U.S.P. 1,758,422, 13.5.30. Appl., 12.6.28).—The apparatus, particularly suitable for

separating precious metals from sweepings, comprises a vertical tube with closed bottom and open top (guarded by a dust bag) and divided at an intermediate point by a collar supporting a screen underneath which is an air inlet. At the commencement of operations the material is laid on the screen, the apparatus is then assembled, and the separation effected by puffs of air.

B. M. VENABLES.

Mixer. J. JOHNSON, Assr. to TURBO-MIXER CORP. (U.S.P. 1,757,197, 6.5.30. Appl., 14.11.28).—A bladed impeller is surrounded by stationary deflecting blades which support a screen or other disintegrating means.

B. M. VENABLES.

Wet-mixing method and apparatus. F. D. PFEFFER and F. TROTTER, Assrs. to UNITED STATES GYPSUM CO. (U.S.P. 1,758,200, 13.5.30. Appl., 19.3.26).—A proportioning and mixing device is described in which the actual mixing is effected by a centrifugal disc provided with pins.

B. M. VENABLES.

Obtaining solids from liquids. A. B. JONES, Assr. to INDUSTRIAL ASSOCIATES, INC. (U.S.P. 1,758,745, 13.5.30. Appl., 5.8.26).—The liquid is subjected to spray-drying in two stages, viz., concentration and solidification, at different temperatures and in the presence of different quantities of gases. Preferably, moderately heated gas is supplied to the solidification stage and exhausted gas therefrom together with any entrained dust to the concentration stage, where an additional quantity of more highly heated gas is supplied in such a way that it meets the raw liquor spray before mingling with the already used gas; the exhaust, however, is common to both quantities.

B. M. VENABLES.

[Self-balancing] centrifugal dryer. W. V. ORR (U.S.P. 1,757,585, 6.5.30. Appl., 6.5.29).—An outer basket is rigidly attached to the shaft, and is arranged to retain some liquid, but the inner basket (containing the material) is of spherical or other bulged shape, and is permitted a limited movement on spherical seats; when it deflects owing to uneven loading it will displace liquid from the outer basket on the heavy side, thus restoring the balance.

B. M. VENABLES.

Rotating filter. E. B. GOODWIN (U.S.P. 1,758,910, 13.5.30. Appl., 24.7.28).—A hollow, truncated cone is rotated about a horizontal axis by a hollow shaft on one end of which, beyond the cone, driving means is provided, and on the other end is a feed scoop. The interior of the cone is fitted with an interrupted helix tending to carry material up to the small end. The feed apertures in the shaft are some distance within the filter, and nearer the small end is a supply of wash-liquor, the large end being closed by filter medium.

B. M. VENABLES.

Multi-disc suction filter. V. C. BENJAMIN and P. W. PRUTZMAN, Assrs. to CONTACT FILTRATION CO. (U.S.P. 1,757,355, 6.5.30. Appl., 4.1.28).—The filter discs lie in a single vertical plane, and the hollow horizontal shaft is used for the application of vacuum only. Automatic ball valves, placed in the short spokes between the discs and the shaft, serve to cut off the vacuum as a disc with nearly dry cakes reaches the top of its travel; the cakes are then scraped off from each side of the disc.

B. M. VENABLES.

Filter mat. R. STRINDBERG (U.S.P. 1,757,690, 6.5.30. Appl., 16.3.28).—A filter suitable for cleaning air or gas is constructed of a stiff wire bent zig-zag to form a frame, around which is entwined a strand of wire-cored wool or other suitable substance.

B. M. VENABLES.

Pressure distillation. N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,757,579, 6.5.30. Appl., 16.5.22).—The vapours of, *e.g.*, oil derived from a still are passed through a horizontal drum with inclined shelves down which condensed light oils and feed liquor trickle from another drum above. In the uppermost drum the vapours are bubbled through the light and feed oils, and finally exhaust through a condenser. The top drum may have a cooling coil, and is fed with fresh oil equal in amount to that of the distillate.

B. M. VENABLES.

De-aeration and evaporation of liquid. W. S. ELLIOTT (U.S.P. 1,758,566, 13.5.30. Appl., 27.3.23).—The liquid, *e.g.*, raw feed-water, is heated by splashing over trays in the presence of steam in a vessel at a pressure and temperature corresponding to that of the steam; the liquid falls to a flash boiler, which is a similar vessel maintained at a lower pressure so that a small proportion flashes into steam and carries with it all the dissolved gases. The vapour and gases pass to a condenser-evaporator where the vapour is condensed and runs back to the flash boiler while the permanent gases only are withdrawn by an ejector or other device. The cooling-water side of the condenser is provided with a vapour space, this being maintained at a still lower pressure so that part of the cooling water is evaporated, the vapour from which is condensed in another heat exchanger, yielding distilled water and preheating the raw water. A modification is described by which the yield of distilled water may be increased by applying more heat indirectly to the condenser-evaporator.

B. M. VENABLES.

Viscosimeter and display device. W. D. SMITH (U.S.P. 1,758,677, 13.5.30. Appl., 15.6.25).—A pair of evacuated test tubes are about half filled with the two liquids to be compared, *e.g.*, fresh and used oil; midway of the tubes is a diaphragm with a small aperture through which the oil will flow in a thin stream when the tubes are inverted. Another pair of tubes to demonstrate dirt in used oil may also be mounted in the same frame.

B. M. VENABLES.

Dialysing apparatus. E. HEIBIG (U.S.P. 1,757,364, 6.5.30. Appl., 13.6.28. Holl., 2.2.27).—Cf. B.P. 329,235; B., 1930, 694.

[Pulverised fuel] furnace walls. O. NYGAARD (B.P. 313,875, 6.6.29. U.S., 18.6.28).

Refrigerators. N. V. MIDDEN-EUROPEESCHE OCTROOI-MAATS. (B.P. 319,270, 27.8.29. Switz., 19.9.28).

Absorption refrigerating systems. ELECTROLUX, LTD., Assees. of A. LENNING (B.P. 319,015, 14.9.29. U.S., 15.9.28).

Absorption machines for producing cold or heat. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 313,618, 14.6.29. Ger., 16.6.28).

Evaporator for cooling plants [refrigerators].

O. FRH. VON KOHORN (O. KOHORN & Co., MASCHINENFABR.) (B.P. 334,535, 4.6.29. Austr., 6.3.29).

Absorption process (U.S.P. 1,748,051).—See II. **Carrying out reactions under pressure** (U.S.P. 1,746,687).—See III. **Humidity-control apparatus** (U.S.P. 1,757,931).—See V.

II.—FUEL; GAS; TAR; MINERAL OILS.

Hardness, abrasability, and reactivity of coke. R. A. MOTT (Fuel, 1930, 9, 400—411).—From a comparison of the results of shatter tests with those of an abrasion test carried out by rotating 2—3-in. coke in a smooth-walled drum under standard conditions it is concluded that the $\frac{1}{2}$ -in. shatter index can be used as a measure of the "abrasion hardness" of a coke. A good blast-furnace coke should possess a low abrasability, *e.g.*, a $\frac{1}{2}$ -in. shatter index of not less than 97, combined with a sufficiently high "impact hardness," a measure of which is given by the $1\frac{1}{2}$ -in. shatter index. The relationship between the $\frac{1}{2}$ -in., 1-in., $1\frac{1}{2}$ -in., and 2-in. shatter indices is discussed, and the "normal" relationship, based on the figures for a large number of cokes tested by the Midland Coke Research Committee, is illustrated diagrammatically. Cokes made in fast-coking (14-in.) ovens yield abnormally low 2-in. shatter indices; cokes made by blending coking coals with non-coking materials yield abnormally high 2-in. shatter indices. The standard shatter-test apparatus adopted by the Midland, Northern, and Scottish Coke Research Committees is described. The reactivity of coke has been determined in a combustibility furnace of 1 sq. ft. grate area with a 12-in. bed of coke of standard size. The coke is blown with a specified amount of air and when a steady temperature has been attained gas samples are taken at different levels; the distances above the grate at which (a) the oxygen has just disappeared (reactivity towards oxygen) and (b) the concentration of carbon monoxide reaches 20% (reactivity towards carbon dioxide) are recorded. Different cokes differ little in reactivity at high temperatures. Marked reactivity is not generally desirable in a blast-furnace coke, since it is usually accompanied by low combustion temperatures. Decrease in coke size increases reactivity and decreases the average combustion temperature.

A. B. MANNING.

Briquetting of coal slack without the use of a binder. W. SWIENTOSLAWSKI, B. ROGA, and M. CHORAZY (Fuel, 1930, 9, 421—439; cf. B., 1929, 230, 1037).—The briquettes were made in a laboratory press under controlled conditions, and their mechanical strength and combustibility were determined. Neither anthracite nor semi-bituminous coals of low volatile content could be briquetted without a binder, even under high pressures (3000 kg./cm.²). Briquettes of satisfactory mechanical strength could be obtained from coking coals either (a) by heating to the temperature of incipient plasticity, (b) by prolonged heating at temperatures at which plasticity is fully developed (390—440°), or (c) by a shorter heating at a temperature corresponding to the last stage of the plastic state (450°); the necessary pressures are of the same order as those used industrially, *e.g.*, 200—300 kg./cm.² Agglutinating gas

coals gave briquettes under conditions similar to those used for coking coals. Non-agglutinating gas coals could also be briquetted under suitable conditions; the optimum temperature lay between very narrow limits, viz., 400–420°, and the optimum duration of heating was relatively short; the minimum pressure necessary was 400 kg./cm.² The briquettes possessed considerable mechanical strength, and behaved well on combustion. They were normally resistant to atmospheric conditions, and were not more hygroscopic than other solid fuels.

A. B. MANNING.

Simultaneous production of light oils, smokeless briquettes, and coke. L. VERTU (*Chim. et Ind.*, 1930, 24, 27–30).—Pulverised anthracite or coke is mixed with heavy petroleum, briquetted by pressure, and distilled. The gas evolved is used to supply part of the heat for the distillation. In the upper part of the retort, where the temperature is about 400°, smokeless briquettes are formed, and in the lower part, at about 650°, very hard coke. The oil which distils over is lighter and more volatile than that obtained by distillation of the heavy petroleum. By fractionating the distillate about 7% distils over up to 150°, and the residue may be used as a binder for the pulverised solid and re-treated, whereby a further yield of oil (b.p. below 150°) is obtained and the residue may be used as before.

D. K. MOORE.

Mechanism of charcoal activation. M. E. BARKER (*Ind. Eng. Chem.*, 1930, 22, 926–931).—Primary charcoal, prepared from wood or other carbonaceous materials, has a true density of about 1.45, contains considerable hydrogen and oxygen, which probably act as binders for the carbon grains, and shows no characteristic X-ray diffraction pattern. When such charcoal is activated, e.g., by steam at 800°, an internal shrinkage forms numerous minute voids and capillaries with the production of a large internal and active surface. The absorptive capacity of commercial wood charcoal may thus be increased from 11 to 1480. As a result of such activation, the charcoal granules become of graphitic character having a crystalline surface and a limiting d of 2.15. Part of the occluded gases, but not all, is expelled, and the diffraction rings attributed to graphite are observed.

C. A. KING.

Wood as a source of fuel for internal-combustion engines. G. DUPONT (*Chim. et Ind.*, 1930, 24, 3–19).—The production of gas in gas producers on the vehicle is advocated. The fuel used in the gas producer must be low in ash and very reactive, give rise to no corrosive gas, and the gas must carry with it practically no dust or tar. Wood and wood charcoal are found to meet these requirements, but high-temperature charcoal is too dirty and bulky and wood evolves vapours when the engine is stopped. When pine wood is distilled there is little decomposition below 275°, and at about 300° exothermic decomposition takes place. As the temperature rises the percentage of carbon monoxide evolved increases until 320° is reached, after which it decreases and hydrogen and hydrocarbons are produced. Acetic acid is formed chiefly at 280–310° and tar at 270–380°. Other woods, cellulose, and lignin behave in a similar way, except that lignin and the more ligneous woods undergo the exothermal decomposi-

tion at a somewhat higher temperature. The brown charcoal produced at the temperature at which exothermic decomposition begins (275–290°) is dense, convenient for manipulation, and gives theoretically the best thermal yield (calc. on dry wood); when gasified in the producer it gives a gas with the greatest power per m.³ of engine cylinder, and practically no vapours are evolved in the producer when the engine is stopped. These theoretical figures have been experimentally confirmed. If the wood is distilled in a bath of organic liquid, e.g., pitch, 5–10% of the pitch is absorbed by the charcoal, and this by cracking in the producer should increase the cal. value of the gas; in practice, however, such increase is negated owing to imperfect cracking, smoke formation, and channelling. Impregnation of the wood with acids, e.g., phosphoric acid, before distillation is found to facilitate the evolution of water from the cellulose, whilst the lignin is not affected, and both the yield and cal. value of the charcoal are increased. Impregnation with alkalis, e.g., soda, retards the decomposition of the cellulose.

D. K. MOORE.

Separation of the constituents of coal gas by condensation in stages. A. THAU (*Gas- u. Wasserfach*, 1930, 73, 717–721; cf. *Bronn, B.*, 1929, 766).—The plant at Mont Cenis, comprising four units, each capable of dealing with 5300 m.³ of coke-oven gas per hr., is described (cf. *Engineering*, 1930, 129, 183). The separated mixture of hydrogen and nitrogen is used for the synthesis of ammonia. The plant at Duisberg, which supplies hydrogen for the recently-erected Bergius hydrogenation plant, has a throughput of 7000 m.³ of coke-oven gas per hr., and produces therefrom 3500 m.³ of hydrogen of 95% purity. The utilisation of the residual gas is discussed. The process cannot be used economically for rendering town's gas non-poisonous by the removal of carbon monoxide.

A. B. MANNING.

Utilisation of town's gas as a fuel in heat-treatment furnaces. C. M. WALTER (*J. Inst. Fuel*, 1930, 3, 408–419).—The first regenerative, gas-fired oven furnace heated the secondary air from the working chamber, and was capable of producing temperatures up to 1250°. Better recuperation was provided in later types, but even so thermal efficiencies rarely exceeded 25%, owing to very high radiation losses. The much higher figures now obtained are due to advances in insulation. A detailed account is given of trials with a set of modern recuperator furnaces, burning up to 700 cub. ft. of gas per hr., employed for carburising gear parts at 930°. Control was either by hand or arranged automatically by means of a thermocouple working on the gas supply, together with automatic damper control operated by the same motor. The efficiency is estimated at over 50% during the heating-up period, and the gas consumption is less than half that of furnaces of older type.

C. IRWIN.

Determination of the softening points of asphalts and pitches by the Kraemer-Sarnow method. H. BURSTIN (*Petroleum*, 1930, 26, 789–791).—The softening points of 8 asphalts and 2 pitches have been determined by the Kraemer-Sarnow method and by modifications thereof in which the mercury is replaced by a small metal rod (cf. *Spilker, B.*, 1929, 383; *Holde, B.*, 1929,

584). In general, the modified methods gave lower results than the original, the differences varying from $+1.7^{\circ}$ to -3.7° . Better agreement was obtained when a brass rod, 4 mm. in diam. and weighing 5 g., was used than with the type-metal rod suggested by Spilker (*loc. cit.*).
A. B. MANNING.

Cracking of petroleum oils and low-temperature coal tars. W. H. JONES (J. Inst. Fuel, 1930, 3, 376—392).—Vapour-phase processes have the disadvantages of high fuel consumption, large production of gases and carbon, heavy up-keep and operating costs, but they produce a petrol of high antiknock value. Liquid-phase processes working at 100—750 lb./in.² and 450—500° include practically all the commercial units working to-day. For each oil there is a narrow temperature range which must not be exceeded. The Gyro process, the Cross process, and the Dubbs process representative of three classes are described in detail. Cracking processes may be worked to produce either fuel oil or coke, in the latter case with a higher yield of petrol. Analyses of charging stock and products are given. Results of cracking on a laboratory scale of various American low-temperature tars are given, together with cracking results from neutral oils washed for acid, German lignite tar, and distillates therefrom. Petrol prepared by these methods requires treatment with caustic alkali and sulphuric acid to render it water-white and stable to light.
C. IRWIN.

Conversion of low-temperature tar into light oil. G. KROUPA (Petroleum, 1930, 26, 791—794).—The Semo process is described (cf. Stephan, B., 1930, 173).
A. B. MANNING.

Extinction of ethylene dichloride flames with carbon dioxide. G. W. JONES and R. E. KENNEDY (Ind. Eng. Chem., 1930, 22, 963—964).—The limits of inflammability of ethylene dichloride-air mixtures at 100° were 5.8% and 15.9%. The lower limit at 22° was 6.2%. Ethylene dichloride vapour at 100° is rendered non-explosive by mixing it with at least 2.3 times its own volume of carbon dioxide, and it is recommended that at atmospheric temperature and pressure at least 2.5 vols. of carbon dioxide should be added per vol. of ethylene dichloride.
C. A. KING.

Smoke prevention. GIBBS. **Sieving analyses [of coal dust].** HEYWOOD.—See I. **Infusorial earth and floridin.** MARKMAN and KOVALENKO.—See VII. **"Flotation sulphur" for control of fruit diseases.** SMITH.—See XVI.

PATENTS.

Artificial fuel. J. A. WYLER, Assr. to TROJAN POWDER Co. (U.S.P. 1,752,935, 1.4.30. Appl., 9.1.28).—An infusible artificial fuel consists of a solidified mixture of nitrostarch, methyl and ethyl alcohols, and water. The addition of small quantities of formaldehyde, furfuraldehyde, etc. assists the formation of the gel.
A. B. MANNING.

Preventing or diminishing the formation of smoke in the burning of coal in open fire-places. J. WARR (B.P. 331,943, 19.4.29).—The fire grate is fitted with an enclosed retort, which may take the form of a metal box built behind the fire, and in which the coal is predistilled before being burned in the grate.

The volatile products of distillation are conveyed to a burner which heats the retort from below.

A. B. MANNING.

Production of finely-divided carbon or carbon black. G. C. LEWIS (B.P. 331,821, 1.4.29).—A gaseous mixture containing hydrocarbons is heated at a temperature, e.g., 600—850°, at which reactions occur involving the formation of aromatic hydrocarbons without the production of free carbon, and the products, after being cooled to a temperature, e.g., 250°, above that at which the vapours present would begin to condense, is fed to a burner for the production of free carbon by incomplete combustion.
A. B. MANNING.

Production of activated carbon. O. FUCHS, Assr. to HOLZVERKOHLLUNGS-IND. A.-G. (U.S.P. 1,753,507, 8.4.30. Appl., 9.2.29. Ger., 13.1.28).—Carbonised material is reduced to particles of approximately uniform shape, size, and sp. gr., and is then treated with activating gases for a comparatively short period of time at about 900°.
A. B. MANNING.

Activation of charcoal. G. ENSSLE, Assr. to HOLZVERKOHLLUNGS-IND. A.-G. (U.S.P. 1,753,984, 8.4.30. Appl., 6.2.29. Ger., 13.1.28).—The charcoal is treated at a suitable temperature with a mixture of gases, free from oxygen, containing at least 40% of carbon dioxide and a substantial proportion of a combustible gas. The uncondensable gases resulting from the carbonisation of wood form a suitable activating agent.
A. B. MANNING.

Activation of carbonaceous materials suspended in gases. SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIÈRES (B.P. 317,047, 23.7.29. Fr., 9.8.28).—The carbonaceous material, suspended in the activating gas, is passed through tubular retorts so designed that the products issuing therefrom are cooled by and simultaneously preheat the entering gas and material.
A. B. MANNING.

Apparatus for high- or low-temperature distillation of coal or lignite. H. HARDY (B.P. 317,374, 14.8.29. Belg., 14.8.28. Cf. B.P. 318,520; following abstract).—A number of oven units are arranged in a group, preferably in ring formation. Each unit consists of superimposed heating, distillation, and suction chambers. Between the units are combustion chambers having shutters by which they may be placed in communication with the heating chambers of one or both of the adjacent units; also conduits are provided whereby the hot gases from one oven unit may be conducted to another while permitting one or more intermediate units to be by-passed. By these means the temperature of each oven unit can be independently regulated. The suction chambers of each unit communicate with means for collecting and separating the tar and other by-products. The coal is carried in a removable mould having a perforated cover held in place by locking means in order to prevent swelling of the coal during distillation.
A. B. MANNING.

Low-temperature distillation of coal or lignite with production of semi-coke and valuable by-products. H. HARDY (B.P. 318,520, 3.9.29. Belg., 4.9.28).—The coal is contained in moulds which are conveyed through the distillation chamber by an

arrangement of rack bars and pinions, or similar device. The distillation chambers alternate with suction chambers and combustion chambers in such a manner that the moulds are heated only from one side, the volatile distillation products passing from the other side into the suction chambers and thence to the condensers. The heating of the distillation chambers is so arranged that the moulds pass successively through zones of gradually rising temperature until distillation is complete, and then through a cooling zone. The heat evolved in the latter is utilised for preheating the air for combustion. The moulds are charged and discharged in a separate plant. (Cf. preceding abstract.)

A. B. MANNING.

Drying, low-temperature carbonisation, distillation, or combustion of granulated or pulverulent fuels. C. GEISSEN (B.P. 310,032, 12.4.29. Ger., 20.4.28).—A layer of the material is supported on a horizontal grate and the active gases or vapours are supplied through openings in the grate which are provided with mushroom- or valve-shaped distributing devices. The shafts of the latter almost fill the openings in the grate, leaving only narrow annular passages which offer a high resistance to the flow of the gases or vapours.

A. B. MANNING.

Working of chamber ovens for production of gas and coke. C. OTTO & Co., G.M.B.H. (B.P. 318,935, 12.9.29. Ger., 12.9.28).—The ovens are arranged in groups and adjustable closing devices for each group are provided in the common gas and air supplies in such a manner that each group can be worked at a different carbonising period. By this means it is possible to vary the gas output over a considerable range without putting any of the groups of ovens out of operation.

A. B. MANNING.

Manufacture of liquid products from coal or carbonaceous materials by heating under pressure in the presence of hydrogen, or gases or vapours containing hydrogen. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 314,859, 31.5.29. Holl., 3.7.28).—“Luxmass,” the alkaline material containing iron oxide obtained as a by-product from bauxite in the preparation of aluminium oxide, is used as a catalyst in the hydrogenation process. It is found to be much improved for this purpose if it is first repeatedly extracted with hot water, and then dried at 120°.

T. A. SMITH.

Destructive hydrogenation of coal, oils, etc. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,817, 2.4.29).—The vapours originating from the destructive hydrogenation of carbonaceous materials in the liquid phase are superheated and are then passed, together with hydrogen, over a hydrogenating catalyst at a temperature below that prevailing in the superheating zone. Cracking catalysts, e.g., iron coated with zinc, but not hydrogenating catalysts, may be used in the superheating zone.

A. B. MANNING.

Recovery of the products of destructive hydrogenation and cracking processes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,916, 11.4.29).—The processes of condensing the vapours and scrubbing the gases leaving the reaction vessel are carried out

simultaneously by introducing the washing oil into the gaseous and vaporous products before they enter the condensers.

A. B. MANNING.

Gas producers. SOUTH METROPOLITAN GAS CO., and C. C. CARPENTER (B.P. 332,921, 30.4.29. Cf. B.P. 305,764; B., 1929, 313).—Loss of fuel during clinkering is diminished by substituting for the natural draught a suction so controlled that the draught is kept above the minimum necessary for avoiding dangerous escape of hot gas through the clinkering doors. The substituted draught is preferably equal to or only slightly less than the normal draught during the non-clinkering period. It is also preferable to apply the suction at a level above the normal off-take of the producer in such a manner that the gases are drawn through a bed of freshly charged coke; with such an arrangement a diminished suction may be applied also during the non-clinkering period.

A. B. MANNING.

Removal of sulphur compounds from gases. W. J. HUFF, L. LOGAN, and O. W. LUSBY (B.P. 305,026, 25.1.29. U.S., 28.1.28).—The gases are passed over a purifying material comprising at least one metal of group VI or VII in intimate admixture with a metal of another group, e.g., copper (80%) and chromium (20%), or copper (80%), uranium (10%), and chromium (10%). The treatment is carried out above 250°, and removes hydrogen sulphide and organic sulphur. The purifying material is revived by the action of an oxidising gas.

A. B. MANNING.

Ammonia-recovery process [from coke-oven gas]. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,747,616, 18.2.30. Appl., 2.8.22).—The gas is scrubbed with hot wash-liquor, consisting of aqueous condensate from the coke ovens, to remove the fixed ammonium compounds, and is then passed up a cooler in counter-current to cold wash-liquor, which takes up part of the free ammonia. The hot wash-liquor is conducted to the lower part of an ammonia still, where it is mixed with milk of lime and distilled; the cold liquor undergoes separate distillation in the upper part. The combined vapours together with the gas from the cooler pass to a saturator, where the ammonia is recovered. Means are provided for separating the tar. The process considerably reduces the amount of wash-liquor to be evaporated and more effectively utilises the remainder.

W. J. WRIGHT.

Manufacture of acetylene from hydrocarbons or mixtures containing them in the electric arc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,917, 24.4.29).—Acetylene is produced in 4–8% yield in one operation, without appreciable deposition of carbon, when methane is passed through an electric arc of at least 50 kw., the ratio of cub. m. of gas per hr. to the kw. power of the arc being between 0.6 and 1.6. A direct or an alternating current of 500–2000 periods is employed.

C. HOLLINS.

Treatment of hydrocarbons. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 334,032, 30.7.29. Holl., 23.10.28).—Blown asphalt is prepared in an apparatus consisting of a separating vessel and a heating coil through which the asphalt may be circulated. Air is blown through a meter into the bottom of the separating

vessel and volatile products are removed. Air connexions are also made to the heating coil so that this may be blown through if necessary. The vapours from the separator are condensed, the liquid being used as fuel. The permanent gases are also used as fuel.

T. A. SMITH.

Manufacture of liquid fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,550, 11.3.29).—Coal, tars, or mineral oils, or their products by distillation, destructive hydrogenation, or cracking, and preferably the middle oil fraction of the same, are subjected to the action of solvents which separate the initial materials into portions richer and poorer in hydrogen; the latter, after removal of the solvent, is subjected to destructive hydrogenation under pressure either alone, or with other carbonaceous substances, or even with a portion of the fraction rich in hydrogen, thereby producing a motor fuel of low b.p. Suitable solvents are liquid sulphur dioxide or ammonia, used, if desired, with cyclohexane or cyclohexanol and methyl alcohol.

H. S. GARLICK.

Natural-gas liquefaction. H. DUMARS, Assr. to BOWEN-DUMARS POWER CORP., and H. F. CUNTZ (U.S.P. 1,747,761, 18.2.30. Appl., 8.9.22).—Natural gas, at well temperature and pressure, is admitted to an expansion engine where the temperature and pressure are reduced, but not to such an extent that liquefaction takes place. The cooled gas is passed into heat-exchange relation with another portion of the gas, at well temperature and pressure, which after precooling is admitted to a second expansion engine where liquefaction of at least a portion of the gas takes place.

H. S. GARLICK.

Absorption process [for natural gas]. P. D. BARTON, Assr. to UNITED OIL CO. (U.S.P. 1,748,051, 25.2.30. Appl., 4.5.26).—Natural gas is passed into contact with a colloid solution composed of water and a colloid capable of absorbing gasoline, whereby the gasoline is absorbed and subsequently separated by stratification. The process is applicable to any mixed fluids and to absorbing medium capable of forming an emulsion selectively with one of them.

H. S. GARLICK.

Oil-cracking process. A. SCHWARZ, Assr. to PETROLEUM AND PRODUCTS CORP. (U.S.P. 1,756,887, 29.4.30. Appl., 25.2.24).—Petroleum oil is distilled at cracking temperature and under superatmospheric pressure in a cracking zone and the vapours are passed under successively decreasing pressures into successive masses of subdivided solid particles (*e.g.*, iron wool) wherein a portion of the vapours are condensed and held in minutely subdivided form over an extended area, while the rest makes intimate contact with the condensate. Hot residue from the cracking zone is passed countercurrent to the flow of vapours, and fresh oil may be introduced into the last of the series of the condensing system.

H. S. GARLICK.

Cracking of hydrocarbons. E. C. HERTHEL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,755,600—1, 22.4.30. Appl., [A] 30.6.25, [B] 27.7.27).—(A) In conjunction with an oil-cracking pressure still is a heat-insulated reflux tower of baffle construction into which fresh oil is introduced one or two baffles below the top of the tower. Initially the oil flows downwardly in one or more com-

pact streams which are not broken up and sprayed or distributed through the vapours until the oil is part way down the tower, thus avoiding difficulties due to entrainment of constituents of fresh oil in the vapour escaping from the vapour end of the tower. (B) Vapours from a pressure distillation are passed into a reflux condenser into which a light oil, which is substantially vaporised completely, under the prevailing conditions, is introduced, in direct contact with the vapours at a series of points so distributed that the proportion supplied at the part nearest the vapour inlet to the reflux condenser is less than the proportion supplied at a further point in the path of vapour travel.

H. S. GARLICK.

Cracking of [hydrocarbon] oils. C. P. DUBBS (B.P. 332,913, 27.3.29).—The oil is heated in a cracking zone to cracking temperature and passed directly into an adjacent reaction or separating zone under lower pressure where cracking is completed and the treated oil separated into vapours, which are dephlegmated, and an unvaporised liquid residue, which is removed without coming into contact with either reflux condensate or with fresh oil. To obtain a liquid residue suitable for fuel oil, the polymerisation of the unvaporised residue is checked (a) by controlling the reduction of pressure in the reaction zone with consequential temperature reduction therein; (b) by introducing as a cooling medium, into direct contact with the oil, subsequent to the heating but prior to its expansion, an oil of lower temperature than that of the oil being cracked; (c) by regulating the speed of withdrawal of the residue and so maintaining a controlled liquid level; and (d) by dissipating the remaining heat from the residue immediately after its withdrawal in such a way that the amount of sludge or coke formed in the residual oil is normally less than 6%.

H. S. GARLICK.

Cracking of hydrocarbons. G. EGLOFF and J. C. MORRELL, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,748,065, 25.2.30. Appl., 28.6.26).—Oil is subjected to cracking temperature and pressure and the cracked vapours are removed and collected. The residue from the expansion chamber, containing carbonaceous and pitchy material, is passed continuously through a colloid mill and passed again either through the same or another cracking coil.

H. S. GARLICK.

Manufacture of hydrocarbons which are saturated with hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,511, 2.4. and 14.10.29).—Crude hydrocarbon mixtures are treated with hydrogen, or gases containing or supplying hydrogen, under ordinary or increased pressure and at temperatures above 200° (preferably 300–360°), in the presence of catalysts preferably deposited on or applied to carriers such as Florida or fuller's earth, silica gel, etc. The catalysts consist of solid oxides or sulphides of the metals of groups II–VII or compounds of these metals capable of supplying such oxides or sulphides, preferably those of group VI or mixtures thereof, again preferably in conjunction with metals of group VIII or with heavy metals of group I, *e.g.*, silver, gold, or suitable compounds thereof. Examples are oxides or sulphides of molybdenum or tungsten and nickel or cobalt. The process is

continued until not only any sulphur or oxygen compounds present in the materials under treatment, but also nitrogen and halogen compounds as well as resinifying agents, are rendered non-injurious, without the occurrence of any appreciable splitting up of the hydrocarbons themselves. When treating polynuclear compounds the conditions should be such that in addition to purification of the hydrocarbons a hydrogenation of some, but not all, of the double linkings in the molecule takes place. The purified hydrocarbons thus obtained are hydrogenated in the presence of hydrogenating catalysts activated with metals of groups II—VII, or compounds thereof, not already contained in the catalyst of the first stage.

H. S. GARLICK.

Pyrolysis of unsaturated hydrocarbons. IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, and J. MCAULAY (B.P. 332,998, 4.6.29).—Olefines are passed at high space velocity (*e.g.*, 100 or 6400 vols. per min. through unit vol. of tube) through a tube heated above 1000° (*e.g.*, at 1050° or 1100°) for conversion into light oil.

C. HOLLINS.

Converting high-boiling mineral oils containing unsaturated compounds into lower-boiling hydrocarbons with simultaneous decoloration. N.V. MIJNBOW- EN CULTUURMAATS. "BOETON" (B.P. 333,553, 9.4.29. Holl., 16.1.29).—Crude oil containing unsaturated compounds is treated outside the still at temperatures above 60° (preferably at 70—100°) with concentrated aqueous solutions, liquid at ordinary temperatures, containing at least 58% of one or more solid hygroscopic chlorides of heavy metals, *e.g.*, ferric chloride solution of *d* 1.6; the product after removal of the salt solution is neutralised if necessary prior to distillation.

H. S. GARLICK.

Desulphurisation of liquid hydrocarbons. STADTBERGER HÜTTE A.-G. (B.P. 332,910, 26.2.29).—The liquids are completely dehydrated and freed from a portion of their sulphur content by known methods and then introduced into the bottom of a tubular receptacle (fitted with heating coil and stirrer) charged with finely-divided copper, independent of any support, that has been prepared by precipitating metallic copper from its salt solutions by means of iron or other suitable base metal and subsequently heating it above 100° in a reducing atmosphere. A filtering device to retain any copper particles is disposed in front of the outlet.

H. S. GARLICK.

Production of hydrocarbons of low b.p. from those of higher b.p. H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,816, 2.4.29).—The vapours of middle oils are passed with hydrogen, under a total pressure of at least 20 atm. and at 400—500°, first over a contact mass consisting of iron coated with zinc, and then over a porous contact mass consisting of ferric oxide.

A. B. MANNING.

Apparatus for removing carbonaceous deposits from oil apparatus. L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,027—8, 29.4.30. Appl., 22.7.25. Renewed 2.2.29).—(A) A single, continuous, flexible element, *e.g.*, chain or cable, is suspended in such a way from spaced points at the top of the enlarged reaction chamber of an oil-cracking apparatus that its

withdrawal causes disruption of the deposited carbon. (B) Separate flexible elements may be attached diagonally to the chain described in (A) and so arranged that the system may be withdrawn as a whole, or part may be withdrawn through the top and the rest through the bottom of the reaction chamber.

H. S. GARLICK.

Treatment of acid sludge and fuel product obtained therefrom. R. A. HALLORAN, W. N. DAVIS, and G. A. DAVIDSON, Assrs. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,745,692, 4.2.30. Appl., 6.9.23).—The acid sludge obtained in the treatment of petroleum oils, with or without the addition of fuel oil, is mixed with dirty dilute acid in a closed retort and heated to hydrolysing temperature under a pressure exceeding 50 lb. until separation into acid and a liquid fuel is obtained.

H. S. GARLICK.

Manufacture of lubricating oils. Refining of hydrocarbon oils [for production of lubricating oils]. E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,756,153 and 1,756,156, 29.4.30. Appl., 16.6.28).—The constituents of raw stock suitable as components of lubricating oil are vaporised therefrom and the vapours bubbled through a series of hot, alkaline, liquid oil baths, the vapours being introduced beneath the surface of each of the baths. The oil baths are maintained alkaline by supplying a caustic alkali to the treating bath through which the vapours last pass and overflowing the liquid from the bath to the preceding one and so on until discharged from the last one. Vapours of the desired lubricating oil are taken off from the last hot oil bath and condensed out of contact with the liquid flowing countercurrent to the vapours through the series of hot oil baths.

H. S. GARLICK.

Lubricant. F. L. KOETHEN, Assrs. to ACHESON GRAPHITE Co. (U.S.P. 1,758,446, 13.5.30. Appl., 10.11.26).—A water-free lubricating compound comprises a fine suspension of an iron soap in a mineral oil.

H. S. GARLICK.

[Lubricant for] treatment of drill-stem joints. DE R. FRIZELL, Assr. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,758,598, 13.5.30. Appl., 7.6.27).—A water-insoluble grease consists of a calcium soap and a mineral lubricating oil containing suspended, comminuted, spherical, metallic particles, *e.g.*, 60% of lime soap cup-grease and 40% of air-floated zinc dust.

H. S. GARLICK.

Resolution of oil emulsions and reagents therefor. F. H. PENN (U.S.P. 1,758,802, 13.5.30. Appl., 23.12.25. Renewed 20.4.28).—The emulsion is treated with 0.02—0.1% of a reagent consisting of an acid derivative of a mineral oil, *e.g.*, a sulphonated paraffin oil (2 pts.), and a fatty acid glyceride, *e.g.*, cotton-seed, olive, or castor oils (1 pt.), with or without a trace of phenol.

H. S. GARLICK.

Motor fuel. W. GAUS, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,775,674, 16.9.30. Appl., 7.5.26. Ger., 8.5.25).—See B.P. 251,969; B., 1927, 645.

Refining of low-boiling hydrocarbons. J. METZGER, A. KREUTZER, and T. HELLTHALER, Assrs. to A. RIEBECK'SCHE MONTANWERKE A.-G. (U.S.P. 1,776,039,

16.9.30. Appl., 30.4.26. Ger., 10.6.25).—See G.P. 439,608; B., 1927, 695.

Production of solid lubricants. W. PUNGS and E. FRESE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,193, 16.9.30. Appl., 25.4.29. Ger., 9.5.28).—See B.P. 317,630; B., 1929, 862.

[Air-blast] extraction of dust from minerals such as coal. S. HUNTER (B.P. 334,792, 21.11.29).

Pulverised-fuel burners. BABCOCK & WILCOX, LTD. From FULLER LEHIGH Co. (B.P. 334,755, 14.10.29).

Retort furnace (U.S.P. 1,757,644). Heating furnace for tar etc. (U.S.P. 1,757,707). Pressure distillation (U.S.P. 1,757,579). Viscosimeter (U.S.P. 1,758,677).—See I. Polymerisation process (U.S.P. 1,746,168). Polymerised diolefines (B.P. 333,872 and 333,894). Organic compounds by dehydrogenation (B.P. 333,975). Oxidation of organic products (B.P. 309,498).—See III. Ammonia oxidation (U.S.P. 1,748,646).—See VII. Waterproof concrete (U.S.P. 1,744,869). Asphalt emulsions (B.P. 332,591). Pavement materials (B.P. 333,937). Paving blocks (B.P. 333,047). Road-making materials (B.P. 332,897). Wood-impregnating product (B.P. 310,804—5).—See IX. Rubber compounding (U.S.P. 1,743,433).—See XIV.

III.—ORGANIC INTERMEDIATES.

Flash points of mixed solvents. F. D. SNELL (Ind. Eng. Chem., 1930, 22, 893—894).—Mixtures of carbon tetrachloride with acetone and with toluene were prepared, representing commercially occurring cases of mixtures of non-inflammable solvents with inflammable solvents of higher and lower volatility, respectively. The flash points of the mixtures as prepared and after keeping until 50% evaporation had occurred were determined, the acetone mixtures showing a rise and the toluene mixtures a fall in flash point. The significance of these results with reference to "flame-proofed" mixtures sold as fabric cleaners, larvicides, etc. is discussed. S. S. WOOLF.

PATENTS.

Manufacture of chlorine and bromine derivatives of compounds of the acetylene series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,946, 25.5.29).—Chlorine or bromine is introduced into an acetylenic compound by direct substitution by action of an aqueous hypochlorite or hypobromite, preferably in presence of excess of free alkali. The preparation of dichloroacetylene, m.p. -64° to -68° , b.p. 32° ; dibromoacetylene; dibromodiacetylene, m.p. $52-53^{\circ}$ (decomp.); bromopropiolic acid, m.p. $84-86^{\circ}$; chloropropiolic acid, m.p. $69-70^{\circ}$; 1-bromomethinylcyclohexan-1-ol, m.p. $55.5-56^{\circ}$, b.p. $111-112^{\circ}/11-12$ mm.; and bromomethinyl-diethylcarbinol, m.p. $16-18^{\circ}$, b.p. $84-85^{\circ}/13-14$ mm., is described. C. HOLLINS.

Photochemical process [of polymerisation]. H. S. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,746,168, 4.2.30. Appl., 28.5.28).—Polymerisable compounds, e.g., olefines, vinyl compounds, resinous or rubber-forming compounds, etc., are prefer-

ably mixed or maintained in contact with a metallic vapour in the presence of the liquid or solid phase of the metal (e.g., mercury) and subjected to the resonance radiation of that metal. II. S. GARLICK.

Manufacture of polymerisation products of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,894, 16.5.29).—Rubber substitutes are obtained by polymerising butadienes at 70° in presence of hydrazine derivatives, e.g., $\alpha\alpha'$ -hydrazoisobutyric acid, ethyl hydrazinedicarboxylate, phthalhydrazide, *s*-benzoylacethydrazide, benzhydrazide, or hydrazine hydrazinedicarboxylate. C. HOLLINS.

Manufacture of soluble polymerisation products from diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,872, 15.3.29).—Diolefines (butadiene, isoprene) are treated at $60-100^{\circ}$ with alkali metals dispersed in an organic solvent (decahydronaphthalene, xylene). The products are viscous or tenacious masses, only slightly elastic, and soluble in all proportions in hydrocarbon or ester solvents. C. HOLLINS.

Concentration of aqueous acetic or formic acid. IMPERIAL CHEM. INDUSTRIES, LTD., and F. D. LEICESTER (B.P. 332,983, 17.5.29).—Acetic acid is selectively adsorbed when the vapour of the dilute acid is passed over active carbon at $120-130^{\circ}$, and may be recovered in concentrated form (96%) by heating the carbon to 200° . For formic acid the adsorption occurs at $100-110^{\circ}$. Reduction of pressure assists the recovery of either acid. The selective adsorption may also be accomplished with dilute liquid acids without heat. C. HOLLINS.

Manufacture of glacial acetic acid from dilute acetic acid. I. G. FARBENIND. A.-G. (B.P. 312,173, 21.5.29. Ger., 18.5.28).—Dilute acetic acid is cooled, with vigorous agitation, to -26° (eutectic point of acetic acid-water), the ice crystals are removed, and the concentrated acid (60%) is treated with a substance which forms a double compound from which acetic acid may be regenerated, e.g., carbamide $[\text{CO}(\text{NH}_2)_2, 2\text{AcOH}]$, eutectic point -17° or potassium acetate $(\text{AcOK}, 2\text{AcOH})$, eutectic point -24° . The double compound is frozen out and glacial acetic acid is distilled from the separated product in a vacuum. Preferably the amount of substance added should be less than sufficient for combination with all the acetic acid present, and the mother-liquors are returned to the process. C. HOLLINS.

Carrying out ester condensations. [Synthesis of acetoacetic esters.] DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 311,707, 8.5.29. Ger., 14.5.28).—A 94% yield of ethyl acetoacetate is obtained from ethyl acetate and sodium ethoxide by distilling off as completely as possible during the reaction the alcohol formed. The process is applicable to esters of methyl, ethyl, and propyl alcohols. C. HOLLINS.

Manufacture of stable polymerisation products from vinyl esters. I. G. FARBENIND. A.-G. (B.P. 319,682, 29.5.29. Ger., 26.9.28).—Unchanged vinyl ester, acetaldehyde, acetic acid, etc. are removed and a stable polymeride is obtained by prolonged (several

hours') contact of the polymeride, in the form of raspings, clippings, spun threads, or thin sheets, with water.

C. HOLLINS.

Manufacture of acrylyl chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,079, 14.9.29).— β -Chloropropionyl chloride gives acrylyl chloride in 80–85% yield when the vapour is led over a suitable catalyst (barium chloride, broken clay, alumina) heated at or above the b.p. of the starting material, *e.g.*, 280–300°. The chloropropionyl chloride may, if desired, be distilled through a column charged with the contact material.

C. HOLLINS.

Manufacture of trisubstituted thioureas [thiocarbamides]. I. G. FARBENIND. A.-G. (B.P. 314,542, 27.6.29. Ger., 30.6.28).—Alkaline carbamates derived from primary amines are heated in water or alcohol with a salt of a secondary amine. Dimethylamine methyl-dithiocarbamate yields trimethylthiocarbamide, and from dimethylamine phenyldithiocarbamate *N*-phenyl-*N'*-dimethylthiocarbamide is prepared.

C. HOLLINS.

Manufacture of organic compounds by dehydrogenation. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,975, 5.6.29).—For the dehydrogenation of hydrogenated aromatic hydrocarbons, petroleum fractions, etc., a mixed catalyst is used comprising cobalt, iron, or metals of groups V, VI, and VII, or compounds of these, in admixture with one another or with other substances, the mixed catalyst being pretreated above 200° with hydrides or other compounds of solid non-metals of groups V, VI, and VII. The added substances are preferably lead, tin, zinc, cadmium, or their compounds, or solid oxides of group V non-metals or the corresponding acids or salts. *cyclo*Hexane is converted largely into benzene when the vapour is led at 480° over active charcoal which has been impregnated with ammonium molybdate, lead nitrate, and phosphoric acid and pretreated with hydrogen selenide at 300°. Other catalysts mentioned are: ammonium tungstate, cobalt nitrate, and antimonie acid on active charcoal, treated with hydrogen sulphide at 350°; ammonium vanadate, cobalt, nitrate, and phosphoric acid on active charcoal, treated with hydrogen and carbon disulphide at 350°; uranium oxide and cobalt oxide, treated with hydrogen and carbon disulphide at 350°. Petroleum fractions may be enriched in aromatics, and pentane converted into pentene by the use of such catalysts.

C. HOLLINS.

Manufacture of organic products. [Oxidation by air or oxygen at high pressures.] D. FUTACCHI (B.P. 309,498, 10.4.29. Fr., 11.4.28).—The oxidation of methyl alcohol to glycol, of ethyl and methyl alcohols to propylene glycol, of petroleum to alcohols, aldehydes, and ketones, of paraffins to fatty acids and amino-fatty acids (in presence of ammonia), etc., is carried out by treating the starting material in liquid form or in solution in an inert liquid with air or oxygen at pressures of 150–800 kg./cm.² below 500° in presence or absence of catalysts. *E.g.*, air at 450 kg./cm.² is forced through two similar tubes in series, the second of which contains methyl alcohol heated under pressure to 180°; the first tube provides a safety chamber into which the

liquid flows when the air pressure is released in the event of overheating. The product consists of glycol 40%, methyl alcohol 5%, glycerol 20%, and a viscous mixture of carbohydrates.

C. HOLLINS.

Oxidation of benzene hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 318,550, 3.9.29. Ger., 5.9.28).—The process of B.P. 309,005 (B., 1929, 917) is extended to tetrahydronaphthalenes. The production of α -keto-tetrahydronaphthalene, b.p. 130–135°/15 mm. (with 20% of the α -hydroxy-compound), of α -ketoethyltetrahydronaphthalene (semicarbazone, m.p. 223°) from *ar*-ethyltetrahydronaphthalene, and of *ar*-chloro- α -keto-tetrahydronaphthalene, b.p. 160°/20 mm. (semicarbazone, m.p. 219°), is described.

C. HOLLINS.

Manufacture of polymerisation products of styrene. I. G. FARBENIND. A.-G. (B.P. 307,936, 15.3.29. Ger., 16.3.28).—The polymerisation of styrene is effected in aqueous emulsion in presence of an emulsifying agent or colloidal solution at temperatures not substantially above 60° and without superatmospheric pressure. Styrene is stirred, *e.g.*, with 1.3 pts. of milk for 10 days at 60° to give a resinous polymeride which is elastic at 100°.

C. HOLLINS.

Manufacture of aldehydes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,009, 4.7.29).—The yields of aldehydes by Gattermann's method from carbon monoxide and aromatic hydrocarbons under pressure are increased by adding titanium chloride to the aluminium chloride. The action of chlorine on a titaniferous bauxite and carbonaceous matter gives a suitable catalyst. The preparation of benzaldehyde from benzene at 30–35° and 60 atm., *p*-tolualdehyde from toluene at 35–40° and 60 atm., and *p*-chlorobenzaldehyde from chlorobenzene at 60° and 50 atm., is described.

C. HOLLINS.

Manufacture of arylaminophenolcarboxylic acids. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 333,783, 28.9.29).—The alkali-metal salt of a *m*-hydroxydiphenylamine is heated with carbon dioxide under pressure, *e.g.*, at 150–170° and 5–10 atm. for 16 hrs. The following 4-arylaminoalicyclic acids are described: anilino-, m.p. 180–181°; *o*-toluidino-, m.p. 180–182°; *m*-toluidino-, m.p. 176–177°; *p*-toluidino-, m.p. 185–186°; *o*-anisidino-, m.p. 199–200°; *p*-anisidino-, m.p. 176–177°; *o*-chloroanilino-, m.p. 198°; *m*-chloroanilino-, m.p. 189–190°; *p*-chloroanilino-, m.p. 188–190°; *m*-4-xyldino-, m.p. 175°; *p*-xyldino-, m.p. 175–176°; 6-methoxy-*m*-toluidino-, m.p. 165°; 5-chloro-*o*-toluidino-, m.p. 180–181°; 4-chloro-*o*-toluidino-, m.p. 208–210°; 6-chloro-*m*-toluidino-, m.p. 195–197°; 2-chloro-*p*-toluidino-, m.p. 190°; 2,4-dichloroanilino-, m.p. 215°; 3:4-dichloroanilino-, m.p. 199°; 4:5-dichloro-*o*-toluidino-, m.p. 203°; *p*-phenoxyanilino-, m.p. 189°.

C. HOLLINS.

Manufacture of 2-chlorobenzthiazoles. I. G. FARBENIND. A.-G. (B.P. 310,815, 30.4.29. Ger., 30.4.28).—A 2-thiol- or 2-hydroxy-benzthiazole is treated with phosphoryl chloride or thiophosphoryl chloride (PSCl₂) or with phosphorus pentachloride diluted with either of these. The preparation of 2-chlorobenzthiazole, b.p. 248°, and its 5-methyl (m.p. 40–47°, b.p. 266° or

132°/15 mm.) and 6-nitro- (m.p. 190°) derivatives is described. 6-Nitro-2-anilinobenzthiazole has m.p. 247°.

C. HOLLINS.

Preparation of thymol. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 312,907, 29.4.29. Ger., 1.6.28. Addn. to B.P. 293,753; B., 1928, 837).—In place of propylene in the process of the prior patent propyl or isopropyl alcohol or ester or other compound yielding propylene is employed, preferably in presence of catalysts effecting the conversion into propylene. *E.g.*, *m*-cresol is heated in an autoclave at 350° with isopropyl alcohol, water is distilled off, and the heating repeated with more alcohol; thymol equivalent to 60–70% of the unrecovered *m*-cresol is obtained. If propyl chloride is used as a source of propylene, zinc oxide or other acid absorbent is added.

C. HOLLINS.

Manufacture of C-alkylated phenols. SCHERING-KAHLBAUM A.-G. (B.P. 309,865, 15.4.29. Ger., 16.4.28. Addn. to B.P. 254,753 and 274,439; B., 1928, 740; 1929, 164).—The fission of dihydroxydiphenylmethanes by hydrogen in presence of catalysts is facilitated and nuclear hydrogenation avoided by diluting the hydrogen with 33% of inert gas, preferably steam. The steam is advantageously generated *in situ*, by using the catalyst in the form of oxide. *E.g.*, 4:4'-dihydroxydiphenylpropane is converted by hydrogen at 200° and 20–25 atm. pressure in presence of mixed carbonates or hydroxides of nickel and copper (with traces of cobalt and iron) into *p*-hydroxyisopropylbenzene and phenol.

C. HOLLINS.

Manufacture of condensation products from aldehydes and phenols [mothproofing compounds].

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,561, 11.5.29).—Mothproofing compounds which are not dyes are obtained by condensing the hydrol (etc.) prepared from an aldehyde (non-hydroxylated, if aromatic) and 1 mol. of a *p*-halogenated phenol or naphthol having a free *o*-position and no salt-forming group, with a second mol. of the same or a similar phenol or naphthol, preferably in presence of sulphuric acid, hydrochloric and acetic acids, or zinc chloride. Examples are: benzaldehyde-*o*-sulphonic acid with 2 successive mols. of 2:4-dichlorophenol, or 2:3-dichlorophenol and *p*-chlorophenol, or 2:4:5-trichlorophenol and 2:4-dichlorophenol; formaldehyde with 2:4-dichlorophenol and *p*-chlorophenol.

C. HOLLINS.

Manufacture of sulphonated condensation products [from phenols and halogenated benzyl halides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,960, 1.5.29. Addn. to B.P. 320,056; B., 1930, 315).—The process of the prior patent, especially when used with polyhalogenated aralkyl halides, is conducted in presence of zinc chloride. The condensation of phenol with trichlorobenzyl chloride and of *p*-chlorophenol with *o*-chlorobenzyl chloride, followed by sulphonation of the products, is described.

C. HOLLINS.

Manufacture of condensation products from aliphatic aldehydes and bases of the naphthalene series [anti-agers]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,941, 24.5.29).—Odourless, finely-divided products, useful as anti-oxidants, are

obtained by condensing aliphatic aldehydes with amines of the naphthalene series in alcohol in presence of acid. *E.g.*, α -naphthylamine and commercial aldol in boiling ethyl alcohol containing a little formic acid yield on precipitation with water a fine powdery resin, m.p. 210°. The product from 1:5-naphthylenediamine and aldol has m.p. 200°; the 1:8-diamine gives a similar product, m.p. 250°.

C. HOLLINS.

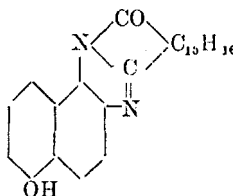
Manufacture of derivatives of naphthalene [acyl-naphthalenes and 4-substituted α -naphthoic acids].

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,666—7, 4.6.29).—(A) Naphthalene, or a 1-aralkyl-naphthalene having a free 4-position, is condensed with an acid chloride in presence of aluminium chloride etc., and the products are oxidised if desired. 1-Benzyl-naphthalene and benzoyl chloride yield phenyl 4-benzyl- α -naphthyl ketone, m.p. 110°, which is oxidised by nitric acid to 1:4-dibenzoylnaphthalene, m.p. 106°. 1-*p*-Chlorobenzyl-naphthalene, b.p. 176–177°/0.2 mm., is similarly converted into phenyl 4-*p*-chlorobenzyl- α -naphthyl ketone, m.p. 123°, and 1-benzoyl-4-*p*-chlorobenzoylnaphthalene, m.p. 118. 4-Benzyl- α -naphthyl methyl ketone, m.p. 78°, from benzyl-naphthalene and acetyl chloride, gives on oxidation 4-benzoyl- α -naphthoic acid, m.p. 184°. (B) 4-Alkyl- α -naphthyl ketones are oxidised, *e.g.*, with nitric acid or alkaline hypochlorite. Phenyl 4-methyl- α -naphthyl ketone gives 4-benzoyl- α -naphthoic acid, m.p. 184°; 4-methyl- α -naphthyl methyl ketone gives 4-methyl- α -naphthoic acid, m.p. 165°, or, with more oxidant, naphthalene-1:4-dicarboxylic acid.

C. HOLLINS.

Manufacture of hydroxy-1':8'-naphthoyleu-naphthiminazoles and sulphonic acids thereof.

I. G. FARBENIND. A.-G. (B.P. 316,143, 22.7.29. Ger., 23.7.28).—The naphthiminazoles obtained by condensing 1:8-naphthalic anhydride with 1:2-naphthylene-diaminesulphonic acids are fused with alkali. The compound from the diamine-5-sulphonic acid gives 6-hydroxy-1':8'-naphthoyleu-naphthiminazole (annexed formula), m.p. 323°. The 8-hydroxy-compound, m.p. 350°, and the 6:8- and 9:7-hydroxy-sulphonic acid are obtained from the iminazoles derived from 1:2-naphthylenediamine-7-sulphonic, -5:7- and -6:8-disulphonic acids, only



the α -sulphonic groups in the disulphonic acids being hydrolysed. The products have pharmaceutical application.

C. HOLLINS.

Manufacture of derivatives of the naphthoyleu-diaryliminazole series [from naphthalenetetracarboxylic acid and *o*-diamines]. I. G. FARBENIND. A.-G. (B.P. 334,111, 22.10.29. Ger., 22.10.28).—The bisiminazoles obtained by condensing 1:4:5:8-naphthalenetetracarboxylic anhydride with *o*-diamines (*o*-phenylenediamine, 3:4-diaminophenetole, 1:2-naphthylenediamine) are nitrated and reduced (with sodium sulphide or in the vat). The iminazoles from the three diamines mentioned yield blue-grey to black, violet-brown, and olive-green vat dyes, respectively.

C. HOLLINS.

Manufacture of derivatives of quinoline. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,911, 25.3.29).—Arylamines are condensed with esters of malonic acids in a high-boiling solvent (nitrobenzene, excess of the malonic ester), the alcohol formed being distilled off during the reaction. 2:4-Dihydroxyquinolines from α -naphthylamine and *m*-toluidine are described.

C. HOLLINS.

Manufacture of derivatives of benzanthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,568, 15.2.29).—Halogen, nitro-, or hydroxyl substituents introduced by known methods into a 3-alkoxy-benzanthrone enter position 2. The preparation of the following compounds is described: 2-nitro-1-methoxybenzanthrone, m.p. 237°, by nitration with nitric acid in nitrobenzene at 45–50°; 2-bromo-1-methoxybenzanthrone, m.p. 186–188°; 2-chloro-1-ethoxybenzanthrone, m.p. 140–141°; 2-chloro-1-methoxybenzanthrone, m.p. 183–185°; 2:3-dihydroxybenzanthrone, m.p. 335° (dimethyl ether, m.p. 156–158°), from 3-methoxybenzanthrone by oxidation with manganese dioxide and sulphuric acid at 5–10°, followed by reduction with bisulphite; 9-chloro-2-nitro-3-methoxybenzanthrone, m.p. 288–290°, from 9-chloro-3-methoxybenzanthrone; 2:9-dinitro-3-methoxybenzanthrone, m.p. 290–291°, from 9-nitro-3-methoxybenzanthrone; 11-chloro-2:9-dinitro-3-methoxybenzanthrone, m.p. 285°, and the more soluble 2-mononitro-compound, m.p. 232–235°, from 11-chloro-1-methoxybenzanthrone.

C. HOLLINS.

Production of intermediates for vat dyes. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 332,907, 23.1.29).—Monosulphuric esters of leucoanthraquinones are obtained by removal of one ester group from a disulphuric leuco-ester by means of zinc and aqueous sodium hydroxide or of hydrochloric acid. Alternatively, a mixed sulphuric and organic ester of the leuco-compound may be hydrolysed to the monosulphuric ester, *e.g.*, with dilute sodium hydroxide solution. In the examples the starting materials are anthraquinone, 1- or 2-acetamidanthraquinone, and 2-chloroanthraquinone.

C. HOLLINS.

Carrying out chemical reactions under pressure. [Manufacture of anthrarufin.] J. F. WAIT, ASSR. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,746,687, 11.2.30. Appl., 18.12.24).—In the manufacture of anthrarufin by hydrolysis of anthraquinonesulphonic acid in an autoclave under pressure, the rise in pressure and temperature is obtained by means of injected steam.

B. M. VENABLES.

Pyrolysis of unsaturated hydrocarbons (B.P. 332,998).—See II. **Leuco-esters of aminoanthraquinones etc.** (B.P. 333,507).—See IV. **Wetting agents etc.** (B.P. 310,941).—**Reserving agents for wool** (B.P. 333,559). **Colouring of higher fatty acids** (B.P. 309,148).—See VI. **Sodium salicylate** (U.S.P. 1,755,362).—See VII. **Methyl α -hydroxyethyl ketone and diacetyl** (B.P. 315,264).—See XVIII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of dyes containing metals. Soc. CHEM. IND. IN BASLE (B.P. 311,385, 10.5.29. Switz.,

10.5.28).—The prechromed dyes of B.P. 301,772 (B., 1929, 165) are treated with solutions of salts of copper, cobalt, nickel, tungsten, uranium, aluminium, tin, titanium, or molybdenum, whereby a second metal is introduced. The chromium compound of nitro-1:2:4-aminonaphtholsulphonic acid \rightarrow β -naphthol gives two-metal complexes with copper sulphate, tungsten trioxide in sodium acetate, sodium uranate, cobaltous chloride, nickel sulphate.

C. HOLLINS.

Manufacture of [thioindigo] vat dyes. I. G. FARBENIND. A.-G. (B.P. 309,379, 8.4.29. Ger., 7.4.28).—Violet-blue to blue vat dyes are obtained by condensing substituted thioindoxyl with 5- or 7-alkoxyisatin α -halides etc. which contain further substituents. Examples are: 7-methoxy-4-methylisatin α -chloride with 5-chloro-7-methylthioindoxyl; 5-chloro-7-methoxy-4-methylisatin α -chloride with 5-chloro-7-methyl-5-chloro-4:7-dimethyl-, 5:7-dichloro-4-methyl-, 7-chloro-5-methyl-, 5-chloro-, or 5:6:7-trichloro-thioindoxyl; 7-chloro-5-methoxyisatin α -chloride with 5:6:7-trichlorothioindoxyl; 4-chloro-7-methoxyisatin α -chloride with 5-chloro-, 5:7-dichloro-, 5:6:7-trichloro-, 5:7-dichloro-4-methyl-, or 5-chloro-4:7-dimethyl-thioindoxyl.

C. HOLLINS.

Production of dry powders of anthraquinone acetate silk dyes. J. S. WILSON, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,236, 2.2.29).—Insoluble anthraquinone dyes for acetate silk are mixed with an alkali soap and dried below 50°. The state of dispersion of the dyes is thereby preserved.

C. HOLLINS.

Preparation of water-soluble dyes of the anthraquinone series. CHEM. FABR. VORM. SANDOZ (B.P. 333,188, 4.5.29. Ger., 27.11.28).—A 2-bromo-1-amino-4-*p*-acetamidoanilinoanthraquinone is heated with a sulphite, water, and phenol at 140°, whereby the bromine is replaced by a sulphonic group without hydrolysis of the acetamido-group. The treatment of the *p*-acetamido- and *p*-acetmethylamido-compounds is described.

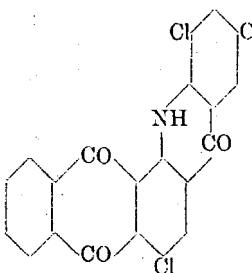
C. HOLLINS.

Manufacture of dianthraquinonylamines of the anthraquinoneacridone series [vat dyes]. I. G.

FARBENIND. A.-G. (B.P. 311,283, 7.5.29. Ger., 8.5.28).—An anthraquinone-1:2-acridone, halogenated in the 4-position of the anthraquinone residue as well as in the benzene residue, *e.g.*, the trichloro-compound having the annexed formula, is condensed with an aminoanthraquinone, *e.g.*, 1-amino-4- or 5-benzamidoanthraquinone, or α -aminoanthraquinone, to give grey vat dyes.

C. HOLLINS.

Manufacture of condensation products of the anthraquinoneacridone series. I. G. FARBENIND. A.-G. (B.P. 314,899, 3.7.29. Addn. to B.P. 311,283; preceding).—The process of the prior patent is extended to the condensation of the halogenated anthraquinone-acridones there specified with halogenated arylamines of the benzene or naphthalene series; *e.g.*, 2:5- and



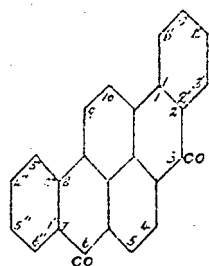
3 : 5-dichloro- and 3 : 4 : 5-trichloro-anilines give with trichloroanthraquinoneacridone bluish-green vat dyes.

C. HOLLINS.

Manufacture of green vat dyes of the [di]benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,762, 4.9.29).—Pure dimethoxydibenzanthrone obtained according to B.P. 181,304 (B., 1922, 582 A) is chlorinated with sulphuryl chloride in nitrobenzene at 80–90° in presence of ferric chloride, and, if desired, an acid-fixing agent (calcium oxide, magnesia). The products are brilliant green vat dyes.

C. HOLLINS.

Manufacture of vat dyes [dibenzpyrenequinones] containing halogens. I. G. FARBENIND A.-G., and J. Y. JOHNSON (B.P. 333,165, 22.4.29. Addn. to B.P. 287,050 and 287,845; B., 1929, 590, 591).—1 : 2 : 7 : 8-



Dibenzpyrene-3 : 6-quinones (other than iodo-derivatives) are halogenated in organic media (nitrobenzene) or in aqueous suspension or in absence of solvents, preferably in presence of halogen carriers (iron, iodine). The products dye redder shades than those of the prior patents, and are often pure red. The halogens probably enter positions 3', 4', 6' and 3'', 4'', 6'' (annexed formula) instead of at 5' and

5''. The preparation of chloro-, bromo-, chlorobromo-, dichloro-, and dichlorodibromo-derivatives is described.

C. HOLLINS.

Production of dyes and dye intermediates [azo dyes from diazotised leuco-esters of aminoanthraquinones or amino-vat dyes]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,507, 8.2.29).—The sulphuric esters of leucoaminoanthraquinones or leuco-vat dyes containing amino-groups are diazotised by treatment with sodium nitrite and acid below 5°. The diazo compounds may be coupled with coupling components to give azo dyes still containing the leuco-ester residue. Examples are the following (the amine being used in the form of its leuco-ester): β -aminoanthraquinone \rightarrow β -naphthol, 2 : 3-hydroxynaphthoic 5-chloro-*o*-toluidide, anilide, etc.; 1 : 5-amino-naphthoic acid, phenol, resorcinol, acetoacetic toluidide; 3-chloro-2-aminoanthraquinone \rightarrow β -naphthol, carbonyl-J-acid, γ -acid, cresidine \rightarrow phenyl-J-acid, H-acid, *m*-phenylenediamine \leftarrow 4-nitro-*o*-aminophenol, 2 : 3-hydroxynaphthoic *p*-anisidide, β -naphthylamide, etc.; 2 : 6-diaminoanthraquinone, or α -aminoanthraquinone, or 1-chloro-2-aminoanthraquinone, or 5 : 5'-dichloro-7 : 7'-diaminothioindigo \rightarrow 2 : 3-hydroxynaphthoic 5-chloro-*o*-toluidide (cf. also B.P. 333,506; B., 1930, 1024).

C. HOLLINS.

Azo dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 333,513, 11.4.29. Addn. to B.P. 330,607; B., 1930, 897).—In place of 3 : 4'-diaminoazobenzene in the process of the prior patent there is used a 3-aminobenzene-1-azo-4'-aminonaphthalene. Examples are: *m*-nitroaniline \rightarrow α -naphthylamine \rightarrow salicylic acid, reduced, \rightarrow γ -acid (deep brown on viscose silk); 4-nitro-*o*-toluidine \rightarrow Cleve acid \rightarrow β -naphthol, reduced,

\rightarrow 2S-acid (blue); 4-nitro-*o*-anisidine \rightarrow Cleve acid \rightarrow *o*-cresotic acid, reduced, \rightarrow benzoyl- γ -acid (brown).

C. HOLLINS.

Manufacture of monoazo dyes for wool. I. G. FARBENIND. A.-G. (B.P. 311,708, 10.5.29. Ger., 14.5.28).—*p*-Nitroaniline-*o*-sulphonic acid is diazotised and coupled with an alkylated naphthylaminesulphonic acid in which the alkyl group contains more than two carbon atoms, e.g., isobutyl-2-naphthylamine-7-sulphonic acid for a blue-violet fast to washing, fulling, and light.

C. HOLLINS.

Manufacture of substantive *o*-carboxyazo dyes containing copper. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,573, 10.5.29).—Azo dyes, obtained by coupling tetrazotised benzidine-3 : 3'-dicarboxylic acid with arylated or aroylated amino-naphtholsulphonic acids in which the aryl or aroyl residue carries a solubilising group, are converted into their copper complex compounds. The coupling may be made in the presence of a coppering agent (except cuprammonium compounds), or the latter may be added to the dye bath. Examples of coupling components are: *p*-5-hydroxy-7-sulpho-2-naphthylaminophenoxyacetic acid (violet-blue), the corresponding phenylthiolacetic acid (blue), 5-sulpho-*o*-anisyl-J-acid (violet), ω -sulpho-*p*-tolyl-J-acid, *m*-carboxyphenyl-J-acid (blue-violet), *o*-carboxybenzoyl-J-acid (violet), *p*-5-hydroxy-7-sulpho-2-naphthylaminophenylacetic acid (violet-blue), the aminophenoxyacetic acids containing residues of J- and γ -acids (reddish-grey), 2-*p*-sulphophenoxyphenyl-J-acid (bluish-violet), *p*- β - γ -dihydroxypropoxyphenyl-J-acid (reddish-blue).

C. HOLLINS.

Manufacture of dyes [of the azophthalein series]. J. R. GEIGY A.-G. (B.P. 312,330, 24.5.29. Ger., 24.5.28).—Azo couplings of resorcinol, especially with diazotised *o*-aminophenols, are condensed with 4-dialkylamino-2-hydroxybenzoyl-*o*-benzoic acids in sulphuric acid at 70° to give azophthaleins. Examples are: 4-dimethylamino-2-hydroxybenzoyl-*o*-benzoic acid with 3-amino-*o*-cresol-5-sulphonic acid \rightarrow resorcinol (reddish-blue on wool, blue on chroming) or with 4-chloro-*o*-aminophenol-6-sulphonic acid \rightarrow resorcinol (similar), or with anthranilic acid \rightarrow resorcinol (chromed brown-red on wool); 4-diethylamino-2-hydroxybenzoyl-*o*-benzoic acid with 1 : 2 : 4-aminonaphtholsulphonic acid \rightarrow resorcinol (greenish-blue).

C. HOLLINS.

Manufacture of water-insoluble azo dyes on the fibre [ice colours]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 332,940, 28.3.29).—Whilst 2 : 4-dihydroxyquinoline gives valueless dyeings when coupled on the fibre, the presence in the quinoline molecule of further fused rings or of arylazo or arylazimino-substituents, or the direct linking together of two 2 : 4-dihydroxyquinolyl residues, confers improved affinity for the fibre, and such quinoline derivatives give useful ice colours. Examples are: 2 : 2' : 4 : 4'-dihydroxy-6 : 6'-diquinolyl with diazotised 1-aminoanthraquinone (brown-red) or *o*-aminodiphenyl ether (orange-yellow); 2 : 4-dihydroxy- α -naphthaquinoline with diazotised 2 : 5-dichloroaniline (brown-orange) or aminoazotoluene (yellow-red); 1 : 3-dihydroxy- β -naphthaquinoline, m.p. 345°, with diazotised 5-nitro-*o*-toluidine (red-orange) or

1-aminoanthraquinone (red-orange); 2:4-dihydroxy- $\beta\beta$ -naphthaquinoline, m.p. 355°, with diazotised 2:5-dichloroaniline (orange-yellow); 2-(2:4-dihydroxy-6-quinolyl)benzotriazole, m.p. 330°, with diazotised 4:6-dichloro-*m*-toluidine (red-yellow) or 4-chloro-*o*-anisidine (yellow-orange); 2:4-dihydroxy-6-quinolineazo-2-chlorobenzene, m.p. 345° (prepared from *o*-chloroaniline \rightarrow anthranilic acid and ethyl malonate), with diazotised 4:6-dichloro-*m*-toluidine (golden-yellow) or 2:5-dichloroaniline (pure yellow).
C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 332,932, 21.3.29. Cf. B.P. 331,247; B., 1930, 942).—2:3-Hydroxynaphthoic 4-chloro-2:5-diethoxyanilide is coupled with a non-carboxylated and nonsulphonated diazo or tetrazo compound in substance or on the fibre. The dyeings are fast to light and to bucking with 0.2% sodium hydroxide for 6 hrs. without pressure. Examples of diazo components are 2:5-dichloroaniline (orange-brown), 4-chloro-*o*-toluidine (yellow-red), 2:5-dichloro-*p*-toluidine (brick-red), 4:6-dichloro-*m*-toluidine (brown-red).
C. HOLLINS.

Manufacture of vat dyes and intermediates derived from pyranthrone. A. SHEPHERDSON and A. J. HAILWOOD, ASSTS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,200, 16.9.30. Appl., 5.5.27. U.K., 7.7.26).—See B.P. 278,102; B., 1927, 901.

Chromium compounds of azo dyes. H. KÄMMERER and K. HOLZACH, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,775,644, 16.9.30. Appl., 17.2.28. Ger., 6.8.25).—See B.P. 297,722; B., 1928, 888.

Naphthoylenediaryliminazole derivatives (B.P. 334,111).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Silk-soaking process. I. Effect of soap and other alkali on silk sericin. R. HART. II. **Soap and oil as soaking agents.** R. HART and G. W. SEARELL (Ind. Eng. Chem., 1930, 22, 980—983, 984—985).—I. On soaking raw silk in a solution of soap there is a marked decrease in the concentration of fatty material and particularly of alkali in the solution, which is due to absorption by the silk and to interaction between the silk sericin and the alkali of the soap. At the same time a large amount of free fatty acid is liberated, which is found both in the treated silk and in the spent liquor. The quantitative distribution of the ingredients after soaking is tabulated. It appears that a small but constant fraction of the sericin is capable of reacting with an excess of alkali in the cold, its combining weight being probably of the order of that of fatty acids.

II. The composition of the liquor and soaked silk was examined after soaking raw silk in an emulsion of neat's-foot oil and soap; the fatty matter absorbed by the silk consisted of almost equal parts of free fatty acid and soap.
F. R. ENNOS.

Study of silk fibroin in the dispersed state. M. HARRIS and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 965—967).—Colloidal suspensions of silk fibroin, prepared by grinding in the pebble mill, extracting with

water, and removing the larger particles by centrifuging, contain no amino-nitrogen, are immune to bacterial attack, are digested by trypsin, and have $[\alpha]_D^{20}$ —43.98°. When the protein is colloiddally dissolved by means of concentrated aqueous solutions of neutral salts (50% lithium bromide or 70% calcium thiocyanate) and subsequent dialysis to remove the salts, a partial hydrolysis independent of temperature occurs, as is shown by the conversion of about 5% of the total nitrogen into the amino-form, the power of the fibroin to support bacterial growth, and a change of the rotation towards dextro-rotation.
F. R. ENNOS.

Inhibitory action of certain substances on the growth of mould fungi [on cotton goods]. R. G. FARGHER, L. D. GALLOWAY, and M. E. PROBERT (J. Text. Inst., 1930, 21, T245—260; cf. Morris, B., 1927, 470).—Many inorganic and organic substances have been prepared and examined in order to find an ideal antiseptic (the properties of which are defined) for use in the cotton industry. With the exception of those of thallium, the salts of different metals show no outstanding toxicity. Arsenic, antimony, selenium, and thallium in organic combination are ineffective, whilst boron is somewhat better. The investigation of organic compounds has been developed along three lines of attack: acetanilide and its derivatives, anilides of acids other than acetic acid, and the organo-mercury compounds. The introduction of substituents into acetanilide leads to a fall in toxicity which is slight for methyl (*o* or *p*), chlorine (*p*), or nitro- (*p*), and marked for hydroxyl (*p*) or *N*-methyl. The toxicities of acet- β -naphthylamide and 8-acetamidoquinoline are similar to that of acetanilide, whilst acet- α -naphthylamide, 5-acetamidoquinoline, and 2-acetamidopyridine are relatively toxic. Acet-*o*-toluidide, acet- α -naphthylamide, and 5-acetamidoquinoline, which contain a carbon substituent *ortho* to the acetamido-group, are less toxic, respectively, than acet-*p*-toluidide, acet- β -naphthylamide, and 8-acetamidoquinoline. Combination of aniline with other acids yields only one promising substance, salicylanilide, but this is as toxic as thallium carbonate, fulfils all the conditions for the ideal antiseptic (except for its slight solubility), and its use is patented under the name "Shirlan." It is a poor bactericide, but appears to be non-toxic to plant life. Introduction of mercury into the nucleus of phenol gives rise to two monosubstituted derivatives, and the more soluble of these, tested as the *o*-chloromercuriphenol, is seven times as toxic as mercuric chloride, whilst the less soluble isomeride is only as toxic as the latter. The order of decreasing toxicity with substituent groups is given, and it is found that solutions of these mercury compounds are much more stable to metals than are the inorganic mercury salts. The outstandingly toxic substances found are salicylanilide, *o*-chloromercuriphenol, *p*-acetoxymercuriacetanilide, thallium carbonate, *p*-nitrophenol, and trichlorophenol. The organisms and methods of testing employed, the preparation of salicyl derivatives by means of salicyl chloride and salol, and of the mercury derivatives of phenol, the mercuriation of aniline and *p*-toluidine, and the chemical characteristics of many of the compounds prepared are described.
B. P. RIDGE.

Swelling of cotton hairs in water and in air at various relative humidities. G. E. COLLINS (J. Text. Inst., 1930, 21, T311—315).—A close parallelism exists between the extent of the dimensional changes of cotton hairs and the amount of absorption between the temperature limits 20° and 100°. No evidence of increased swelling at temperatures up to 200° has been obtained.

B. P. RIDGE.

Extensibility of cotton hairs. G. E. COLLINS (J. Text. Inst., 1930, 21, T316—324).—The extension and recovery of single hairs from a sample of cotton previously treated with sodium hydroxide solution to smooth out the convolutions have been observed at various humidities from zero to 100%. Extensibility increases considerably with increase in humidity, and is about 8—10 times as great at saturation as at zero humidity. The hair makes a more complete recovery from a given extension at the higher humidity. On the whole, fine hairs extend rather less than coarse ones. Absolute values for Young's modulus range from 6×10^{10} dynes/cm.² at zero humidity to 0.6×10^{10} dynes/cm.² in water. A close correlation is obtained between the percentage extension imposed at a given humidity and the subsequent recovery.

B. P. RIDGE.

Length changes of cotton hairs in solutions of caustic soda. M. A. CALVERT (J. Text. Inst., 1930, 21, T293—308).—The shrinkage of single cotton hairs, not previously submitted to a swelling treatment, when immersed in 26.8% sodium hydroxide solutions has been investigated under various conditions for raw, scoured, chloroform-extracted, and mechanically treated (rubbed) hairs. The results obtained are expressed as curves showing the relation between percentage contraction and concentration of the immersion liquid. Scoured hairs show a maximum "free" shrinkage (*i.e.*, shrinkage without loading) near to 13% concentration, whilst the shrinkage of raw hairs under these conditions is much less, and the peak of 13% concentration is absent. Restricted shrinkage (shrinkage under continuous load) for scoured hairs gives curves similar to those for raw cotton hairs under free shrinkage. The restricted shrinkage of hairs extracted with chloroform is not increased by the removal of fats and waxes, whilst the severity of the scouring treatment has no effect on free shrinkage, since the latter is as great for water-boiled hairs as for those scoured with caustic soda solution under pressure. Mechanical treatment of the hairs in such a way as to remove the cuticle increases free shrinkage, which then shows a maximum near to 11% sodium hydroxide concentration. In this respect the cuticle-free hairs resemble bast fibres, which have no cuticle, and show a pronounced maximum for concentrations of 8.8—11%. The effect of removing the cuticle of the cotton hair is also reflected by marked increase in the width of the hairs on treatment with the sodium hydroxide solutions as compared with the corresponding width increases for raw and soda-boiled material. The apparatus and technique employed in making the observations are described.

B. P. RIDGE.

Determination of "soundness" in wool and cloth. C. RIMINGTON (J. Text. Inst., 1930, 21, T237—244).—The mechanism of the chemical reaction between

tyrosine and Pauly's reagent (diazobenzenesulphonic acid), which forms the basis of the method elaborated, is discussed. A weighed sample of the wool is treated with the reagent under standardised conditions, dissolved in 10% sodium hydroxide solution, and the colour intensity of the resulting liquid is matched against that of a 0.1% solution of the dye New Acid Brown S. The degree of damage of the sample is expressed numerically in such a way that 100 units of damage represent the extent of unsoundness present when 0.1 g. of the wool in question, after the above treatment, yields a solution the colour intensity of which is exactly equal to that of the standard solution of dye. The factors which influence the method are examined, and a simplified procedure for use in works is described.

B. P. RIDGE.

Mechanism of the deformation of fibres. K. ECKLING and O. KRATKY (Naturwiss., 1930, 18, 461—464).—The mechanism of the process of deformation of a cellulose ester thread and of the chemical changes which are involved during its conversion into cellulose are discussed, having regard to changes in the lattice arrangement, slip of the molecules parallel to their axis but without orientation, irreversible changes of the degree of dispersion, and the conditions governing the extension of a system of rod-shaped bodies immersed in a viscous medium. Threads subjected to but slight tension do not on hydrolysis of the ester exhibit any marked increase of the orientation of the particles, but threads which have been extended by a considerable force exhibit markedly greater orientation after hydrolysis; the cause of this phenomenon is, however, obscure.

H. F. GILLBE.

Cooking process. II. Cooking wood with sodium carbonate. S. I. ARONOVSKY and R. A. GORTNER (Ind. Eng. Chem., 1930, 22, 941—945; cf. B., 1930, 552).—Aspen sawdust was cooked with 20% and 40% sodium carbonate solutions (dry weight) at 170° and 186° for 2 and 12 hrs. under 100—150 lb. steam pressure. Comparison of the composition of the resulting residues and black liquors with that of those obtained when water alone was used showed that sodium carbonate cannot be regarded as an inert ingredient in a cooking liquor. The residual woods obtained after the sodium carbonate treatment were lighter in colour, softer, and contained more cellulose and less α -cellulose, whilst the liquors after cooking were richer in total organic matter, lignin, and volatile acids than the corresponding fractions obtained by cooking with water; the more stable pentosans associated with the cellulose were not removed by the sodium carbonate cook.

F. R. ENNOS.

Effect of fine division on the solubility of cellulose. D. F. J. LYNCH (Ind. Eng. Chem., 1930, 22, 952—953).—The solubility of cellulose, *e.g.*, peanut-hull pulp, cottonwood pulp, etc., in cold concentrated (17.5%) sodium hydroxide solution (α -cellulose test) is not materially affected by the state of division, but in hot dilute (7.14%) sodium hydroxide (nitrators' alkali-soluble determination) a very fine degree of division increases the solubility, although the mere cutting of fibres into short lengths has little effect.

F. R. ENNOS.

Relationship between α -cellulose content and potassium hydroxide solubility of certain

degraded celluloses. H. LEB. GRAY, C. J. STAUD, and J. T. FUESS (Ind. Eng. Chem., 1930, 22, 1018—1020).—Determinations were made of the α -cellulose content and solubility in hot 10% potassium hydroxide solution of a series of oxidised celluloses and hydrocelluloses prepared by the action of acid potassium permanganate and hydrochloric acid solutions, respectively. Down to 75% of α -cellulose the elliptical curves showing the relationship between the α -cellulose content (x) and the potassium hydroxide solubility (y) are identical for both series of degraded celluloses, and are expressed by $y^2 = 697.2 + 45x - 0.526x^2$.

F. R. ENNOS.

Viscosity and strength of the jelly and film of acetylcellulose. T. ARAKI and M. KUSAGAWA (J. Soc. Chem. Ind., Japan, 1930, 33, 267B).—The jelly strength of the reversible gel of acetylcellulose (8 varieties) and benzyl alcohol, and the viscosity of dopes prepared from the former, were measured. The jelly strength of 10% gel at 10° became nearly constant after 5–6 hrs.' cooling, and the values of various samples from the same manufacturers were inversely proportional to the viscosity. The quality of the acetylcellulose cannot be evaluated exactly from the tensile strength (6–7 kg./mm.²) of films obtained by drying the cellulose solutions on a glass plate.

E. LEWKOWITSCH.

Determination of the viscosity of cuprammonium solutions of cellulose. J. TANKARD and J. GRAHAM (J. Text. Inst., 1930, 21, T260—266).—Sufficient cellulosic material to give a 1, 1.5, or 2% solution (according to the extent of chemical modification of the cellulose) is placed in a glass tube, 28 cm. long and 1 cm. internal diam., which is closed at one end and contains a weight formed from a short piece of steel rod. Cuprammonium solution containing copper 15 g., ammonia 240 g., and nitrite <0.5 g. per litre is admitted and the tube is closed with a special stopper and slowly rotated end-over-end for several hours. The measurement of viscosity is carried out at constant temperature (25°) by the falling-sphere method, using steel balls $\frac{1}{8}$ in. in diam., and the following advantages are claimed: accurate determinations of viscosity can be made over a wide range; Ladenburg's correction for wall effect is applicable, thus obviating the necessity for calibrating each tube; small variations in the diameter of the tube are unimportant; duplicate measurements can be made on each solution; and the special stopper ensures that the volume of solution for any one tube is constant in all experiments. Methods previously employed for the determination of the viscosity of solutions of cellulose in cuprammonium are discussed.

B. P. RIDGE.

Recovery of viscose spinning bath. I. Rapid analysis of sodium sulphate in the spinning bath. K. TANEMURA and S. MIYOSHI. II. Sp. heat of Glauber's salt. K. TANEMURA (J. Soc. Chem. Ind. Japan, 1930, 33, 323—324 B, 324—325 B).—I. The temperature at which sodium sulphate begins to crystallise out is observed, its connexion with the sodium sulphate content having been previously determined and graphed.

II. The sp. heat of sodium sulphate decahydrate is calculated from vapour pressure measurement and other data to be 0.42.

C. W. GIBBY.

Higher orientation in cellulose materials. I. K. HESS and C. TROGUS (Naturwiss., 1930, 18, 437—441).—The orientation of the cellulose molecules in a variety of substances such as paper, cellophane, vulcanised fibre, and presspahn has been studied by means of X-ray photographs. The alterations of the mechanical properties of such materials by calendering and analogous processes are correlated with the changes of molecular orientation.

H. F. GILLBE.

Higher orientations in cellulose materials. II. X-Ray diagram of paper. K. HESS and C. TROGUS (Z. physikal. Chem., 1930, B, 9, 169—172; cf. preceding abstract).—From the X-ray examination of paper made from ordinary and mercerised cellulose and of kaolin it has been shown that in paper cellulose has a higher degree of orientation than in the form of fibres, and that when kaolin is used as a filler it, too, is oriented.

R. CUTHILL.

Automatic recording waterproof tester. A. L. HODGES (Phil. Mag., 1930, [vii], 10, 327—328).—A device, simple in principle, is described which accurately records the time taken by water to seep through various fabrics and papers.

W. GOOD.

Fine structure of artificial silks. HALL.—See VI. Fermentation of cornstalks. BORUFF and BUSWELL.—See XVIII. Decomposition of photographic films. OLSEN and others.—See XXI.

PATENTS.

[Shaped] bodies from fibrous materials. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 312,178, 21.5.29. Ger., 21.5.28).—In the manufacture of billiard or skittle balls etc., strips of woven fibrous material are compressed together with artificial resin, e.g., phenol-formaldehyde condensation products, and subjected to heat and pressure.

H. ROYAL DAWSON.

Humidity-control apparatus [for fabrics]. H. A. BRITAIN, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,757,931, 6.5.30. Appl., 17.7.25).—Samples of fabric are brought to a moisture content of 6% or other definite figure by suspending them in a current of air that has been bubbled through sulphuric acid of dilution requisite to give the necessary relative humidity, irrespective of the original condition of the air.

B. M. VENABLES.

Manufacture of cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 315,278, 10.7.29. U.S., 10.7.28).—Cellulosic material is treated with an alkylating or aralkylating agent (ethyl sulphate) in the presence or absence of a small quantity of an alkaline material (sodium carbonate or hydroxide) for several hours at 0–45°, and afterwards with caustic soda in the solid form or in concentrated solution, the temperature being maintained at 30–60° until the cellulose ether is formed.

F. R. ENNOS.

Manufacture of silk from acylcellulose. I. G. FARBENIND. A.-G. (B.P. 312,671, 29.5.29. Ger., 30.5.28. Addn. to B.P. 233,342; B., 1925, 985).—The material is spun into a salt solution (ammonium nitrate), to which is added an inorganic salt (ammonium thiocyanate, zinc chloride) having a swelling or solvent action on the

acetylcellulose, together with sugar and a small quantity of a solvent (acetone), if desired. F. R. ENNOS.

Production of filaments, threads, yarns, ribbons, etc. from cellulose esters and ethers. BRIT. CELANESE, LTD., and (A) W. I. TAYLOR and R. P. ROBERTS, (B) H. DREYFUS, W. I. TAYLOR, and R. P. ROBERTS (B.P. 334,195 and Addn. B.P. 334,198, [A] 28.3.29, [B] 21.5.29).—Products of relatively low lustre are made by extruding solutions of cellulose esters or ethers in (A) a volatile solvent (acetone) or (B) a mixture of a volatile solvent and a diluent (water) into an evaporative medium which is at a higher temperature than the spinning solution, the latter being maintained at 10–20° below the minimum temperature required for the production of products of normal high lustre; the temperatures of both spinning solution and evaporative medium may be separately controlled by the methods of B.P. 320,632 (B., 1930, 53). F. R. ENNOS.

Treatment of materials containing cellulose esters. BRIT. CELANESE, LTD. (B.P. 316,521 and Addn. B.P. 318,468, [A] 29.7.29, [B] 2.9.29. U.S., [A] 28.7.28, [B] 1.9.28).—Fabrics, yarns, etc. of cellulose esters are superficially saponified by application of (A) a paste consisting of caustic alkali and a thickening agent (gum tragacanth, starch, etc.) or (B) a solution containing caustic alkali, after which they are immediately dried by heat and thoroughly steamed or washed, whereby the temperature at which they may be safely ironed or calendered is raised. F. R. ENNOS.

Compositions or materials comprising cellulose derivatives [of reduced inflammability]. BRIT. CELANESE, LTD. (B.P. 312,609, 27.5.29. U.S., 28.5.28).—Plastic or celluloid-like masses, moulding powders, films, lacquers, artificial fibres, etc. having a basis of cellulose esters or ethers are rendered less inflammable by incorporating therein, at any convenient stage, one or more aliphatic acyl derivatives of nuclear-chlorinated arylamines of the benzene series, *e.g.*, *o*-chloroacetanilide. F. R. ENNOS.

Treatment of cellulosic fibres with alkali. G. TAGLIANI, Assr. to CHEM. WORKS, FORMERLY SANDOX (U.S.P. 1,776,052, 16.9.30. Appl., 7.9.27. Ger., 29.10.26).—See B.P. 279,784; B., 1928, 228.

[Photographic] manufacture of wallpapers. J. MACCALLUM and M. O'HARA (B.P. 334,591, 7.6.29).

Copper from ammoniacal solutions (B.P. 313,045 and 310,425).—See VII. **Compound glass** (B.P. 316,955).—See VIII. **Saturant for fibrous bases** (B.P. 334,232).—See XIII. **Rubberised material** (B.P. 332,537).—See XIV. **Cellulose and pulp** (B.P. 332,935).—See XV. **Film-forming element** (U.S.P. 1,746,751). **Photographic paper and films** (B.P. 318,511 and U.S.P. 1,752,665).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fine structure of artificial silks in relation to dyeing and finishing processes. A. J. HALL (J. Soc. Dyers and Col., 1930, 46, 257–261).—The rigidity, creasability, lustre, and dyeing properties of viscose and cellulose acetate fibres are correlated with the

parallelism of their micelle chains and the effect on these of stretching. Methods of measuring these properties are described. Resistance to creasing decreased rapidly for cellulose acetate and less rapidly for viscose silk with increased stretching; a small amount of stretching is thus able seriously to reduce the resistance to creasing of artificial silk materials. The rigidity of artificial silk yarns increased with stretching. Curves show that cellulose acetate and viscose silk yarns gradually lose their power of recovering to their original length as the degree of stretching is increased; viscose yarn shows less power than cellulose acetate yarn to recover. It is considered that parallelism of the micelle chains in viscose fibres as produced by stretching at the time of spinning or during dyeing reduces the affinity of the fibres for direct dyes (cf. Hall, B., 1929, 430); parallelism also gives increased lustre. The retention of lustre by cellulose acetate silk when immersed in a stretched condition in boiling water is ascribed to the effect of the stretching in maintaining the cellulose acetate micelle chains parallel (cf. B.P. 277,089; B., 1929, 596). Greater parallelism of the micelle chains confers increased rigidity and creasability on both types of silks. A. J. HALL.

Diagnosis of colour faults in finished [wool] goods. F. L. GOODALL (J. Soc. Dyers and Col., 1930, 46, 263–267).—Residual alkali in scoured wool materials is frequently responsible for the appearance of various defects in subsequent dyeing processes. Wool containing alkali will, if dyed while still wet, dye a much fuller shade than wool free from alkali; the difference in shade is less marked if the wool is dried before dyeing. Alkaline moist wool is readily attacked by bacteria in such a way that it loses its affinity for acid and chrome colours and gains an increased affinity for basic dyes. Stained and off-shade faults may be produced during the steaming and particularly the drying of dyed wool containing residual alkali. The steaming of undyed wool containing alkali increases very considerably its affinity for dyes. Fading faults in dyed wool materials may be caused by the presence of alkali and sulphur dioxide. Thus bisulphite formed by interaction might combine with certain azo dyes to form azo sulphites strikingly different in colour from that of the parent dyes (cf. King, B., 1928, 639, 707; Goodall, B., 1928, 639). Such sulphur dioxide may be derived from the presence in the wool material of sulphur-dyed cotton yarn or wool yarn which has been previously stored, or from paper wrappers made from sulphite wood pulp, or may arise from oxidation of the natural sulphur content of wool itself. Again, under optimum conditions, sodium bisulphite may cause faults by complete reduction of the dye molecule, as in the presence of sodium hyposulphite; Erio Floxine 2GI is susceptible to such attack, and some vat dyes have been found to be affected. Azo sulphites are not so fast to light as are the parent dyes, so that uneven fading of dyed wool may result from the uneven distribution of sodium bisulphite within it. A. J. HALL.

Flash points of mixed solvents. SNELL.—See III. **Length changes of cotton hairs in solutions of caustic soda.** CALVERT.—See V.

PATENTS.

Manufacture of agents for wetting, cleansing, and emulsifying, and for protecting fibre. ORAN-
IENBURGER CHEM. FABR. A.-G. (B.P. 310,941, 3.5.29.
Ger., 3.5.28).—A neutral fat or a fatty acid is condensed
with a hydrocarbon and an organic compound containing
oxygen or sulphur, and sulphonated, in one or more
stages. *E.g.*, a mixture of castor oil, xylene, and acetic
anhydride is treated with chlorosulphonic acid at 27°.

C. HOLLINS.

**Dyeing with the reserving of wool. Manufacture
of reserving agents for wool.** A. CARPMAEL. From
I. G. FARBENIND. A.-G. (B.P. 333,554 and 333,559, [A]
6.5.29, [B] 9.5.29).—(A) Wool in unions is reserved against
direct cotton dyes by adding to the dye-bath an *N*-alkyl-
ated, -aralkylated, or -arylated amide of a sulphonic
or carboxylic acid, *e.g.*, bis-(1 : 2-dichlorobenzene-4-sul-
phonyl)benzidine-2 : 2'-disulphonic acid. (B) Such
amides are made by condensing 2 mols. of an aromatic
sulphonyl or carboxylic halide with 1 mol. of an arylen-
diamine, the reactants being chosen so that the product
contains one or more free sulphonic acid groups, but no
nitro-, hydroxyl, or unsubstituted amino-group, and at
least one of the reactants must contain a substituent
meta to the reacting group. The bis-1 : 2-dichlorobenz-
enesulphonyl derivatives of benzidine-2 : 2'-disulphonic
acid and of 2 : 4-tolylenediamine-6-sulphonic acid are
excluded from the claims. Examples are : bis-(2 : 4-di-
chlorobenzoyl)-*m*-phenylenediamine-4'-sulphonic acid,
bis-(*o*-xylene-4-sulphonyl)-, bis-(2-chlorotoluene-4-sul-
phonyl)-, bis-(1 : 2 : 3-trichlorobenzene-4-sulphonyl)-, bis-
[1-chloro-4-(1' : 2'-dichlorobenzene-4-sulphonamido)benz-
ene-2-sulphonyl]-, bis-[3-(1' : 2'-dichlorobenzene-4-sul-
phonamido)benzene-3-sulphonyl]- and bis-(1 : 2-dichloro-
benzene-4-sulphonyl)benzene-*m*-sulphonamidobenz-
ene-*m*-sulphonyl-benzidine-2 : 2'-disulphonic acids, and
bis-(*m*-sulphobenzoyl)-2 : 2'-dichlorobenzidine.

C. HOLLINS.

**Dyeing etc. [with azo dyes containing a leucoan-
thraquinone or leuco-vat dye ester residue].**
D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES,
LTD. (B.P. 333,506, 8.2.29).—The dyes of B.P. 333,507
(B., 1930, 1019) are applied to the fibre and, if desired,
developed thereon by oxidation and hydrolysis as in
dyeing or printing with Soledon colours. The dyes may
also be produced by coupling on the fibre, with or without
subsequent development. Mordant metals may be
applied before or after development. The leuco-ester
may be the coupling component, *e.g.*, *p*-nitroaniline →
leuco-2-aminoanthraquinone sulphuric ester gives a
red-purple changing to brown on development.

C. HOLLINS.

Dyeing of regenerated cellulose materials.
IMPERIAL CHEM. INDUSTRIES, LTD., and (A) and (B)
R. BRIGHTMAN, (C) R. BRIGHTMAN and W. L. B. WELLA-
COTT (B.P. 333,239, 333,243, and 333,260, [A] 7.2.29,
[B] 8.2.29, [C] 5.3.29. Cf. B.P. 331,839; B., 1930, 983).—
Viscose silk is dyed evenly by the application of disazo
dyes of the types : (A) *p*-phenylenediamine → 1 mol. of
 α -naphthol, α -naphtholsulphonic acid, or an *N*-substituted
2 : 8-aminonaphtholsulphonic acid, and 1 mol. of a 1 : 8-
or 2 : 8-aminonaphtholsulphonic acid, the *p*-diamine being

applied as usual as a *p*-nitroaniline or a *p*-aminoacetanil-
ide with reduction or hydrolysis after the first coupling ;
(B) a naphthylaminesulphonic acid, or an aminonaphthol-
sulphonic acid other than J-acid, → middle component
of the benzene series → a 1 : 8-aminonaphtholsulphonic
acid : (C) a 3 : 3'-diaminodiphenylcarbamide not carry-
ing carboxylic or sulphonic groups → 1 mol. of a carb-
oxylated or sulphonated phenol or naphthol and 1 mol.
of a sulphonated naphthylamide, naphthol, dihydroxy-
naphthalene, aminonaphthol, or β -substituted amino-
naphthol, the carbamide dyes being also obtainable by
phosgenation of suitable aminoazo compounds. Both
(A) and (B) give blue or greenish-blue dyeings ; (C) gives
orange to brown and violet. Examples are : (A) 6-nitro-
cresidine → β -naphthol, reduced → 2 S-acid ; 5-nitro-*o*-
anisidine → N.W.-acid, reduced, → γ -acid or S-acid ;
4-chloro-5-nitro-*o*-anisidine → benzoyl- γ -acid, reduced,
2 S-acid ; (B) 2 : 8-aminonaphthol-3 : 6-disulphonic
acid → 2 : 5-dimethoxyaniline → S-acid ; γ -acid →
cresidine → 2 S-acid ; Brönner acid → *m*-toluidine →
S-acid ; Brönner acid → *o*-anisidine → H-acid ; (C)
3 : 3'-diaminodiphenylcarbamide → salicylic acid and
 γ -acid (yellowish-brown), or salicylic acid and M-acid
(maroon), or *o*-cresotinic acid and benzoyl- γ -acid (pink) ;
m-nitroaniline → salicylic acid, reduced, and phos-
genated with reduced *m*-nitroaniline → H-acid (bluish-
red) ; 4-nitro-*o*-anisidine → Schäffer acid, reduced, and
phosgenated (red-violet).

C. HOLLINS.

**Colouring of [cellulose ester or ether] textile
materials.** BRIT. CELANESE, LTD., G. H. ELLIS, and
W. B. MILLER (B.P. 332,624, 23.2.29).—The artificial
silks, *e.g.*, cellulose acetate silk, are dyed with oxidation
Aniline Black in the presence of considerably greater
proportions of acid than those commonly used in the
dyeing of cotton materials ; the excess of acid may be in
the form of an acid salt or mineral acid, but preferably as
an organic acid. A satisfactory solution for dyeing
acetate silk by the padding and ageing method is pre-
pared by mixing a solution containing aniline hydro-
chloride 127 g., glacial acetic acid 25 g., water 248 g.,
alcohol 100 g., and 50% gum arabic solution 200 g. with
a solution containing sodium chlorate 80 g., cupric
chloride 3 g., and water 217 g., immediately before use.

A. J. HALL.

**Production of differential [dyeing] effects on
fabrics made of or containing organic esters of
cellulose.** LYONS PIECE DYE-WORKS (B.P. 310,844,
26.4.29. U.S., 1.5.28).—Fabric is saponified according
to any desired pattern by application of an alkaline
paste, by printing, brushing, or spraying methods, then
drying, steaming, and washing ; the saponified parts are
then dyed with cotton dyes. The paste may contain the
hydroxides or carbonates of sodium and potassium,
together with a swelling agent such as pyridine, acetone,
and alkali thiocyanates.

A. J. HALL.

**Protection of wool, fur, feathers, hair etc. from
attack by moth.** A. CARPMAEL. From I. G. FARBEN-
IND. A.-G. (B.P. 333,584, 15.5.29).—Hydroxylated
diphenyls, *e.g.*, 2 : 2'-dihydroxydiphenyl, its 3 : 3' : 5 : 5'-
tetrabromo- or tetrachloro-, dichloro-, dibromo-, and
dichlorodibromo-derivatives, are used for mothproofing.

C. HOLLINS.

Production of white and coloured discharges [on ice colours from 2:3-hydroxynaphthoic arylamides]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 332,986, 22.5.29).—Goods padded with a 2:3-hydroxynaphthoic arylamide are printed with a non-volatile acid and developed with a diazo compound which does not couple in acid medium. For coloured discharges an acid-coupling diazo compound or a soluble leuco-ester of a vat dye is used for development. Examples are: anilide, printed with aluminium sulphate, developed with diazotised 4-amino-4'-methoxydiphenylamine for white on blue; *p*-chloroanilide, printed with aluminium sulphate or lactic acid, developed with diazotised 4-amino-4'-methoxydiphenylamine for white on navy-blue; anilide, printed with diazotised 4-chloro-*o*-toluidine and aluminium sulphate, developed with diazotised 4-amino-4'-methoxydiphenylamine for red on blue; 5-chloro-*o*-toluidide, printed with diazotised *m*-chloroaniline and aluminium sulphate, developed with diazotised 6-benzamidocresidine for orange on violet; anilide, printed with leucotetrabromindigo hydrogen sulphate, ammonium oxalate and vanadate, and sodium chlorate, developed with diazotised 4-amino-4'-methoxydiphenylamine for light blue on dark blue. In each case the goods are passed through an acid bath after development. C. HOLLINS.

Protection of wool, fur, etc. from attack by moth and other textile pests. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,583, 15.5.29).—Organic fluorine compounds are applied to wool, fur, etc. as mothproofing agents. Examples are 4-fluoro-3-nitrobenzoic acid, fluoroacetic acid, 1:5-fluoronaphthalene-sulphonic acid, 4:4'-difluorodiphenyl, fluoro- ψ -cumene, fluoroform; the last-named is suitable for use as vapour in treating upholstered furniture. C. HOLLINS.

Improvement of textiles and the like by rendering them resistant to attack by textile pests [moths], bacteria, and mould. I. G. FARBENIND. A.-G. (B.P. 312,163, 14.5.29. Ger., 21.5.28).—The textile is treated with a quaternary phosphonium salt, and the phosphorus compound may, if desired, be insolubilised by incorporation of other salts on the fibre. Examples are: triphenylbenzylphosphonium chloride with or without after-treatment with potassium perchlorate; sulphonated chlorocresotic anilide as precipitant for triphenylbenzylphosphonium sulphate subsequently applied; benzyltriethylphosphonium chloride; triphenyl-*p*-chlorobenzylphosphonium chloride and talcum. C. HOLLINS.

Treatment of textile filaments, yarns, threads, or the like. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,290, 11.1.29. Cf. B.P. 332,263; B., 1930, 944).—Yarns are treated in a continuous manner by means of suitable rotary, oscillatory, or reciprocatory devices at regular or irregular spaced intervals along their length (cf. B.P. 325,823; B., 1930, 553) with a mechanical reserve or a substance otherwise capable of modifying the dyeing properties of the yarn (e.g., an alkali is used for saponifying cellulose acetate and ester yarns) and are then utilised in the manufacture of textile materials which are to be dyed subsequently; alternatively, the treated yarns are dyed before use in manu-

factured goods. Multicoloured goods are thus obtained. A. J. HALL.

Manufacture of vulcanised fibres and the like. I. G. FARBENIND. A.-G. (B.P. 312,998, 31.5.29. Ger., 4.6.28).—A roller or continuous process is employed in which the sulphuric acid ordinarily used is replaced by a mixture of sulphuric acid, an inorganic salt, and an organic substance which is soluble, but stable, in the acid; the destructive action of the parchmentising acid is thus reduced. Artificial tanning agents (e.g., phenol-formaldehyde condensation products) and ureas are suitable organic substances, and the amount of inorganic salt (e.g., magnesium or ammonium sulphate) used may reach the saturation point. A. J. HALL.

Weighting of natural silk. R. CLAVEL (B.P. 332,778, 15.8.29. Ger., 16.3.29. Addn. to B.P. 266,640; B., 1927, 295. Cf. also B.P. 283,019; B., 1928, 260).—The modified process, in which an acid instead of an alkaline bath is used for fixing the silk treated with metallic salts, consists in treating the natural silk with stannic chloride solution, freeing it from excess of this solution by squeezing or centrifuging, and then treating it, without previous rinsing, in a fixing bath consisting of monosodium phosphate solution to which acids or acid salts and protective colloids may be added, squeezing, rinsing, and treating with a solution containing soap and sodium silicate. A. J. HALL.

Sizing of dyed yarns. BRIT. CELANESE, LTD. (B.P. 315,428, 13.7.29. U.S., 13.7.28).—Dyed yarns, especially of acetate silk, are sized in a substantially neutral (p_H 7–7.5) glue or gelatin bath at 60–80°, e.g., a solution of gelatin (9 pts.), glycerin (2 pts.), castor oil (1.5 pts.), and sodium benzoate (0.015 pt.) in water (260 pts.) neutralised with sodium carbonate (1.33 pts.). Bleeding and change of shade on sizing are thus avoided. C. HOLLINS.

Treatment [to improve the hot-ironing properties] of [cellulose ester and ether] materials. BRIT. CELANESE, LTD. (B.P. 313,970, 20.6.29. U.S., 20.6.28).—The fibrous materials, particularly cellulose acetate silk, are dyed with logwood black (cf. B.P. 263,222; B., 1927, 186); the fusing point of the dyed material is thereby raised (220° to 240–260° in the case of cellulose acetate silk). A. J. HALL.

Machine for washing, dyeing, or like treatment. M. DASSONVILLE (B.P. 334,730, 26.9.29. Fr., 9.3.29).

Pirn tubes, cop tubes, cheese tubes, bobbins, and like supports for textile threads, yarns, and the like. BRIT. CELANESE, LTD., W. A. DICKIE, and F. C. HALE (B.P. 334,174, 21.5.29).

Mothproofing compounds (B.P. 333,561).—See III. **Colouring of varnishes (B.P. 309,148).**—See VI. **Coloured polyvinyl ester varnishes (B.P. 334,145).**—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of the proportion of sulphur dioxide converted into trioxide when gas from roasted pyrites is passed over catalysts. J. SANS

and A. EHLINGER (Bull. Soc. chim., 1930, [iv], 47, 759—760).—The gases from the catalyst chamber are dried in passing through phosphorus pentoxide, and then pass over potassium chloride, which absorbs the sulphur trioxide. The residual sulphur dioxide is determined.

C. W. GIBBY.

Extraction of potash from polyhalite. H. H. STORCH (Ind. Eng. Chem., 1930, 22, 934—941).—The highest concentration obtained at 25° by extracting equal parts of polyhalite and water was 5.07% K_2SO_4 after 504 hrs.' contact. This concentration was reached at 100° in 120 hrs., and whilst sufficiently rapid to indicate the value of the process as a means of making a direct fertiliser, the rate of extraction is too slow for its use as a leaching process for the production of potassium sulphate. When heated, vigorous evolution of steam occurs at 298° with the formation of an amorphous mass which sinters at 551°. The optimum temperature for later extraction with water appears to be 430—465°, and an 11.4% solution of potassium sulphate may be obtained. Countercurrent leaching is undesirable as the concentration of potassium sulphate results in the formation of $K_2SO_4 \cdot 5CaSO_4 \cdot H_2O$ which later re-forms polyhalite. On evaporation of calcined polyhalite extracts practically all the calcium sulphate is deposited as syngenite ($CaSO_4 \cdot K_2SO_4 \cdot H_2O$) and the system is essentially that of K_2SO_4 — $MgSO_4$ — H_2O . As mined, polyhalite may contain up to 10% NaCl, 90% of which may be separated by rapid washing with the loss of only about 4% K_2SO_4 .

C. A. KING.

Infusorial earth from Akhaltzikh and floridin from Kutais. A. MARKMAN and M. KOVALENKO (Masloboino Zhir. Delo, 1929, No. 8, 10—13).—Kutais (Caucasus) floridin, an oil clarifier, giving on analysis SiO_2 71.68, $(Fe_2O_3 + Al_2O_3)$ 7.15, Fe_2O_3 4.37, CaO 2.65, MgO 1.52, loss (at 105°) 11.05, (on ignition) 16.10%, is 1.5—2 times more active than infusorial earth as a decoloriser and absorbent for substances containing phosphorus. Optimal conditions were determined. The oil-absorption value of floridin is one half that of infusorial earth.

CHEMICAL ABSTRACTS.

Minimum voltage to reduce aluminium oxide. A. B. NEWMAN and G. G. BROWN (Ind. Eng. Chem., 1930, 22, 995—1000).—With the object of calculating the minimum electrical energy required, under ideal conditions, to electrolyse a saturated solution of aluminium oxide (in cryolite) at 900—1000°, the sp. heat of crystallised alumina has been determined from the diffusion of heat through a cylinder of the powdered material. The minimum voltage is computed to be 0.947 at 950°.

C. A. KING.

Technical manufacture of anhydrous aluminium chloride. C. WURSTER (Z. angew. Chem., 1930, 43, 877—880).—The various technical methods are reviewed historically, with especial regard to the development of a continuous process based on the interaction of carbon monoxide, chlorine, and alumina.

H. F. GILLBE.

Vanadium [pentoxide] catalyst. J. E. ADADUROV and G. K. BORESKOV (J. Appl. Chem., Russia, 1930, 3, 11—20).—A vanadium catalyst, immune against water, arsenious oxide, hydrochloric acid, and hydrofluoric acid, and giving 99% conversion of a gas containing

7% SO_2 and passing at a velocity of 100 c.c. per min. at 400°, the length of the contact layer being 20 cm., is prepared from a mixture of sodium or potassium silicate, vanadate, and stannate solutions. These are treated with small portions of sulphuric acid, heated at 50—70°, and the precipitate is collected, pressed, dried, and heated gradually to the contact temperature.

CHEMICAL ABSTRACTS.

Utilisation of gases obtained in distillation of phosphorus in furnaces of the type of blast furnaces. E. V. BRITZKE, N. E. PESTOV, and N. N. POSTNIKOV (Min. Suir. Tzvet. Met., 1929, 4, 375—387).—The gases, containing phosphorus and phosphine, are divided into two parts after removal of dust: with one, phosphoric acid is formed continuously by passing the gas with superheated steam and a little air through lime at 600—750°; the other is oxidised with air to phosphoric acid in a Cowper stove. The products are superphosphates containing 50% P_2O_5 and combustible gases at 500—600° with a calorific value not below that of blast-furnace gas.

CHEMICAL ABSTRACTS.

Separation of constituents of coal gas. THAU.—See II. **Recovery of lead from lead sulphate waste.** FINK and GREENSPAN.—See X. **Analysis of phosphoric acid.** ISHIBASHI.—See XVI.

PATENTS.

Treatment of gases [for sulphuric acid manufacture]. W. S. ALLEN, Assr. to GEN. CHEM. CO. (U.S.P. 1,743,700, 14.1.30. Appl., 17.11.25).—In the contact process for making sulphuric acid, the burner gases are freed from arsenic by passing them through granulated ilmenite, previously activated by heating with sulphuric acid.

W. J. WRIGHT.

Continuous production of hydrofluoric acid. G. L. SCOTT, Assr. to GEN. CHEM. CO. (U.S.P. 1,748,735, 25.2.30. Appl., 30.6.26).—Fluorspar and sulphuric acid are separately and continuously introduced into a cylindrical rotary furnace, in which they are subjected to thorough agitation to prevent caking, immediate contact with the heated surface of the furnace being prevented by allowing the materials to form a bed. Agitation is conveniently effected by means of iron rails of a length approximately equal to that of the cylinder, these rails tending to ride up the sides as the cylinder rotates, and then falling back with considerable force.

W. J. WRIGHT.

Ammonia-oxidising method. I. HECHENBLEIKNER and N. TITLESTAD, Assrs. to CHEM. CONSTRUCTION CO. (U.S.P. 1,748,646, 25.2.30. Appl., 18.5.27).—Difficulty in regulating the steam supply to stripping towers in which ammonia is recovered from ammonia liquor results in very rich gaseous mixtures for the oxidation process, with high combustion temperatures and consequent burning of the gauzes. External sources of heat may be entirely eliminated by utilising the heat of the exothermal reaction in the converter to separate the gaseous ammonia from the liquor, the process being made automatic by transferring the reaction heat to water, and using this to heat the stripping tower.

W. J. WRIGHT.

Method of concentrating caustic alkalis. R. M. WINTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P.

332,250, 16.1.29).—The evaporating plant is constructed of, or lined with, copper-nickel alloy (20–50% Ni) if heated with steam, or of nickel-chromium alloy (20–50% Cr) if fired direct, the parts exposed to the alkali being resistant to corrosive action. The alloys should be substantially free from iron. [Stat. ref.]

H. ROYAL-DAWSON.

Production of saturated lime-water. NAAML. VENN. NECKAR WATERREINIGER MAATS., and J. A. HERINGA (B.P. 334,011, 5.7.29).—Water flows uninterruptedly at high velocity from below upwards through a quantity of lime sludge arranged upon a resistant layer of gravel spread over the whole cross-section of the saturator in such a manner that the sludge is wholly penetrated by the water.

H. ROYAL-DAWSON.

Extracting magnesia from dolomite. RHEINISCHE-WESTFÄLISCHE KALKWERKE (B.P. 319,690, 16.9.29. Ger., 26.9.28).—Half-burnt and ground dolomite is treated, before being wind-sifted, with sufficient water, and preferably also with carbon dioxide, to convert the magnesium oxide into the hydroxide or basic carbonate.

H. ROYAL-DAWSON.

Manufacture of sodium nitrate. G. H. GLEASON, ASSR. to ANGLO-CHILEAN CONSOLIDATED NITRATE CORP. (B.P. 315,262, 14.6.29. U.S., 10.7.28).—To obtain a product suitable for agricultural purposes, in the form of spherical particles of 10–40-mesh and of 99% concentration, the commercial nitrate is heated at a temperature slightly above its m.p., e.g., at 350°. At this temperature most of the impurities do not melt, and remain undissolved. After filtering, the fused material is conducted to a spray distributor, having nozzles of specified shape and dimensions, and projected into a cooling atmosphere, the spray particles being instantaneously chilled by suitable means, such as an air blast.

W. J. WRIGHT.

Production of potassium nitrate. P. HÖFER, and KALIFORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 332,359, 13.6.29).—A hot solution of the nitrate of a readily hydrolysable metal, e.g., aluminium or iron, is allowed to react with one of potassium chloride and then cooled to separate potassium nitrate. The mother-liquor is concentrated with the evolution of hydrogen chloride, either until nitrous fumes begin to escape, or until most of the chlorine has been expelled from the solution. Nitric acid, potassium chloride, and water are then added to restore the original concentration. The solution is cooled, potassium nitrate separated, and the operation repeated.

H. ROYAL-DAWSON.

Treatment of superphosphate. L. ADELANTADO (B.P. 333,518, 9.5.29).—Superphosphate is made neutral to methyl-orange by means of ammonia, and then lixiviated with water or water acidulated with sulphuric acid, the solution being evaporated to a suitable density for crystallisation. By treating the hot leach liquors with sulphuric acid, all the calcium is precipitated and a solution of orthophosphoric acid is obtained, which may be converted into monoalkali phosphates. Alternatively, these may be produced by treating the leach liquors with alkali sulphates instead of sulphuric acid.

W. J. WRIGHT.

Manufacture of sodium salicylate. M. E. PUTNAM, ASSR. to DOW CHEM. CO. (U.S.P. 1,755,362, 22.4.30. Appl., 6.4.23).—Rhombic crystals of sodium salicylate hexahydrate are obtained by neutralising a hot solution of salicylic acid with sodium hydrogen carbonate to obtain a solution which does not deposit the anhydrous salt when cooled to 5–6°. After cooling to this temperature, the solution is seeded with hexahydrate crystals and set aside until 70% of its content of salicylate has crystallised. The mother-liquor is decanted and returned to the neutralising vat. A. R. POWELL.

Production of the cyanamides of the alkaline-earth metals and magnesium. N. CARO and A. R. FRANK, ASSEES. of STICKSTOFF-WERKE GES. M.B.H. (B.P. 332,468, 1.10.29. Ger., 1.10.28. Addn. to B.P. 279,812 and 281,611; B., 1929, 53, 206).—The reactions described in the prior patents are accelerated by the use as catalysts of silver, or copper, or salts of these metals, which, whilst they do not decompose the ammonia formed, act as catalysts for the water-gas reaction.

H. ROYAL-DAWSON.

Manufacture of sodium aluminate. J. V. N. DORR, G. M. DARBY, A. TERRY, JUN., and H. N. SPICER, ASSRS. to DORR CO. (U.S.P. 1,747,759, 18.2.30. Appl., 11.2.25).—The proportions of bauxite to caustic soda should be regulated so that the molecular ratio of Na_2O to Al_2O_3 is about 2 : 1. Digestion is carried out in continuous agitators at atmospheric pressure and at a temperature above 87.7°, and during digestion the product is continuously diluted so as to maintain a sp. gr. of 1.20–1.32, the red mud being separated by sedimentation and decantation.

W. J. WRIGHT.

Manufacture of a base-exchange material. Base-exchange substance. Preparation of metallo-silicates. A. S. BEHRMAN, ASSR. to PERMUTIT CO. (U.S.P. 1,756,623–5, 29.4.30. Appl., [A] 24.4.24, [B] 5.12.24, [C] 11.4.25).—(A) A solution of sodium silicate is treated with an excess of dilute sulphuric acid to obtain a colloidal solution of silica which, after cooling to 5°, is treated with a solution of sodium aluminate until the mixture reacts slightly alkaline. The stiff gel which rapidly forms is dried at 80° and wetted again, whereby it breaks up into vitreous granules having an exchange capacity of 250 grains of calcium carbonate per lb. (B) Bauxite, gibbsite, limonite, or other natural or artificial granular aluminium or iron hydroxide is screened through 20-mesh and the dust removed by sieving on 60-mesh. This product is then heated under pressure with a solution of sodium silicate to obtain a zeolite-like substance. (C) In carrying out the reactions described in (A), the two solutions are allowed to flow simultaneously into a small mixing vessel from pipes so proportioned as to give the correct mixture, and the mixture is allowed to fall continuously into a large receiving vessel through a long pipe which ensures thorough mixing in the receiver.

A. R. POWELL.

Improvement of glauconite [for water softening]. H. KRIEGSHEIM and W. VAUGHAN, ASSRS. to PERMUTIT CO. (U.S.P. 1,757,373, 6.5.30. Appl., 4.5.25. Renewed 23.10.29).—The crude sand is heated for 30 min. at 50–60° with a 0.4–1% solution of sodium hydroxide to remove humus and disintegrate clayey material. The

sand is then stirred with a cold 1% solution of aluminium sulphate, drained, and agitated with a 2–3% solution of sodium silicate. The treatment removes all substances which impart colour or odour to water and at the same time hardens the sand.

A. R. POWELL.

Production of calcined bauxite. F. C. FRANZ, ASSR. to ALUMINUM CO. OF AMERICA (U.S.P. 1,756,425, 29.4.30. Appl. 19.6.25).—Bauxite is mixed with powdered coal and a binder, *e.g.*, iron ore or clay, and the mixture is burned on a grate by drawing air downwards through the mass by means of a suction fan. The product consists of porous sintered lumps suitable for the electrothermic production of pure alumina.

A. R. POWELL.

Recovery of copper [hydroxide] from dilute ammoniacal solutions. I. G. FARBENIND. A.-G. (B.P. 313,045, 18.5.29. Ger., 5.6.28).—The dilute cuprammonium solution is treated with sulphuric or hydrochloric acid in amount necessary to neutralise the ammonia contained in the complex compounds, the precipitated hydroxide being collected.

H. ROYAL-DAWSON.

Recovery of copper from spent copper lyes. I. G. FARBENIND. A.-G. (B.P. 310,425, 25.4.29. Ger., 25.4.28).—The lyes, *e.g.*, "precipitating water" obtained in the manufacture of cuprammonium silk, are treated with an acid-resistant adsorbent material such as silicic acid, active carbon, etc. The adsorbed metal is dissolved out by an acid (*e.g.*, sulphuric acid). The adsorbent may be used again after being washed free from excess acid.

H. ROYAL-DAWSON.

Production of iron carbonyl. VEREIN. STAHLWERKE A.-G. (B.P. 319,356, 9.9.29. Ger., 22.9.28).—Finely-divided copper is admixed with the iron to be acted on by carbon monoxide, to accelerate the reaction; similarly, copper oxide or similar compound may be added to iron oxide before reduction is effected.

H. ROYAL-DAWSON.

Production of aluminium sulphate. M. BUCHNER (B.P. 333,835, 20.11.29. Ger., 24.11.28).—Aluminium nitrate is heated with sulphuric acid under reduced pressure with admission of air or steam, the nitric acid being expelled at 300° and recovered. Apparatus of chromium-iron or chromium-iron-nickel alloy is used. To obtain aluminium nitrate free from iron, clay or other aluminiferous material is mixed with a deficiency of nitric acid and set aside for some days at a low temperature; it is then heated to 80–100° at 6–8 atm. and finally to 150–160°. The iron may also be completely removed by treating the aluminium nitrate solution with concentrated nitric acid or nitric acid vapour, whereby aluminium nitrate is precipitated, or by heating the solution to 130° to give it basicity, the basic aluminium nitrate remaining in solution at this temperature.

W. J. WRIGHT.

Manufacture of [finely-divided] bismuth hydroxide. I. G. FARBENIND. A.-G. (B.P. 332,504, 30.11.29. Ger., 3.12.28).—An aqueous solution of a bismuth salt is precipitated by means of an alkali such as ammonia, adherent water is removed from the precipitate by first washing with a solvent which is more volatile but soluble in water (methyl or ethyl alcohol, acetone),

then washing with a second volatile solvent (ether, ethyl chloride, chloroform), and finally evaporating the second solvent. The product is suitable for therapeutical purposes.

H. ROYAL-DAWSON.

Preparation of stannic oxide gels. J. J. ETRIDGE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 333,670, 5.6.29).—An aqueous solution of ammonia is added to an aqueous solution of a stannic salt, the conditions being so controlled that about 95% of the base is added before precipitation begins. The solution is then kept, and stannic hydroxide is precipitated in the form of a hydrogel, which is washed, collected on a filter, and slowly dried. If desired, other catalytic oxides may be combined with the gel by treating it with aqueous solutions of the corresponding salts.

W. J. WRIGHT.

Zinc oxide manufacture. E. C. GASKILL, ASSR. to ST. JOSEPH LEAD CO. (U.S.P. 1,743,964, 14.1.30. Appl. 17.11.26).—Zinc ore after roasting, sintering, and crushing is mixed with carbonaceous material and introduced into the preheater of a sealed electric-resistance furnace, whence it passes through a second preheater to the electrodes. The zinc vapour leaves the furnace through a lateral pipe leading into a vertical one, and at the point where the former enters the latter just sufficient air is admitted to oxidise the vapour, and this is ignited. The zinc oxide is drawn by means of a fan into collecting bags. An advantage of this process is that the zinc vapour leaving the furnace is free from sulphur oxides, since the absence of oxygen ensures the retention in the furnace of all the sulphur. The ignition of the vapour in a closed pipe instead of in the atmosphere gives a product of uniform average fineness between 0.4 and 0.25 μ .

W. J. WRIGHT.

Purification of zinc chloride. J. L. McCLELLAN, ASSR. to NAT. VULCANIZED FIBRE CO. (U.S.P. 1,744,981, 28.1.30. Appl. 13.4.27).—The solution of zinc chloride, preferably of *d* 1.52, is treated at normal temperature with zinc nitrite in sufficient amount to react with all the ammonium chloride present, the mixture being agitated. When the reaction is complete, the temperature of the solution is raised to 82.2°, so as to decompose the ammonium nitrite, and then to the b.p. in order to precipitate iron salts as oxide, which is removed.

W. J. WRIGHT.

Manufacture of white zinc chloride. J. O. BETTERTON, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,747,751, 18.2.30. Appl. 1.9.27).—Small amounts of zinc are removed from lead bullion, after desilvering by the Parkes process, by circulating the molten bullion through a chamber containing chlorine, the temperature being maintained at about 340–400°. The zinc chloride formed is skimmed off, and freed from lead by melting it in presence of excess of zinc at about 370°, and, while fused, is treated with nitre to yield a white product.

W. J. WRIGHT.

Treatment of mixtures of oxy-salts of arsenic, antimony, and tin. M. F. PERKINS, C. W. HANSON, and B. M. O'HARRA, ASSRS. to AMER. SMELTING & REFINING CO. (U.S.P. 1,756,007, 22.4.30. Appl. 20.1.27).—The alkali slag produced in the Harris process of lead refining is dissolved in warm water, the insoluble sodium antimonate collected and smelted with carbon

to produce antimony, the lead and copper are removed by agitation with tin, stannous salts, or sodium sulphide, and the purified liquor is electrolysed for the recovery of pure tin. The spent electrolyte is treated with milk of lime to remove arsenic as calcium arsenate, and the filtrate is evaporated for the recovery of sodium hydroxide and chloride for use again in lead refining.

A. R. POWELL.

[Electrolytic] manufacture of [per-]compounds containing oxygen. I. G. FARBENIND. A.-G. (B.P. 313,124, 23.5.29. Ger., 7.6.28. Addn. to B.P. 316,648; B., 1929, 814).—During electrolysis the electrolyte described in the prior patent is maintained saturated with a sparingly soluble mercury compound (such as a sulphate or phosphate).

H. ROYAL-DAWSON.

Cleansing compositions. HENKEL & Co. G.M.B.H. (B.P. 332,530, 22.3.29. Ger., 5.2.29).—The polish, suitable for aluminium utensils etc., consists of a mixture of trisodium or other phosphate and water-glass, with or without a further admixture of known cleansing agents. [Stat. ref.]

H. ROYAL-DAWSON.

Production of phosphates and hydrogen. BAYERISCHE STICKSTOFF-WERKE A.-G. (B.P. 308,684, 18.3.29. Ger., 26.3.28).—Phosphorus, or hydrogen phosphide, and water are caused to react under pressure at temperatures up to 600°, and in the presence of ammonia, alkalis, alkaline-earth metals, bases, or salts, to form phosphates and hydrogen. The solution of phosphates may be sufficiently concentrated that, on squirting from the reaction chamber, it forms a powdery product or it may be crystallised. Catalysts (metals, alloys, or various phosphorus compounds) may be used.

H. ROYAL-DAWSON.

Separation of oxy-salts from alkali mixtures containing same. H. HARRIS (U.S.P. 1,775,676, 16.9.30. Appl., 3.8.25. U.K., 8.8.24).—See B.P. 245,479; B., 1926, 236.

Production of soluble lead reagents. D. W. PARKES, Assr. to H. W. ROBINSON (Re-issue 17,804, 16.9.30, of U.S.P. 1,740,312, 17.12.29).—See B., 1930, 144.

Heat-insulating material (U.S.P. 1,757,470).—See I. **Products from coal** (B.P. 314,859). **Removing sulphur compounds from gases** (B.P. 305,026). **Ammonia from gas** (U.S.P. 1,747,616).—See II. **Welding agent** (B.P. 313,487).—See X. **Urea-calcium nitrate and fertilisers** (B.P. 332,945 and 332,948).—See XVI.

VIII.—GLASS; CERAMICS.

Physiological and chemical experiments with "U.-V." glasses and a method of determining their quality. H. VALENTIN (Pharm. Ztg., 1930, 75, 982—984, 995—998, 1005—1008).—A marked tanning of the skin and a slight improvement in strength were observed in children who had occupied for several months class-rooms having "Ultravit" glass windows. No increase in the hæmoglobin in the blood was noted. The germination period of seeds was considerably shortened under "Ultravit" glass, although the rapidity of growth of the plants was increased only by about

10%. Fungus cultures declined rapidly under "Ultravit" glass. Changes in chemical compounds are largely due to the action of the ultra-violet rays, but in some cases decomposition is caused by the visible rays. A simple method of testing "U.-V." glasses is described, which is based on the action of light in converting potassium nitrate solution into the nitrite. Of the glasses examined, the "Uviolglas" (Schott und Gen.) was the best and cheapest, whilst the glass used in the above biological tests, "Ultravit," was least transparent to ultra-violet rays.

F. SALT.

Determination of the degree of refractoriness of clays by their content of water of constitution. N. P. CHIZHEVSKI (J. Russ. Met. Soc., 1926, 177—183).—Hygroscopic moisture, removed at 120°, had no effect on the refractory properties of Tomsk, Salair, and Borovitchy clays. The kaolin content (Sabell) and (approximately) the m.p. of clays are directly proportional to the percentage of water of constitution, the removal of which is nearly complete at 900°.

CHEMICAL ABSTRACTS.

Russian porcelain manufacture. I. F. PONOMAREV (Sep., 3 pp.).—Determinations of the limits of solubility at 1300° in fused felspars of the ingredients of the porcelain mass were made. The limit of saturation of the fused mass with kaolin was 14%, with quartz 70%, and with alumina 3.5%. With silica and alumina the limit of saturation occurred when the fused mass had the composition $K_2O, 1.15Al_2O_3, 12SiO_2$. With silica and kaolin no further saturation was observed.

CHEMICAL ABSTRACTS.

PATENTS.

[Roller-type] glass-annealing lehrs [for plate or sheet glass]. R. HADDAN. From SIMPLEX ENG. Co. (B.P. 331,927, 13.4.29).—A lehr primarily intended to work in conjunction with glass-rolling apparatus is described. The claims cover the provision of a system of rollers for charging and conveying the sheets to and through the furnace, of doors for shutting off parts of the furnace when not required or for cleaning purposes, and of means for handling the annealed sheets at the cold end of the furnace, full details of which are given.

M. PARKIN.

Manufacture of compound glass. SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 316,955, 6.8.29. Fr., 3.8.28).—A sheet of plastic material, e.g., cellulose acetate or nitrocellulose, is superficially softened by immersion in a bath of substantially non-volatile plasticiser, e.g., benzyl alcohol, and while therein is placed between two glass sheets free from air bubbles and the composite sheet is united by gentle pressure. Vacuum may be used to assist removal of air bubbles. Alternatively, one or both sheets may be coated with a solution of the plastic material and the two united in the bath.

M. PARKIN.

Preventing or minimising the obscuration of windows and other transparent surfaces by moisture. H. E. GRIFFITHS (B.P. 332,966, 10.10.29).—A paste is claimed consisting of soap (preferably carbolie) mixed with glycerin, water, and a drying agent.

E. B. HUGHES.

Tunnel or channel dryer for earthenware and the like. H. HAAS (B.P. 314,520, 30.5.29; Ger., 30.6.28. Cf. B.P. 173,234; B., 1922, 969 A).—In a multi-chamber dryer the fan, which circulates the drying air, is situated between the drying chambers at one end and the humidifying or steaming chambers at the other, thus serving to raise the temperature of the ware before drying commences. The tunnel is closed at the inlet and opened only for the intermittent introduction of trucks of green ware.

M. PARKIN.

Manufacture of porous ceramic articles or products. R. LAHAUSOIS (B.P. 311,761, 22.4.29. Belg., 16.5.28).—Porous ceramic articles suitable for insulating purposes, abrasives, filters, etc. are prepared by intimately mixing dry ground porcelain mass with carbon grains of size suited to the degree of porosity required, moistening with water (or alkali, to form with the paste an enamel which coats the cells and promotes cohesion) sufficiently for "dry-moulding." The moulded shapes, set in airtight containers and packed round with a reducing agent (carbon), are then fired first at 1200—1300° in a reducing atmosphere, followed, after being removed and stacked so as to ensure good circulation of air, by a second firing under oxidising conditions at a red heat to burn out the carbon.

M. PARKIN.

Refractory composition and manufacture of articles therefrom. L. J. TROSTEL, ASSR. to GEN. REFRACTORIES Co. (U.S.P. 1,752,867, 1.4.30. Appl., 30.3.27).—Either natural mullite or that produced from dumortierite or by calcining cyanite, andalusite, or sillimanite at 1400° forms 2—20% (preferably 10%) of a mixture with 60—70% of crude and 20—30% of calcined diaspore; such mixture, when formed into refractory articles, has a lower firing shrinkage than have mixtures usually employed.

M. PARKIN.

Manufacture of grinding wheels, hones, and the like. L. BRUNELLI, G. CARRA, and F. SOFIA (B.P. 332,524, 21.2.29).—Carbonaceous slag or waste from burnt coal is mixed with emery etc. and borax or powdered silica and baked at 800—1300°.

H. ROYAL-DAWSON.

Glass tank furnaces. HARTFORD-EMPIRE Co., Assees. of P. G. WILLETTS (B.P. 334,799, 27.11.29. U.S., 15.12.28).

Manufacture of laminated glass. L. J. KOLB, Assee. of A. G. WORRALL (B.P. 334,768, 25.10.29. U.S., 30.10.28).

Composition [of plaster and oil] for modelling, sculpture, pottery, etc. J. T. TUSSAUD (B.P. 334,000, 27.6.29).

IX.—BUILDING MATERIALS.

Calcium ferrites and iron cements. V. S. NAGAI and K. ASAOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 312—315 B; cf. B., 1930, 907).—Iron cement contains a large amount of calcium ferrite, but sinters at 1450—1500°, i.e., rather above the corresponding temperature for Portland cement. This is due to the small amount of calcium aluminate and to the lack of eutectic formation.

C. W. GIBBY.

PATENTS.

Manufacture of cementitious material. H. BERRY (B.P. 332,925, 30.4.29).—In the production of cementitious material from a base material (e.g., aluminium silicates) and a binder comprising oxides or hydroxides of calcium or magnesium and sulphuric or sulphurous acid, or salts thereof, the original heating to calcine the raw material (of the binder) and/or a second heating is effected in an atmosphere containing sulphurous or sulphuric acid and, if desired, steam. Thus natural carbonates of calcium or magnesium may be calcined and then dipped into a solution (e.g., 10—15%) of aluminium sulphate acidulated with sulphuric acid, and the product heated at 700° (magnesium compound) or 800° (calcium compound) in an atmosphere of steam and acid vapour, cooled, and incorporated with a base material. Alternatively, blast-furnace slag containing silicates and lime may be heated in an atmosphere of steam and acid vapour at 700—900°.

S. K. TWEEDY.

Manufacture of waterproof concrete. R. CROSS, ASSR. to SILICA PRODUCTS Co. (U.S.P. 1,744,869, 28.1.30. Appl., 20.4.25).—A bituminous substance, such as asphalt, is incorporated with a concrete mix of hydraulic cement, mineral aggregate, and a gelatinising clay, the mixture, after setting, being heated at a temperature sufficient to melt the asphalt.

W. J. WRIGHT.

Preparation for waterproofing cement used in buildings. A. STEWART (B.P. 334,053, 20.8.29).—A mixture of soft soap, ammonia, alum, washing soda, lard, boiled linseed oil, and water is boiled, cooled, and mixed with cement and sand.

L. A. COLES.

Hydraulic binding medium [impervious to sea- and hard water]. SOC. ANON. DES CHAUX ET CEMENTS DE LAFARGE ET DU TEIL (B.P. 317,783, 29.7.29. Fr., 22.8.28).—The product is prepared from 80—50 pts. of aluminous cement produced by fusion, clinkering, or fritting and 20—50 pts. of gypsum (raw or calcined at or above 200°) or of anhydrite; alternatively, a crushed mixture of calcium sulphate with the constituents of aluminous cement is calcined.

L. A. COLES.

Manufacture of porous [heat- and sound-]insulating bodies. H. BOHLANDER (B.P. 333,957, 28.5.29).—A mixture of two inorganic fibrous materials, one chosen for its insulating properties (e.g., slag-wool) and the other for its strength (e.g., asbestos), preferably with the fibres set parallel and with or without the addition of kieselguhr, magnesia, etc. and/or binding agents, is worked into a coherent, felt-like mass, e.g., by dipping in an aqueous solution of a binder or by spraying with steam or moist air.

L. A. COLES.

Preparation of asphalt emulsions. R. LICHTENSTERN (B.P. 332,591, 22.4.29).—From 1 to 3% of Trinidad or Bermudez asphalt or montan wax is mixed with bitumen, particularly petroleum asphalt, and a hot solution of caustic alkali is added. The emulsions become miscible with water in all proportions when the proportion of added asphalt or wax is increased up to 25—50%; the stability is further increased by the addition of resins and fatty acids etc.

H. ROYAL-DAWSON.

Manufacture of pavement materials. N.V. KONINKLIJKE STEARINE KAARSENFABR. GOUDA (B.P. 333,937, 23.5.29. Holl., 5.2.29).—Bituminous emulsions are mixed cold with stony material (e.g., basalt lava, blast-furnace slag, limestone) of porosity such that the emulsion adheres strongly to it; the stony material may, if desired, be pretreated with oils, bituminous materials, solvents, or mixtures of these. L. A. COLES.

[Rubber-bitumen] paving blocks. DUNLOP RUBBER CO., LTD., H. C. YOUNG, F. W. WARREN, and F. H. TOOP (B.P. 333,047, 29.7.29).—A paving block has a comparatively resilient rubber upper surface, produced as in B.P. 315,512 (B., 1929, 830); the lower portion of this surface contains a higher proportion of sulphur so as to form a vulcanite layer intermediate between the upper surface and the base. The base is composed of bitumen aggregate which is attached to the vulcanised rubber top by pressing, firmness of attachment being secured by means of undercut grooves and the application of a bond of bitumen or bitumen-rubber adhesive. D. F. TWISS.

Treatment of road-making materials. C. G. FOX (B.P. 332,897, 20.3.29).—The stone aggregate etc. is treated with solutions (e.g., 0.1–3%) containing pyridine or pyridine bases prior to impregnation with bitumen or tar emulsions. Alternatively, the bitumen or tar emulsions may be treated, preferably immediately before use, with solutions containing the bases, and then applied to the road-making material. S. K. TWEEDY.

Drying of lumber. J. P. WILSON, ASSR. to WILSON-OTWELL & CONE, INC. (U.S.P. 1,757,892, 6.5.30. Appl., 6.10.27).—Immediately after sawing, the lumber is immersed in hot water which is kept at the b.p. until the wood is heated uniformly throughout. The wood is then removed and dried artificially in the usual way. A. R. POWELL.

Treatment of wood and other fibrous material. G. E. S. SANNA (B.P. 311,227, 22.4.29. Nor., 7.5.29).—The material is coated or impregnated with a mixture of cyclic carbon compounds with other substances such as formaldehyde and acetone or alcohol, and is then heated at 120–170° and under 10–40 kg./cm.² H. ROYAL-DAWSON.

Wood-impregnating product. Impregnation of wood. SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 310,804–5, 29.4.29. Fr., 30.4.28).—(A) The product comprises coal tar, creosote, or lignite oil, shale petroleum, etc., or a mixture thereof, having dissolved or combined therein, while hot, 1–5% of an antiseptic salt of a metalloid which improves the silting power of the base (e.g., a compound of antimony or arsenic, such as arsenic sulphide, halogen-containing compounds being excluded). Arsenic or its oxide, or antimony, or free sulphur may also be incorporated in addition. Arsenic sulphide may be produced *in situ* by interaction of sulphur and arsenious oxide. (B) The wood, if desired after preliminary heat treatment, is impregnated first with an antiseptic aqueous solution (A) having great penetrability which contains a solution of arsenic sulphide in ammonium hydrosulphide, and then with an antiseptic liquid the principal antiseptic constituent of which is insoluble in such aqueous solu-

tion. Solution A may also contain a soluble fatty acid salt (e.g., acetate, propionate), and/or an alkaline salt (alkali sulphide), and/or hydrophenolic compounds (wood tars), or a substance (ammonia, soap, or alkali resinate) capable of lowering its surface tension. The arsenic sulphide may be produced *in situ* by interaction of arsenic hydride and alkali sulphide. S. K. TWEEDY.

Recovery of surplus preservatives in treatment of timber. R. N. RAWSON (U.S.P. 1,756,797, 29.4.30. Appl., 3.6.26).—The timber is immersed in the preservative, e.g., creosote, in a retort which is then evacuated and heated to above the b.p. of water at the internal pressure. The steam thus produced in the pores of the wood drives out the surplus preservative, which is removed from the retort prior to breaking the vacuum. A. R. POWELL.

Plates, slabs, tiles, etc. for building and sound- or heat-insulating purposes. NAAML. VENN. DE NIEUWE ISOLEER MAATS. "DE NIM" (B.P. 314,354, 25.6.29. Holl., 25.6.28).

Sheets or slabs for building purposes. A. H. J. WRIGHT (B.P. 333,978, 6.6.29). L. A. COLES.

Road surface and its preparation. J. ROBINIUS (B.P. 334,597, 7.6.29).

Preparation of road-surfacing compositions. S. E. FINLEY (B.P. 334,701, 28.8.29).

Heat-insulating material (U.S.P. 1,757,470). **Wet-mixing apparatus** (U.S.P. 1,758,200).—See I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Constitution of the Fe-C-Si system. A. KRÍŽ and F. POBOŘIL (Iron and Steel Inst., Sept., 1930. Advance copy. 20 pp.).—In the iron-carbon-silicon ternary system the eutectoid concentrations up to 6% Si, the maximum solubility curve of carbide in γ -solid solution, and the transformation points on heating have been determined. From these data the constitutional diagrams of the ternary iron-carbon-silicon alloys for 1, 2, 4, and 6% Si have been prepared. C. J. SMITHELLS.

Mechanism of the dissolution of cementite in carbon steel and the influence of heterogeneity. E. WALLDOW (Iron and Steel Inst., Sept., 1930. Advance copy. 38 pp.).—The transformation of α - into γ -iron with simultaneous dissolution of Fe_3C is found to occur successively in different parts of a specimen during heating. It is suggested that this is due to uneven distribution of silicon which influences the transformation. Two new hardening structures are described, hypotroostite, which occurs in hypoeutectoid steels, and sorbotroostite, which occurs in eutectoid steels. C. J. SMITHELLS.

Physics of the hardening of steel. F. WEVER (Naturwiss., 1930, 18, 452–459).—The influence of the rate of cooling on the thermal transitions of carbon steels has been determined for velocities up to 10,000° per sec., and a space model showing the relationships between temperature, carbon concentration, and cooling velocity has been prepared, which, in conjunction with observations of the fine structure of the metal, supplies a simple

theory of the hardening process according to which martensite is a thermodynamically unstable phase.

H. F. GILLBE.

Quantitative analysis of steels by spectrum analysis. F. TWYMAN and A. A. FITCH (Iron and Steel Inst., Sept., 1930. Advance copy. 12 pp.).—The method of wedge spectra, using the logarithmic sector, has been found satisfactory for the following determinations, all obtainable from one spectrogram: Si up to 0.8%, Cr up to 4%, Ni up to 5%, Cu up to 1%. The accuracy is 10% of the contained element in the case of silicon, and 5% in the other cases. One spectrogram can be prepared in $\frac{1}{2}$ hr., and measurement of each constituent takes 5 min.

C. J. SMITHELLS.

Analysis of basic slags and representation of their composition in a triangular diagram. O. QUDRAT (Iron and Steel Inst., Sept., 1930. Advance copy. 9 pp.).—The usual methods for the determination of ferric oxide and of sulphide sulphur in basic slags are criticised and the following methods recommended. The total sulphur and the sulphate sulphur are determined gravimetrically; the difference indicates the content of sulphide sulphur, *a*. Then the sulphide sulphur, *b*, is determined iodometrically in a separate sample, and the bivalent iron contained in the solution of decomposed slag is determined oxidimetrically. The difference *a* - *b* indicates the sulphide sulphur, *c*, lost in the iodometric determination. This is equivalent to the tervalent iron which is reduced when the slag is decomposed. By subtracting this figure from that for the bivalent iron, the accurate figure for bivalent iron is obtained. For rapid iodometric determination of sulphide sulphur the tervalent iron is first reduced with zinc. C. J. SMITHELLS.

Reactions between iron sulphide, sulphur dioxide, and iron oxides in the metallurgy of copper. A. C. HALFERDAHL (Ind. Eng. Chem., 1930, 22, 956—963).—Copper calcines usually contain magnetite which is not reduced by ferrous sulphide below 1300°, the maximum temperature of smelting. In actual smelting practice it is proposed to maintain a lower-grade bath of matte in the hottest part of the furnace independently of the customary matte pool. The converter slag would be poured from a height in this hot part so that the slag would plunge into the matte bath and then flow to the other end of the furnace as in present practice. Siliceous flux would be charged to flux the reduced magnetite. In the smelting operation it is considered that the reduction of sulphur dioxide by carbon (coke) is not necessarily a desirable reaction, and the chief point is to maintain a hot focus at the hearth to accelerate reduction of magnetite by the low-grade matte.

C. A. KING.

Relation between macro- and micro-structure in some non-ferrous alloys. M. L. V. GAYLER (Inst. Metals, Sept., 1930. Advance copy. 12 pp.).—Examination of the micro- and macro-structures of a 5% lead-tin alloy, a 7% copper-aluminium alloy, and an 11% silicon-aluminium alloy cast from various temperatures into different moulds shows that with increase of casting temperature the macrostructure becomes coarser and the microstructure finer. Variation in the ratio of the cross-section of an ingot to that of the mould affects both

structures, but if this ratio is kept constant and the alloy is cast just above the liquidus, casting into steel or graphite moulds has little effect on either structure; with higher casting temperatures the alloy cast in steel has a different macrostructure from, but similar microstructure to, the same alloy cast in graphite. At low casting temperatures the nature of the melting atmosphere does not affect the structure, but at higher temperatures hydrogen refines the macrostructure. Copper-aluminium alloys which have been freed from gas by the nitrogen process, and subsequently melted in a vacuum, still exhibit inverse segregation. The nature of the furnace atmosphere has little effect on the modification of silicon-aluminium alloys, and a normal alloy cannot be modified by the extremely rapid cooling produced by casting it into a heavy, water-cooled, copper mould.

A. R. POWELL.

Influence of reheating on dilatation and hardness of tempered aluminium-silicon alloys. P. CHEVENARD and A. PORTEVIN (Compt. rend., 1930, 191, 252—254. Cf. Guillet and Ballay, B., 1930, 717).—The alloys, tempered at 525°, were reheated to various temperatures up to 400°, the heating being effected at the rate of 250° per hr., and then maintained at the desired temperature for 8 hrs. The diminution of dilatation at 20°, the increment in length, and the increase in Brinell hardness, for an alloy containing 0.94% Si are plotted against temperature of reheating. The first two curves have a maximum at 275°, and show that between 200° and 350° a reheating for 8 hrs. suffices to precipitate the whole of the silicon, but that above that temperature re-dissolution occurs. The hardness curve shows a maximum at the lower temperature of 175°, due to the fact that though hardness is increased by precipitation of the silicon, it is decreased by the diminished fineness of structure consequent on reheating. It is concluded that silicon as an independent constituent does not explain the contraction resulting from reheating aluminium-copper alloys (cf. B., 1928, 301).

C. A. SILBERRAD.

Unusual corrosion of aluminium by alkali. O. W. STOREY (Trans. Amer. Electrochem. Soc., 1930, 58, 43—48).—A peculiar case of pitting of aluminium electric-oven walls was found to be caused by sodium hydroxide formed from the sodium silicate adhesive used in sticking together the corrugated sheets of asbestos which form the insulation. Asbestos millboard coated with sodium silicate so as to make it adhere directly to aluminium does not cause appreciable corrosion of the metal, but when the sodium silicate is on the other side of the millboard and excessive moisture is present the sodium hydroxide formed by hydrolysis diffuses through the asbestos layer and attacks the aluminium. Methods of avoiding this type of corrosion are indicated.

H. J. T. ELLINGHAM.

Effect of addition of salts and bases on corrosion by hypochlorite. G. N. QUAM (Food Ind., 1930, 2, 121—122).—The corrosive action of hypochlorite solutions on metals is reduced by the addition of sodium phosphate (0.1—0.5%), sodium hydroxide and carbonate (0.5%), or calcium hydroxide (0.1%).

CHEMICAL ABSTRACTS.

Action of boiling barium chloride solutions on metals. V. ZEMLYANITZUIN and P. DOBROVOLSKI (J. Chem. Ind., Russia, 1929, 6, 1047).—The solubility of copper and of lead increases with the duration of immersion and the concentration of the solution. The solubility of iron or aluminium is maximal after 8–16 hrs., a protective coating of hydroxide being formed.

CHEMICAL ABSTRACTS.

Electrolytic recovery of lead from lead sulphate waste. C. G. FINK and L. GREENSPAN (Trans. Amer. Electrochem. Soc., 1930, 58, 69–74).—Electrolysis of a solution containing (per litre) 50 g. of lead sulphate, 200 g. of sodium hydroxide, and a small amount of glue, at 55° with a cathodic current density of 7 amp./ft.² in a diaphragm cell, yielded crystalline but strongly adherent deposits of lead at current efficiencies over 90%. Red lead is simultaneously formed at the anode, but the best yield of this substance was obtained by electrolysis of a solution containing (per litre) 55 g. of lead sulphate and 240 g. of sodium hydroxide at 65–70° with a current density of 400–500 amp./ft.² at a lead-silver alloy anode. The anolyte was moderately stirred and the current efficiency of red lead production reached 23%. Since sodium sulphate can be crystallised from the spent electrolyte and freed from lead compounds by washing with sodium hydroxide solution, it is possible to recover three separate products from this single electrolytic process.

H. J. T. ELLINGHAM.

Town's gas for heat-treatment furnaces. WALTER. —See II. **Determination of Curie points.** REGNER. —See XI.

PATENTS.

Metallurgical furnace. R. D. PIKE (U.S.P. 1,746,904, 11.2.30. Appl., 3.9.27).—High reaction temperatures with the use of oxygen or oxygenated air are attained by inducing combustion downwardly in the central lower part of the furnace within an inverted conical cavity formed by the charge, the walls being protected by feeding additional material as the charge is melted away. The base of the cone is formed by a water-cooled bell and the mixture of fuel and oxygen is injected through a pipe into the bottom of the hollow cone. Damage to the refractory lining is thus minimised.

C. A. KING.

Cupola furnace. T. P. ANTHONY (U.S.P. 1,747,208, 18.2.30. Appl., 27.3.29).—A section of the wall of a cupola is made into an air-regenerative system by allowing some of the furnace gases to pass through the hollow section and around air ducts leading to the tuyères.

C. A. KING.

Iron smelting. R. FRANCHOT, Assr. to FERRO CHEMICALS, INC. (U.S.P. 1,756,349, 29.4.30. Appl., 15.3.28).—About 20–25% of the blast gases is removed from just above the hot zone of the iron blast furnace and utilised for preheating the blast air to 370–480°. The ore charge is so regulated that the ratio $\text{CaO} + \text{MgO} : \text{SiO}_2 + \text{Al}_2\text{O}_3$ does not exceed 8 : 10. A. R. POWELL.

Case-hardening of ferrous articles. P. A. E. ARMSTRONG (U.S.P. 1,748,378, 25.2.30. Appl., 6.10.28. Renewed 31.10.29).—The articles are heated at below 750° in a current of nitrogenous gas produced by the

decomposition of ammonia on the surface of hot carbon activated with a nickel catalyst. A. R. POWELL.

Case-hardening [steel] by nitrogenisation. ELECTRO METALLURGICAL CO., Asscs. of A. B. KINZEL (B.P. 314,423, 27.2.29. U.S., 27.6.28).—The articles are heated at below 580° in a molten bath containing a high proportion of alkali cyanide, e.g., in a eutectic mixture of sodium and potassium cyanides. A. R. POWELL.

Nitridation of steel articles. R. SERGESON, Assr. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,748,623, 25.2.30. Appl., 28.3.29).—A nitride case-hardened layer of relatively large thickness can be obtained by heating alloy steels in ammonia, provided that after working the metal into shape it is annealed at 900°, quenched, and tempered at 650° before nitriding. A. R. POWELL.

[Removal of nitrogen from] steel. M. A. GROSSMANN and D. WILLIAMS, Assrs. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,748,217, 25.2.30. Appl., 28.7.27).—The steel is treated in the ladle with a small quantity of ferrozirconium containing silicon, calcium fluoride being used as a flux. The nitrogen is converted into zirconium nitride, part of which is removed in the slag.

A. R. POWELL.

Iron-carbon alloy. A. SAUVEUR, Assr. to AMER. ROLLING MILL Co. (U.S.P. 1,745,645, 4.2.30. Appl., 4.6.25).—A steel capable of being quenched or case-hardened contains more than 0.05% C and not more than 0.035% Mn, 0.035% Si, 0.01% P, and 0.03% S.

C. A. KING.

Manufacture of low-carbon ferro-alloys [non-rusting iron]. F. M. BECKET, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,748,750, 25.2.30. Appl., 25.2.27).—An iron-chromium alloy with a relatively high carbon content is blown with pure oxygen until the carbon is reduced to 0.2% and the metal becomes very hot; hydrogen is then blown through the bath until the carbon is reduced to 0.1%.

A. R. POWELL.

Silicothermic metallurgy. [Manufacture of ferro-chromium.] W. C. READ, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,758,465, 13.5.30. Appl., 21.11.28).—A mixture of finely-divided chromite, ferrochrome-silicon, sodium nitrate, and lime is ignited from below, e.g., by charging it on to a bath of molten iron or slag or by means of a primer consisting of a mixture of 4 pts. of dry sodium dichromate and 1 pt. of silicon. The resulting product contains more than 60% Cr, less than 3% Si, less than 0.1% C, and about 0.2% N.

A. R. POWELL.

High-silicon and high-manganese steel. W. C. HAMILTON, Assr. to AMER. STEEL FOUNDRIES (U.S.P. 1,746,586, 11.2.30. Appl., 21.4.27).—A pearlitic manganese steel containing 0.2–0.6% C, 1–2.25% Mn, and 0.5–1.5% Si is claimed.

A. R. POWELL.

[Iron-nickel] magnetic material. G. W. ELMEN, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,757,178, 6.5.30. Appl., 2.4.29).—A magnetic material having a constant permeability above 500 consists of an alloy of iron with 30–70% Ni and up to 12% Cr, Mo, and/or W.

A. R. POWELL.

Non-magnetic material. G. W. ELMEN, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,745,612, 4.2.30.

Appl., 24.11.28).—An iron alloy applicable to the armouring of submarine signalling cable contains 20—30% Ni, 3—4% Mo, and 0.15% Mn. C. A. KING.

[Nickel-iron-silver] alloy. M. E. BARKER, Assr. to H. H. SEMMES (U.S.P. 1,757,508, 6.5.30. Appl., 5.9.28).—An alloy of 37—47% Ni, 45—55% Fe, and 3—13% Ag, preferably 50% Fe, 42% Ni, and 8% Ag, is claimed. A. R. POWELL.

Deoxidation of copper. C. S. SMITH, Assr. to C. R. HAYWARD (U.S.P. 1,755,309, 22.4.30. Appl., 8.7.26).—Molten copper is deoxidised by poling in the usual way until the metal contains 0.12—0.15% O and is then cast into rods or bars. The latter are heated at 750—850° in an atmosphere of hydrogen until the desired reduction in the oxygen content is obtained. The metal is then severely worked by rolling or forging in a non-oxidising atmosphere at 900—950°. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 334,430, 24.10.29).—An alloy of aluminium with 0.1—4% Mg, 0.5—2.5% Ni, 0.3—1.5% Fe, 0.5—5% Si, 0—0.5% Ti, 0—0.2% Mn, 0.01—0.2% Cr, and 0—0.5% Cu, to which is added 0.1% Na just before casting, is claimed. A. R. POWELL.

Aluminium-base alloys. ALUMINIUM, LTD., Assees. of R. S. ARCHER and L. W. KEMPF (B.P. 334,656, 5.7.29. U.S., 22.12.28).—Aluminium alloys suitable for the manufacture of pistons for internal-combustion engines contain 7—15% Si, 0.2—3% Mg, 0.5—7% Ni, and 0.3—7% Cu. The preferred composition comprises aluminium alloyed with 14% Si, 2% Ni, 1% Mg, and 0.75% Cu; the alloy is aged at 125—150° for 25 hrs. after quenching from 530°. A. R. POWELL.

Production of tarnish-resisting silver and silver plate. W. S. MURRAY, Assr. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,758,293, 13.5.30. Appl., 19.9.24. Renewed 3.10.29).—Silver or silver plate is immersed in a cold solution containing 20 g. of iodine and 40 g. of potassium iodide per litre or heated at 175° in an atmosphere containing iodine vapour, whereby it becomes coated with a tarnish-resistant film of iodide which may be burnished and polished. A. R. POWELL.

Welding agent for magnesium alloys. I. G. FARBENIND, A.-G. (B.P. 313,487, 25.4.29. Ger., 12.6.28).—The flux consists of a mixture of halides, the cations of which are lithium and potassium, and the anions either chlorine and fluorine or bromine and fluorine. The mixture may be employed in the dry state, as a paste, or, preferably, as a saturated solution. H. ROYAL-DAWSON.

Production of tinplate. RASSELSTEINER EISENWERKS-GES. A.-G., and H. FRÄMBS (B.P. 310,061, 20.4.29. Ger., 20.4.28).—The annealing treatment of the plates consists in heating them either singly or in small packages to about 900°. H. ROYAL-DAWSON.

Manufacture of bearing metals. R. J. SHOE-MAKER, Assr. to S. & T. METAL CO. (U.S.P. 1,745,720—1, 4.2.30. Appl., [A] 29.4.27, [B] 16.9.27. Renewed, [B] 8.11.29).—A bearing-metal alloy comprises lead with (A) up to 1% Na, 0.1—0.25% Ca, and 0.02—0.1% Al; with or without 1—5% Sn; or (B) 0.01—0.05% Li,

0.2—0.8% Na, 0.1—0.7% Ca, 1—2% Sn, and 0.02—0.1% Al. A. R. POWELL.

[Inhibitor for use in] pickling of metals. BARRETT Co., Assees. of P. J. COLE and C. BANTA (B.P. 334,418, 11.10.29. U.S., 31.10.28).—About 0.02% of a naphtho-nitrile or other aromatic nitrile is added to a sulphuric acid pickling bath. A. R. POWELL.

Chemical reagents. [Inhibitors for iron-pickling baths.] F. H. RHODES (U.S.P. 1,746,676—1,746,680, 11.2.30. Appl., 18.7.29).—The inhibitor consists of (A) a monoalkyl-substituted quinoline; (B) a monosubstituted acridine derivative; (C) an amino-derivative of acridine, e.g., 3 : 6-dimethyl-2 : 7-diamino-acridine; (D) dinaphthacridine or a similarly constituted derivative of acridine; or (E) diquinolyl or a derivative thereof. A. R. POWELL.

[Inhibitor for baths used in the] pickling and cleaning of metals. P. I. MURRILL, Assr. to R. T. VANDERBILT Co. (U.S.P. 1,748,494, 25.2.30. Appl., 2.5.29).—The inhibitor comprises a compound having the general formula $C_nH_{2n}(NRX)_2$, where X is an acid radical and RN is an organic base, e.g., an alkylene-dipyridonium salt. A. R. POWELL.

Degreasing of metal articles. A. K. CROAD. From UDYLLITE GES.M.B.H. (B.P. 308,363, 22.3.29).—Turkey-red oils or alkyl-substituted aromatic sulphonic acids or their salts are added as wetting agents to the usual aqueous solutions of alkaline substances employed for cleaning articles to be electroplated. H. ROYAL-DAWSON.

Production of a phosphate coating on metal. M. GREEN and V. M. DARSEY, Assrs. to PARKER RUST-PROOF Co. (U.S.P. 1,755,391, 22.4.30. Appl., 29.4.29).—The articles are dipped for a short time in a freshly prepared bath before being transferred to the main parkerising bath. A. R. POWELL.

Manufacture of chromium-coated wire. H. K. RICHARDSON, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,745,912, 4.2.30. Appl., 3.5.23. Renewed 8.9.26).—Nickel-steel wire is cleaned by making it first the cathode in a bath of alkali hydroxide or carbonate, and then, after washing, the anode in a dilute sulphuric acid bath until it becomes passive. The wire is then chromium-plated by passing it continuously through the usual chromic acid bath. A. R. POWELL.

Separation of comminuted matter (U.S.P. 1,758,422).—See I. **Calcined bauxite** (U.S.P. 1,756,425). **Copper from spent lyes** (B.P. 310,425). **White zinc chloride** (U.S.P. 1,747,751). **Treatment of mixed oxy-salts** (U.S.P. 1,756,007).—See VII. **Sheets for building purposes** (B.P. 333,978).—See IX. **Nickel anode** (U.S.P. 1,757,714). **Arc-welding and cutting** (U.S.P. 1,746,081 *et seq.*).—See XI. **Film-forming element** (U.S.P. 1,746,751). **Reproducing designs in metal** (B.P. 331,856).—See XXI.

XI.—ELECTROTECHNICS.

Materials for dry cells. I, II. S. MAKINO (J. Soc. Chem. Ind., Japan, 1930, 33, 326).—I. Neither the *E.M.F.* nor the capacity of a dry cell is influenced by the carbon content of the particular kind of carbon used..

They are but little influenced by the difference in resistance between natural and artificial graphite.

II. The capacity of a cell is definitely related to the grain size of the manganese dioxide and graphite, and to the rate of discharge. The electrode distance must be changed according to the rate of discharge.

C. W. GIBBY.

Hydrolysis in standard cells. G. A. HULETT (Trans. Amer. Electrochem. Soc., 1930, 58, 13—18).—The *E.M.F.* of two standard cells, made up in 1904, decreased by over 100 microvolts during the first 2 years, but subsequent changes have not exceeded 60 microvolts, and the values in 1928 were not more than 20 microvolts from those in 1906. A cell made up in 1906 with mercurous sulphate which had been washed with "equilibrium water" (water which has been shaken with mercurous sulphate and mercury at 25°), and with equilibrium water saturated with cadmium sulphate crystals, and the cell filled with this latter solution, has kept the most constant *E.M.F.* of any, the change in 23 years being only 5 microvolts. Other recent work in the author's laboratory (A., 1929, 1147, 1392) is discussed. The marked changes in *E.M.F.* of cells made up with mercurous sulphate in relatively large crystals is ascribed to the formation of a crust of basic sulphate on the crystals when the soluble products of hydrolysis diffuse away. The upper layers of the mercurous sulphate will be affected first and the *E.M.F.* will not alter until this change has penetrated to the layer in contact with the mercury. The behaviour of cells with mercurous sulphate layers of different thicknesses is in harmony with this explanation.

H. J. T. ELLINGHAM.

Magnetometric determination of the Curie points. A. RÆGNER (Iron and Steel Inst., Sept., 1930. Advance copy. 22 pp.).—Three simple devices for the determination of the Curie points are described. With a low-frequency magnetometer, a resistance furnace fed by alternating current is used for heating, as well as for generating an alternating magnetic field. The *E.M.F.* induced in a secondary winding, of which the specimen forms the core, is applied to the grid of a thermionic valve, and changes in the permeability of the specimen are observed. A high-frequency magnetometer, based on the resonance principle, has the furnace coil and specimen as part of a receiving condenser circuit. Another method employs the change in amplitude of the anode current of a valve-generator according to the conditions in the oscillating circuit, of which the furnace and specimen form a part. With these devices A2 temperatures are shown by sharply-reversible changes, the existence of two cementite changes at 60° and 210° is confirmed, and steels containing 0.4—0.9% C are shown to lose their magnetism between A1 and A3.

C. J. SMITHELLS.

Smoke prevention. GIBBS. Dew point of flue gases. JOHNSTONE.—See I. Reduction of alumina. NEWMAN and BROWN.—See VII. Corrosion of aluminium by alkali. STOREY. Electrolytic recovery of lead from lead sulphate waste. FINK and GREENSPAN.—See X. Purification of water. BARTOW and JEBENS.—See XXIII.

PATENTS.

Electrochemical electric current rectifiers. H. ANDRÉ (B.P. 334,093, 4.10.29. Fr., 5.10.28).—An anode of porous silver sulphide impregnated with regenerating material, *e.g.*, sulphuric acid, and a cathode of oxidisable metal are arranged in contact within an evacuated, hermetically-sealed vessel. A method of manufacturing the anode is claimed. J. S. G. THOMAS.

Nickel anode. G. B. HOGABOOM, ASST. to HANSON-VAN WINKLE-MUNNING Co. (U.S.P. 1,757,714, 6.5.30. Appl., 28.2.29).—The anode contains more than 98% Ni, more than 0.1% Si, and 0.1% Mn, and is substantially free from carbon. A. R. POWELL.

Electric arc-welding and cutting. (A) H. M. HOBART, (B) P. K. DEVERS, (C) I. LANGMUIR and P. P. ALEXANDER, (D, F, G) E. THOMSON, (E) E. THOMSON and P. P. ALEXANDER, and (H—K) P. P. ALEXANDER, ASSES. to GENERAL ELECTRIC Co. (U.S.P. 1,746,081, 1,746,191, 1,746,196, 1,746,202—5, 1,746,207—9, and 1,746,210, 4.2.30. Appl., [A] 29.6.26, [B] 25.6.26, [C] 5.9.25, [D, I] 26.8.25, [E] 6.10.25, [F] 18.7.29, [G] 3.9.29, [H] 26.12.24, [J] 7.10.25, [K] 10.1.27. Renewed [I] 8.10.27, [K] 6.1.30).—The arc produced between the welding rod and the work is surrounded by a non-oxidising atmosphere comprising (A) helium; (B) argon, diluted or not with hydrogen or nitrogen; (C) nitrogen containing about 6% H₂ to prevent formation of nitrides; (D) a mixture of carbon monoxide and hydrogen, *e.g.*, water-gas; (E) the vapour of ethyl or methyl alcohol, which dissociates into a mixture of carbon monoxide and hydrogen; (G) a mixture of carbon dioxide and propane or other gaseous hydrocarbon to form water-gas; (H) hydrogen; (I) coal gas or natural gas (this is particularly adapted for blowing away the molten metal during cutting operations with the arc); (J) ammonia or a volatile organic amine which dissociates into nitrogen and hydrogen in the arc; or (K) the vapour of a hydrocarbon when a carburised weld is required. (F) The gas is admitted into the arc through a valve automatically regulated by the voltage of the arc. A. R. POWELL.

Indirectly-heated cathodes for vacuum tubes. ARCTURUS RADIO TUBE Co., ASSES. of S. RUBEN (B.P. 306,832, 25.2.29. U.S., 25.2.28).—A metal filament, *e.g.*, of tantalum, coated with oxide produced by heating, is coated with material of high dielectric strength, *e.g.*, silica. J. S. G. THOMAS.

[Wood-pulp filling for] primary and secondary electric batteries. M. HOLBROOK (B.P. 314,010, 27.2.30).

Thermoelectronic rectifier. S. LOEWE (B.P. 308,311, 21.3.29. Ger., 21.3.28).

Thermo-electric batteries. J. PETRÍK (B.P. 313,602, 30.5.29. Czechoslov., 15.6.28).

Electric-discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 334,519, 3.5.29).

Cathode-ray tubes. ASSOCIATED TELEPHONE & TELEGRAPH Co. (B.P. 313,882, 11.6.29. U.S., 18.6.28).

Röntgen tubes. F. ROTHER (B.P. 334,149, 24.12.29).

Röntgen-ray apparatus. SIEMENS-REINIGER-VEIFA GES. F. MEDEZIN. TECH.M.B.H. (B.P. 334,818, 2.1.30. Ger., 23.2.29).

Acetylene (B.P. 332,917).—See II. Photochemical polymerisation (U.S.P. 1,746,168).—See III. Zinc oxide (U.S.P. 1,743,964). Treatment of mixed oxy-salts (U.S.P. 1,756,007). Per-compounds (B.P. 313,124).—See VII. Magnetic material (U.S.P. 1,757,178). Non-magnetic material (U.S.P. 1,745,612). Degreasing metal articles (B.P. 308,363). Chromium-coated wire (U.S.P. 1,745,912).—See X. Articles from thermoplastic materials (B.P. 333,879).—See XIV. Surgical pads (B.P. 333,980).—See XX. X-Ray screens (B.P. 318,152). Reproducing designs in metal (B.P. 331,856).—See XXI.

XII.—FATS; OILS; WAXES.

Examination of soaps containing salicylic acid for free salicylic acid. E. SCHLENKER (Chem. Umschau, 1930, 37, 262—263).—A salve prepared from potash soap, vaseline oil, and salicylic acid was examined by a modified extraction method, and the total absence of free salicylic acid was confirmed, double decomposition to sodium salicylate and free fatty acid having occurred. A special extraction procedure was necessary, as by the usual methods unsaponifiable matter and potassium salicylate were carried over into the clear ethereal extract in the presence of soap and vaseline; with soda soap troublesome emulsions were formed from which extracts free from sodium salicylate were unobtainable.

E. LEWKOWITSCH.

Lipeometer fat test for determining fat and oils in fat-bearing materials, press cake, or refuse. R. SCHWARZ (Oil & Fat Ind., 1930, 7, 335—336, 347).—The variation of sp. gr. of solutions of fats in *o*-dichlorobenzene with the amount of fat present is practically linear, hence the fat content of seeds, press cakes, etc. may be determined rapidly as follows. The material (100 g., ground to 60—80-mesh) is stirred with 600 g. of the solvent (in an aluminium beaker) at ordinary temperatures, and after 5 min. the solution is filtered under suction and its sp. gr. measured with a hydrometer ("lipeometer"), the scale of which is graduated to read directly in percentages of oil; suitable temperature correction is made from a previously-prepared scale. The results by this method agreed well with those from the standard A.O.C.S. determination (mean error 0.09%).

E. LEWKOWITSCH.

[Separation and determination of] solid fatty acids. W. F. BAUGHMAN and G. S. JAMIESON (Oil & Fat Ind., 1930, 7, 331—332).—A method is described by which the lead salt-alcohol separation may be applied to the oil sample without preliminary isolation of the total fatty acids as required by the Twitchell process (B., 1921, 817 A). Not more than 6 g. of oil (containing 1—1.5 g. of solid acids) are saponified with 40 c.c. of alcoholic potash (40 g. per litre of 95% alcohol), the excess alkali is neutralised with glacial acetic acid (one drop in excess being added), and the solution is made up to 150 c.c. with 95% alcohol, then heated to boiling, and hot lead acetate solution (5 g. per 50 c.c. of alcohol; the excess of lead salt is required in the presence of potassium soaps and potassium acetate) is run in. The mixture is allowed to cool slowly and is kept overnight at 15°. The precipitated lead soaps

are collected, washed with cold 95% alcohol until the washings remain clear on dilution with water, and redissolved by boiling with about 100 c.c. of alcohol and 0.5 c.c. of glacial acetic acid. The lead salts obtained by cooling, filtering, and washing as before are heated with hydrochloric acid to recover the solid fatty acids, which are dried at 110° (in a stream of carbon dioxide if isooleic acid is present). Results in good agreement with those from the Twitchell method are obtained, isooleic acid being separated quantitatively in both cases.

E. LEWKOWITSCH.

Determination of unsaturation of fats and fatty acids. III. Wijs iodine value. J. VAN LOON (Chem. Umschau, 1930, 37, 257—262; cf. B., 1930, 724).—Contrary to the experiences with fresh oil, a linseed oil that had been stored some time showed a Wijs iodine value that gradually increased with the time of reaction (cf. Schmidt-Nielsen and Owe, "Bestimmung der Jodzahl," Oslo, 1923); this was shown to be due to gradual depolymerisation of the old oil and not to substitution of halogen by the reagent, which with fresh oils, fatty acids, and esters (in the absence of conjugated acids) gives constant and accurate figures, even with great excess of reagent and prolonged reaction time. For the most accurate discriminatory purposes it is best to perform the Wijs test on the total fatty acids previously freed from unsaponifiable matter, as the latter (*e.g.*, of parsley-seed oil) may contain appreciable amounts of highly unsaturated substances in which substitution may also occur.

E. LEWKOWITSCH.

Determination of iodine values. S. YUSHKEVICH (Masloboino Zhir. Delo, 1930, No. 2, 9—16).—A comparative study. For rapidity and cheapness Kaufmann's method is preferred.

CHEMICAL ABSTRACTS.

Composition of the saturated fatty acids of Japanese sardine oil. H. IKUTA and S. UENO (J. Soc. Chem. Ind., Japan, 1930, 33, 245—246 B).—The solid fatty acids (24—26%; m.p. 52—52.5°, iodine value 24.5, neutralisation value 207.3) of a sardine oil (iodine value 179.1, thiocyanogen value 90.2, saponif. value 193.8, n_D^{20} 1.4801, m.p. of mixed fatty acids 35—35.6°) were examined by fractional distillation of the methyl esters. Palmitic acid (the principal constituent), myristic and stearic acids were isolated, and the presence of butyric (?), arachidic, and behenic acids, and of an acid, $C_{24}H_{48}O_2$, was recognised.

E. LEWKOWITSCH.

New hydrocarbons produced during hydrogenation of fish oils. S. UENO (J. Soc. Chem. Ind., Japan, 1930, 33, 264—266 B).—Volatile products obtained during the hydrogenation of fish oils had saponif. value 131.0, iodine value 25, acid value 95.0. Examination of the unsaponifiable fraction of these products showed it to consist mainly of hydrocarbons (probably isoparaffins). Fractional distillation of the unsaponifiable portion insoluble in methyl alcohol gave mixtures of hydrocarbons, acetyl value 0, yielding no ether-insoluble polybromides. Hydrocarbons with 1—2 double linkings are also present. The hydrocarbons $C_{13}H_{28}$, $C_{16}H_{34}$, $C_{17}H_{36}$, $C_{18}H_{38}$, $C_{19}H_{40}$, $C_{20}H_{42}$ were isolated, and $C_{10}H_{22}$ and $C_{11}H_{24}$ may have been present (cf. occurrence of pristane in shark-liver oil). The greater part of the hydrocarbons in the products examined

appear to have been produced during the hydrogenation.

E. LEWKOWITSCH.

Semi-drying oils and their oxidation in presence of different catalysts. V. CHELINCHEN (Masloboino Zhir. Delo, 1929, No. 5, 49–52).—The semi-drying oils contain little or no linolenic acid or its isomerides, 10–20% of saturated acid, and 73–85% of linoleic and oleic acids (1.5–2.2:1). The drying oils contain 23–41% of linoleic acid, or, if only 14% is present, a high percentage (62–74%) of linolenic acid is present; the percentage of oleic acid varies widely, and that of the saturated acids varies from 4.5 to 9%. Semi-drying oils could be made to dry by partial oxidation in presence of catalysts (lead oxide, cobalt oxide, manganese dioxide, but not other metallic oxides, sulphur monochloride, dimethylaniline, or charcoal).

CHEMICAL ABSTRACTS.

Application of the thiocyanogen value. IV. **Analysis of oils containing linolenic acid.** I. **Composition of chrysalis oil.** W. KIMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 262–264B).—Kaufmann's work (cf. B., 1929, 401), showing that linolenic acid absorbs thiocyanogen at two double linkings only, is confirmed. A chrysalis oil, having iodine value 141.8 (Wijs), thiocyanogen value 93.0, total fatty acids 93.1%, solid fatty acids 23.89%, unsaponifiable matter 0.99%, has been analysed by the thiocyanometric method, which is considered less susceptible to experimental error than the bromination method. The liquid fatty acids are determined as oleic acid 29.2, linoleic acid 35.9, and linolenic acid 34.9% (cf. 29.8%, 48.9%, 21.3%, respectively, determined by bromination; B., 1929, 607).

E. LEWKOWITSCH.

Colorimetric determination of minute quantities of dissolved phosphorus in oils. H. WAEL (Dansk Tidsskr. Farm., 1930, 4, 197–208).—Stich's method (B., 1927, 851) is examined critically; it is necessary to control the concentration of phosphorus, oil, and reagents so that the silver phosphides produced do not coagulate, and the amount of silver nitrate solution added to the standard and unknown samples must be the same. In the modified test proposed, accurate results are obtained when the phosphorus content of the oil does not exceed 1 in 10,000; if more is present (determined by an approximate preliminary test) the sample should be suitably diluted with almond oil before the final determination, for which standard solutions are prepared containing 0.1, 0.09, 0.081, 0.073, 0.066, 0.059, and 0.053 mg. of phosphorus in 1 g. of oil made up to 10 c.c. with a mixture of ether, alcohol, and acetone (40:20:5 by vol.); then 1 g. of the (diluted) sample is diluted with this mixed solvent to 10 c.c. and 0.2 c.c. of a solution of silver nitrate in acetone (0.25 g./100 c.c.) is added to each solution, the colour developed being compared after 15 min.

E. LEWKOWITSCH.

Extinction of ethylene dichloride flames. JONES and KENNEDY.—See II. **Use of soya-bean oil in paints.** BELYAEV.—See XIII. **Fat content of Gruyère cheese.** SAJOUS.—See XIX. **Castor oil soaps in spirituous preparations.** MEYER.—See XX.

PATENTS.

Manufacture of soaps containing fat solvents. S. ZIMMERMANN (B.P. 334,177, 25.2.29. Austr., 2.10.28).—A small quantity of *n*-propyl or isopropyl alcohol is added, before, during, or after saponification, in order to facilitate the incorporation of large amounts of fat solvents in acid soaps.

E. LEWKOWITSCH.

Manufacture of powdered soap. A. WELTER (B.P. 332,590 and 332,599, 22.4.29).—(A) Electrolytes (*e.g.*, salt) are added to the liquid soap in order to cause salting out during the subsequent heating with open compressed steam or direct fire *etc.* to above 100° (1–10 atm.); the heated, grained-out soap is settled for a short time and atomised. (B) If compressed air (*e.g.*, at 10 atm.) is pumped into the pressure vessel containing the liquid soap, before or during the first stage of heating the soap and prior to atomisation, the use of hot air to dry the powder produced is rendered unnecessary.

E. LEWKOWITSCH.

Extraction of fat or other matters from animal carcasses, slaughterhouse refuse, *etc.* A. SOMMERMEYER (B.P. 310,542, 26.4.29).—The mixture of liquid and solid products from a continuous digester is separated continuously by a mesh screening device while being subjected to pressure in a digester. Hydrolysis of the glue-yielding constituents is thus greatly reduced.

E. B. HUGHES.

Decolorising fatty substances with adsorbents. P. W. PRUTZMAN (U.S.P. 1,745,952, 4.2.30. Appl., 20.4.27).—The oils *etc.* are decolorised by heating, out of contact with air, with a powdered magnesium silicate such as that described in U.S.P. 1,598,254 (B., 1926, 937), at temperatures rising from 105° to above 123°; the oil is cooled to the lower temperature before filtering.

E. LEWKOWITSCH.

Treatment of linseed oil. B. H. THURMAN, Assr. to GOLD DUST CORP. (U.S.P. 1,745,877, 4.2.30. Appl., 29.7.26).—Drying and semi-drying oils are polymerised by heating to 260–315° under a pressure of 2 in. Hg, or less, while a stream of non-oxidising gas or steam is passed through the mass; traces of metallic catalysts, *e.g.*, 0.01–0.1% of lead, may be used to accelerate the process, which yields, within 6 hrs., highly bodied pale oils of low acid value.

E. LEWKOWITSCH.

Production of edible oils from crude cottonseed oil. W. KELLEY, Assr. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,747,675, 18.2.30. Appl., 19.7.26).—The crude oil is agitated with 5–10% of water at about 47°, in order to coagulate colloidal matter which is removed with the water; the oil is then stirred at 100–105° with an acid-activated absorbent clay ("Filtrol") and, after filtering, is heated to about 233° at a pressure below 60 mm., while a current of superheated steam is passed through it to assist the removal of free fatty acids.

E. LEWKOWITSCH.

Manufacture of linoleum cement and linoleum. ARMSTRONG CORK CO., Asses. of E. CLAXTON (B.P. 305,656, 22.1.29. U.S., 9.2.28).—An anticoagulant, *e.g.*, rosin, resin acid or ester (1 pt., which furnishes the resinous constituent of the cement), is added to the drying oil (3 pts.) in order to delay coagulation when the mixture

is heated and oxidised at 77—195° in the usual apparatus until the product contains not more than 7% of matter extractable by light petroleum (*i.e.*, unoxidised oil), in addition to the anticoagulant. The oil may then be thickened to the desired consistency by continued heat treatment, with or without continued oxidation treatment. [Stat. ref.] E. LEWKOWITSCH.

Disintegration of cellular matter (U.S.P. 1,746,731).—See I. **Lubricants** (U.S.P. 1,758,446 and 1,758,598).—See II. **Wetting agents etc.** (B.P. 310,941). **Colouring of oils, fats, waxes** (B.P. 309,148).—See XIII. **Esters of *Hydnocarpus* fatty acids** (B.P. 311,236).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Special primers for house paints. F. C. SCHMUTZ, F. C. PALMER, and W. W. KITTELBERGER (Ind. Eng. Chem., 1930, 22, 855—860).—An accelerated test cabinet, representing wet exterior wall conditions, was devised for testing the relative durability of various paint systems. A series of trials is detailed and illustrated. The incorporation of varnish, *e.g.*, spar varnish, or synthetic resin varnish, into priming coats gives improved adhesion to wood, regardless of the type of pigment used. S. S. WOOLF.

Effect of priming-coat reduction and special primers on paint service on different woods. F. L. BROWNE (Ind. Eng. Chem., 1930, 22, 847—854).—The durability of paint systems on various soft woods is influenced considerably by the different adhesion of the priming coat to the dense summer wood and the lighter spring wood, respectively. A series of exposure tests of standard paints on woods that had given evidence of poor adhesion for paint in an earlier series of exposures is detailed. The function of the primer was studied by variation in the proportion of linseed oil and turpentine used in the priming coat, use of thinners other than turpentine, *e.g.*, benzene, reputed to afford better penetration of soft resinous wood, and the use of special primers containing red lead, zinc dust, aluminium powder, etc. The results of these exposures (lasting over 4 years) indicate that the flaking of paint from summer wood cannot be prevented by any of the means adopted in this series of trials, and the problem of adhesion is considered to be a fundamental one remaining unsolved. S. S. WOOLF.

Use of soya-bean oil in paints. N. BELYAEV (Masloboino Zhir. Delo, 1929, No. 6, 15—16).—The oil cannot substitute linseed or hempseed oil.

CHEMICAL ABSTRACTS.

Adhesion-tension cell in paint investigations. E. L. McMILLEN (Ind. Eng. Chem., 1930, 22, 890—893).—The accuracy of "wettability" measurements with the Bartell-Osterhof cell (B., 1928, 1) is improved by using more accurate surface tension values, constant temperature, and more densely packed pigment cake (by means of smaller pigment additions and higher pressures, *viz.*, 535 atm.). Results obtained with lithopone and various liquids, under these improved conditions, are of the same order as those previously obtained without the refinement of method (*cf.* B., 1930, 110). Confirmation was thus obtained of the fact that, contrary to accepted

views, the best flow characteristics are obtained with liquids of low wettability. The theory of wetting and of the adhesion-tension cell is discussed with reference to paint problems. It cannot yet be stated whether plasticity observed in mixtures of pigments with good wetting liquids is due to adsorbed liquid layers or to flocculation. S. S. WOOLF.

Wetting of pigments and other powders. W. D. HARKINS and R. DAHLSTROM (Ind. Eng. Chem., 1930, 22, 897—902).—The total energy of immersion of powdered crystalline titanite, stannite, and zinc oxides in various liquids was determined in a calorimeter, details of which are given. The values are higher for polar than for non-polar liquids. The data indicate that nearly all the energy is liberated in the adsorption of a unimolecular film from the liquid on the surface of the powder; thus very small quantities of impurities in the liquids often have a great effect on the wetting of powders. The orientation of molecules and the general energy exchanges at solid-liquid interfaces are also discussed. S. S. WOOLF.

Secondary esters and their use in lacquers. J. G. PARK and M. B. HOPKINS (Ind. Eng. Chem., 1930, 22, 826—830).—The properties of *sec.*-butyl-, amyl-, and hexyl acetates, *e.g.*, solvent power (dilution ratio etc.), water-solubility, evaporation rate, are tabulated. From observation of stability and resistance to blushing of lacquers containing them etc., these products are considered satisfactory for use in base lacquers or thinners. S. S. WOOLF.

Properties of shellac films. I. Resistance of shellac films from various varnishes to action of water and chemicals. M. VENUGOPALAN and M. RANGASWAMI (Ind. Eng. Chem., 1930, 22, 911—913).—The resistance to water, brine, acid, alkalis, alcohol, sulphur dioxide, and ammonia of shellac films deposited from solutions in methyl, ethyl, and propyl alcohols and from optimum mixtures containing these (*cf.* B., 1930, 110) was observed. The films from varnishes containing mixed solvents were, in general, less affected than those from single-solvent varnishes. Mixtures of methyl or ethyl alcohol with acetone are best adapted for shellac varnish making. S. S. WOOLF.

Resin formation in benzoles. HOFFERT and CLAXTON.—See II. **Viscosity etc. of acetylcellulose jelly.** ARAKI and KUSAGAWA.—See V.

PATENTS.

Manufacture of aqueous coating compositions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,570, 13.3.29).—Water is emulsified with at least twice its quantity of an anhydrous liquid cellulose lacquer which contains only constituents practically insoluble in water and in which chiefly solvents boiling below 150° are used. Cheapness, relative freedom from objectionable odours, and non-inflammability are claimed. S. S. WOOLF.

Dyeing or colouring of varnishes, waxes, fats, oils, and higher fatty acids. I. G. FARBENIND. A.-G. (B.P. 309,148, 5.4.29. Ger., 5.4.28).—These materials are coloured by dissolving in the liquid or molten substance a non-sulphonated, non-carboxylated mordant

dye and treating the solution (or mixture during dissolution), in absence of water, with a heavy-metal salt of an organic acid, preferably a resin acid, higher fatty acid, or naphthenic acid. *E.g.*, a solution of 4-chloro-*o*-aminophenol \rightarrow 2:3-hydroxynaphthoic anilide in warm cellulose laurate varnish is treated with cobalt naphthenate (red-violet); 4-nitro-*o*-aminophenol \rightarrow phenylmethylpyrazolone in warm linseed oil with cobalt stearate (yellow-brown); 3:5-dichloro-*o*-aminophenol \rightarrow β -naphthol in molten carnauba wax with copper naphthenate (blue-red); 3:5-dichloro-*o*-aminophenol \rightarrow 2:3-hydroxynaphthoic anilide and triethanolamine in warm benzylcellulose varnish with cobalt naphthenate (violet). C. HOLLINS.

Manufacture of coloured polyvinyl ester varnishes. I. G. FARBERIND. A.-G. (B.P. 334,145, 3.12.29. Ger., 3.12.28).—Polyvinyl ester varnishes are coloured by means of water-insoluble amino- or polyamino-anthraquinone dyes, *e.g.*, 1:4-diamino-2:3-diphenoxyanthraquinone (red), 1:4-di-*p*-toluidinoanthraquinone (green), 1-*p*-toluidino-4-methylaminoanthraquinone (greenish-blue), 1:4-di-*p*-toluidino-8-hydroxyanthraquinone (olive-green); the second and fourth of these are soluble in the varnish and give transparent films. C. HOLLINS.

Manufacture of synthetic gummy or resinous materials. CANADIAN ELECTRO PRODUCTS CO., LTD., Assees. of (A) F. W. SKIRROW, (B) H. W. MATHESON and F. W. SKIRROW (U.S.P. 1,746,615 and 1,746,665, 11.2.30. Appl., [A] 15.8.27, [B] 28.2.27).—Resins useful as substitutes for chicle are obtained by exposing a mixture of a vinyl ester (acetate) and an aldehyde (acetaldehyde) to (A) actinic rays, *e.g.*, from a mercury-arc lamp, preferably at 30–40°, or (B) to heat-treatment at 100°, with or without a catalyst (hydrogen chloride, hydrogen peroxide). Contact with iron or copper should in each case be avoided. C. HOLLINS.

Manufacture of [plastic] artificial masses. CON-SORT. F. ELEKTROCHEM. IND. G.M.B.H., and O. DÖRR, Assees. of DÖRR & HOFMAN (B.P. 313,578, 14.6.29. Ger., 14.6.28. Cf. B.P. 271,090; B., 1928, 793).—Filling materials, *e.g.*, ground slate, cork, or wood meal, are incorporated in a solution of an alcohol-soluble polymerised vinyl ester, in such proportions that the product can be sprayed. E. LEWKOWITSCH.

Manufacture of condensation products from a urea and formaldehyde. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,634, 19.4.29).—The dispersions or solutions obtained by condensing urea or its derivatives with formaldehyde in organic solvents (cf. B.P. 319,251; B., 1929, 1047) are submitted to steam-distillation (if desired, with the application of a vacuum and/or moderately superheated steam) in order to remove the volatile constituents and solvents. The residual watery gel is allowed to settle until the bulk of the water has separated spontaneously, and is then rolled, kneaded, and dried at 50–60°, to yield a clear glassy product which may be subjected to fractional extraction with organic solvents. E. LEWKOWITSCH.

Resinous substances and compositions containing them. BRIT. CELANESE, LTD. (B.P. 311,657,

13.5.29. U.S., 12.5.28).—Lactic acid, preferably of 90% concentration, is heated under reflux for several hours, and the volatile matter is then distilled off. The light-coloured resinous product (m.p. 30–35°) may be used in conjunction with cellulose acetate, resins, plasticisers, solvents, colouring materials, etc. S. S. WOOLF.

[Resinous] saturant for fibrous bases and its preparation. I. J. NOVAK (B.P. 334,232, 27.5.29).—Cresylic acid is condensed with formaldehyde by heating with 0.25–0.75 mol. of ammonia per mol. of cresylic acid, and the product is heated with tung oil until the solution is clear at normal temperature; it is afterwards cooled to retard further reaction. F. R. ENNOS.

Process and apparatus for making white lead. E. C. WALKER (B.P. 334,751, 11.10.29).—See U.S.P. 1,732,490; B., 1930, 156.

Manufacture of phenolic resins. N. STRAFFORD and E. E. WALKER, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,202–3, 16.9.30. Appl., 30.6.28. U.K., 7.7.27).—See B.P. 296,514; B., 1928, 826.

Manufacture of abrasive article [flexible sand-paper]. R. P. CARLTON, Assr. to MINNESOTA MINING & MANUF. CO. (U.S.P. 1,775,631, 16.9.30. Appl., 13.3.26).—See B.P. 267,516; B., 1928, 826.

Manufacture of moulded articles [from wood chips or sawdust and synthetic resin]. BOIS BAKÉLISÉ and LA BAKELITE (B.P. 334,804, 6.12.29. Fr., 7.12.28).

Polymerisation process (U.S.P. 1,746,168). **Polymerised vinyl esters** (B.P. 319,682). **Polymerised styrene** (B.P. 307,936). **Anti-agers** (B.P. 333,941).—See III. **Azo pigments** (B.P. 332,932 and 332,940).—See IV. **Shaped fibrous materials** (B.P. 312,178). **Cellulose compositions of low inflammability** (B.P. 312,609).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Extraction of gutta-percha leaf. A. W. K. DE JONG (Rec. trav. chim., 1930, 49, 827–840).—Extraction of finely-powdered gutta-percha leaf with benzene at 25° and addition of 85–95% alcohol to the extract precipitates a gutta-percha which, after melting in water, pressing to a thin film, and then fusing at 100° for 1 hr., contains a large proportion of second-grade (immature) material. It is shown that second-grade gutta-percha is the pure hydrocarbon modified by melting. Shavings of gutta-percha melted in water at 62° can be drawn out to give threads which shrink to the original size in warm water. Gutta-percha appears to exist in three forms with differing solubilities in benzene and light petroleum; the difference is attributed to the size of the particles. Extraction of the leaf with light petroleum (b.p. 80–100°) at 35° and cooling the extract to 17° gives a pure gutta-percha only slightly soluble in benzene at 16°. This method of extraction is recommended.

H. BURTON.

Conditions affecting the vulcanisation of rubber. V. **Effect of accelerators on the heat of vulcanisation.** Y. TOKABE (J. Soc. Chem. Ind., Japan, 1930, 33, 275–276 B; cf. B., 1930, 520).—The heat of vulcanisation of rubber compounds with different amounts of

sulphur and various organic accelerators was followed by means of a differential thermocouple. The profiles of the heating curves were similar to those obtained with unaccelerated compounds, except that the temperature maxima (corresponding to disaggregation maxima of the rubber hydrocarbons) appeared earlier in the former case. The degree of sulphur combination is much the same in both cases. The experiments showed that the accelerators favour the combination with sulphur by promoting the disaggregation of the micellar structure of the rubber.

E. LEWKOWITSCH.

PATENTS.

Dispersed rubber isomeride. H. L. TRUMBULL, Assr. to B. F. GOODRICH Co. (U.S.P. 1,744,844, 28.1.30. Appl., 2.6.27).—Rubber isomerides, *e.g.*, of the thermoplastic type obtainable by heating with isomerising agents, are subjected to mechanical working on a warm mill, while a hydrophilic colloid, *e.g.*, casein, glue, or colloidal clay, is incorporated, and water is then added gradually. A dispersion is thus finally obtained with the colloid paste as continuous phase surrounding the rubber isomeride in such condition that dilution and concentration are possible. If the rubber isomeride is brittle an alternative procedure is to reduce it to powder and subject a mixture with water and a protective colloid to the action of a colloid mill. D. F. TWISS.

Rubber latex cement and its preparation. B. DEWEY and E. C. CROCKER (U.S.P. 1,745,084, 28.1.30. Appl., 20.11.24).—Finely-divided sulphur is dispersed into rubber latex together with a smaller proportion of bentonite; the presence of the latter effectively maintains the sulphur in suspension. A permanent suspension of this type, capable, when dried, of vulcanisation to a hard condition, can be produced by adding a mixture of flowers of sulphur (90 pts.), bentonite (15 pts.), saponin (3 pts.), and water (590 pts.) to latex (462 pts.) containing about 38% of rubber. D. F. TWISS.

Product and process for rubber compounding. A. B. COWDERY, Assr. to BARRETT Co. (U.S.P. 1,743,433, 14.1.30. Appl., 22.11.24).—The volatile constituents are distilled from coal tar in such a way as to give a residue containing a high proportion (60%) of uncombined carbon or carbonaceous matter insoluble in benzene. The residue, having d 1.30–1.35 and m.p. above 176°, can be incorporated in rubber up to the extent of about 15%, and does not produce excessive discoloration of brown mixtures. D. F. TWISS.

Vulcanisation of rubber. W. SCOTT, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,743,243, 14.1.30. Appl., 28.9.27).—An accelerator of vulcanisation is formed by heating an organic derivative of ammonia, *e.g.*, piperidine or diphenylguanidine, with a cyanogen halide reaction product of a mercaptan, *e.g.*, with the product from cyanogen chloride and sodium mercaptobenzthiazole. D. F. TWISS.

Manufacture of elastic products. I. G. FARBENIND. A.-G. (B.P. 318,967, 3.9.29. Ger., 13.9.28).—Highly elastic products are made from esters of polymeric carbohydrates containing residues of saturated or unsaturated cyclic-substituted fatty acids in addition to one or more residues of simple saturated or unsaturated fatty acids, *e.g.*, cellulose naphthenate oleate or starch naphthenate oleate stearate. These can be

worked by any process suitable for working rubber, and can be calendered and vulcanised. They may be used alone or mixed with colouring matters, natural or synthetic rubber, other polymeric carbohydrate compounds, etc.

D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER Co., LTD., and (A) W. H. CHAPMAN, D. W. POUNDER, and E. A. MURPHY, (B) E. A. MURPHY and E. W. B. OWEN (B.P. 332,525—6, 21.3.29).—(A) Mixtures of compounded aqueous dispersions of rubber or similar material to which lather-forming substances, *e.g.*, ammonium oleate, have been added together with substances having delayed coagulating action, *e.g.*, sodium silicofluoride or ammonium persulphate, are converted into a frothy condition by stirring with mesh-work paddles and/or by injecting air or other means. The frothy mass is poured into open moulds and allowed to coagulate, with or without the aid of heat; it is then vulcanised and dried. Alternatively, the coagulating agent may be added to the froth subsequent to its formation, the agitation being continued for a short time longer to ensure uniform distribution. The product is a material of sponge-like or cellular structure. (B) Frothed mixtures of compounded aqueous dispersions of rubber etc. produced as above are formed into shape by spreading, pouring into shallow trays, or by dipping processes etc., and are then "set" by heating in a gentle current of air or by other means. Articles of sponge rubber, *e.g.*, sheet for mats, insulating material, etc., also vulcanite sponge sheet, are thus obtained. D. F. TWISS.

Rubberised material. P. H. W. CLOUD (B.P. 332,537, 19.4.29).—A rubber felt-like material, which may be moulded or patterned, is produced by mixing rubber with animal fibres and fillers, with or without a vulcanisation accelerator and sulphur, and heating to a temperature below that capable of deleteriously affecting the animal fibres. [Stat. ref.] D. F. TWISS.

Direct production of filaments and threads [of rubber etc.]. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 333,005, 13.6.29. Addn. to B.P. 311,844; B., 1929, 612).—The partly set filaments are passed between even-speed rollers which may be rubber-coated and/or embossed. Tape-like filaments are thus produced. D. F. TWISS.

Manufacture of articles [*e.g.*, cables covered with gutta-percha] from thermoplastic materials. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 333,879, 16.5.29).

Polymerisation process (U.S.P. 1,746,168). **Polymerised diolefines** (B.P. 333,894 and 333,872). **2-Chlorobenzthiazoles** (B.P. 310,815). **Anti-agers** (B.P. 333,941).—See III. **Paving blocks** (B.P. 333,047).—See IX.

XV.—LEATHER; GLUE.

Sorrels: new tanning plants. Tannin content of species of *Rumex*. P. SORS (Collegium, 1930, 324–329).—Analyses of the roots of various species of Hungarian sorrels yielded the following results for tans and soluble non-tans (%) on a moisture-free basis: *Rumex maritimus* traces; *R. paluster* (*limosus*) 6.33, 4.59; *R. conglomeratus* 6.33–10.77, 8.87–19.97;

R. sanguineus 12·56, 13·56; *R. odontocarpus* (*Senophyllus biformis*) 3·16, 5·23; *R. hydrolapathum* 11·50—21·32, 7·64—18·98; *R. crispus* 3·62—6·24, 6·63—21·55; *R. patientia* 16·55—21·4, 11·37—29·30; *R. acetosella* traces; *R. acetosa* 2·78—22·66, 7·20—37·40. Some qualitative reactions of these plants are given. The plants are costly to gather, and the tans: non-tans ratio is poor, but can be improved by enrichment or by sulphiting. The liquors are very liable to fermentation owing to high content of non-tans. The high content of dyestuff of some varieties, e.g., *R. patientia*, unfits them for tanning purposes, since they render the leather harsh and brittle. Two of the varieties richest in tannin, *R. acetosa* and *patientia*, are utilised largely for other purposes and the roots form a valuable by-product.

D. WOODROFFE.

Differentiation of vegetable tannins by means of antipyrin-hydrochloric acid and the quartz lamp. W. APPELIUS and L. KRIGUELOUKIS (*Collegium*, 1930, 330—333).—25 C.c. of an infusion of a tanning material or solution of an extract containing about 10% of tannin are boiled under reflux in a flask fitted with a separating funnel, 20 c.c. of a 1% solution of antipyrin and 30 c.c. of a 10% hydrochloric acid solution are added, and boiling is continued for 20 min. The product is filtered through a hardened filter and left in contact with wool strips for 15 min.; these are then washed in cold water, drained, touched with 10% caustic soda, and while still damp examined by ultra-violet light. Characteristic colour reactions and fluorescences are obtained before and after treatment with dilute sodium hydroxide.

D. WOODROFFE.

Problem of tanning. P. D. ZACHARIAS (*Rev. gén. Colloid.*, 1930, 8, 260—264).—A résumé of the author's publications during the last 30 years, showing that they contain a generalised theory of the process of tanning based on an analogy with dyeing. E. S. HEDGES.

Evaluation of spent tan liquors by interferometry. F. ENGLISH (*Collegium*, 1930, 310—323).—It is shown that the interference produced by 1 g. of tannin or other substances in tan liquors is independent of the concentration of the tan liquors. It is proposed, therefore, to use the interferometer to evaluate spent tan liquors, for which purpose it is shown that the equivalent (interference) and percentage content of the tannin irreversibly absorbed by hide powder are important. These figures are obtained by either shake or filter-bell methods of analysis followed by a washing of the tanned hide powder. Both methods yield approximately the same figures for the irreversibly adsorbed tannin. Particulars are given for the evaluation of the interference equivalents for the tannin, adsorbed non-tans, and unadsorbed non-tars from the figures obtained on the analytical tannin solutions, but these can only be applied to spent liquors from pure materials. The factors vary with the composition of the spent liquors.

D. WOODROFFE.

PATENTS.

Production of tannin extracts and cellulose or pulp. FORESTAL LAND, TIMBER, & RAILWAYS CO., LTD., and R. O. PHILLIPS (B.P. 332,935, 25.1.29).—Suitable tannin-containing woods or barks, e.g., quebracho, chestnut, wattle bark, are extracted with a solution of a mixture of sodium sulphite (3 pts.) and sodium bicar-

bonate (1 pt.), using about 50 lb. of mixed chemicals per ton of wood. The resulting tan liquor is led off and the residual cellulose or pulp treated as usual.

D. WOODROFFE.

Moth-protection of fur etc. (B.P. 333,583—4).—See VI.

XVI.—AGRICULTURE.

Soil acidity and soil adsorption. O. LEMNERMANN and L. FRESSENIUS (*Verhandl. II Komm. Internat. Bodenkundl. Ges.*, 1929, A, 36—43; *Bied., Zentr.*, 1930, 59, 385—386).—For the preparation of soil suspensions for p_H determinations a soil:water ratio of 1:1 or 1:0·5 is recommended for sands and loams. For moorland soils a wider ratio (up to 1:25) may be used. With fresh moist samples contact for 15—20 min. is necessary; a shorter period suffices for dried soils. With the quinhydrone electrode equilibrium of potential is not instantaneous, but is accelerated by the use of considerable excess of quinhydrone. Observations of alkaline soils must be made quickly. For the determination of the buffer capacity of soils a 1:1 soil:water ratio is to be used. Daikuhara's method for lime requirement gives uncertain values for practical use. The merits of the "hydrolytic acidity" method and of titration curves for determining lime requirements are discussed. In obtaining soil-titration curves the samples of soil (10 g.) are allowed to react with acid or alkali in 100 c.c. of fluid for 3 days before p_H values are determined.

A. G. POLLARD.

Effect of artificial acidification on soil and on plant growth. E. F. VON OLDERSHAUSEN (*J. Landw.*, 1930, 78, 241—287).—Soils treated with varying proportions of hydrochloric and sulphuric acids are examined by means of pot cultures and by analysis. The development of acidity in soils is closely related to the removal of bases. Acidity, as a result of the removal of electrolytes from the soil, causes a peptisation of the particles and, as a consequence, an increase in the total surface area of the soil and a decreased permeability to water. Reduced plant growth in acid soils is caused not only by actual acidity, but by alterations in the physical properties of soils and a lack of exchangeable nutrient bases. Liming counteracts the removal of bases and improves physical conditions, but normal plant growth cannot be attained without the simultaneous use of fertilisers.

A. G. POLLARD.

Influence of adsorbed ions on soil reaction. B. AARNIO (*Bull. Agrogeol. Inst. Finland*, 1927, No. 22, 13 pp.).—When very dilute acid is added to soil the reaction becomes slightly more basic than in aqueous solution, owing to liberation of adsorbed hydroxyl ions and exchange with anions of the acid. Hence ionic exchange appears to occur among anions.

CHEMICAL ABSTRACTS.

Comparative rate of percolation of water in different soils. G. J. BOUYOUKOS (*J. Amer. Soc. Agron.*, 1930, 22, 438—445).—The method of measurement consists in allowing a soil to slake in excess of water into its ultimate natural structure, stirring gently, and then applying suction and measuring the rate of flow of the water.

CHEMICAL ABSTRACTS.

Carbon dioxide production in soils. T. OEHLER (*Z. Pflanz. Düng.*, 1930, 9B, 415—421).—Artificial

aeration of soil in pot cultures increased crop yields and also the production of carbon dioxide by soil organisms.

A. G. POLLARD.

Potash content of Palatinate soils and their appropriate fertiliser treatment. M. KLING and O. ENGELS (Z. Pflanz. Düng., 1930, 9B, 409—415).—Results of the examination of soils by the seedling method are discussed and recommendations given for suitable potash fertilisation of a number of crops.

A. G. POLLARD.

Utilisation of increasing dressings of potash by different varieties of barley. H. LIESEGANG (Z. Pflanz. Düng., 1930, 9B, 397—409).—The increase in the grain : straw ratio, the weight per 1000 grains, and the proportion of potash in the grain, following potash fertilisation, is compared for five varieties of barley.

A. G. POLLARD.

Action and utilisation of various water-insoluble phosphates in different soils, especially regarding their influence on the phosphate content of plants. M. CLEMM (J. Landw., 1930, 78, 289—308).—Comparison is made of the efficiencies of Rhenania phosphate and basic slag. In Neubauer tests the ammonium citrate-soluble phosphate of Rhenania phosphate was more effective on clay soils than the citric acid-soluble phosphate of slag. Both fertilisers were highly efficient on light sandy soils of weakly acid reaction. In loams of considerable alkalinity seedlings did not take up phosphate from either fertiliser, and in some cases there was an apparent transition of phosphorus from plant to soil. In pot cultures Rhenania phosphate (citrate-soluble) was superior to basic slag (citric acid-soluble). In all cases the increased phosphate assimilation by barley, following fertilisation, was definitely marked by the increased phosphate content of the grain. Changes in the phosphate content of the straw were less definite and were absent where alkaline soils rich in phosphate were used. Ammonium citrate-soluble phosphate affects more particularly the phosphate content of the grain, whereas citric-soluble phosphates chiefly influence that of the straw.

A. G. POLLARD.

Liming as a factor in the mobilisation of phosphoric acid in podsols. D. ASKINASI and S. JARUSSOV (Trans. Inst. Fertilisers, 1928, 57, 5; Bied. Zentr., 1930, 59, 393).—Field and pot-culture observations show that the liming of podsols accelerates the decomposition and mineralisation of humus. The latter process is associated with an increased proportion of nitrogen and phosphate in the soil solution and a corresponding increase in the crop yield and in its nitrogen and phosphate contents.

A. G. POLLARD.

Quantitative analysis of phosphoric acid. VI. Determination of phosphoric acid in a phosphate fertiliser. M. ISHIBASHI (Mem. Coll. Sci. Kyoto, 1930, 13A, 291—301; cf. A., 1929, 529, 783).—The author's improved volumetric phosphomolybdate method is especially suitable for the determination of small quantities of phosphoric acid. For the extraction of free phosphoric acid acetone is more satisfactory than either alcohol or ether.

C. W. GIBBY.

Effect of soil type and fertiliser on the nitrate content of the expressed sap and the total nitrogen content of the tissue of the small grains. R. L.

COOK (J. Amer. Soc. Agron., 1930, 22, 393—407).—Nitrogenous fertilisers increased, whilst other fertilisers diminished, the nitrate content of the sap, which varied directly with the tissue-nitrogen. Spring grains possessed greater ability to accumulate sap-nitrate than did winter grains. The tissue-nitrogen diminished steadily during the growing season.

CHEMICAL ABSTRACTS.

Effect of sodium nitrate on the composition of the expressed sap of the small grains. M. M. MCCOOL and M. D. WELDON (J. Amer. Soc. Agron., 1930, 22, 434—437).—The phosphorus content of the sap of barley, oats, rye, and wheat increased with the age of the crop, whilst the potassium content tended to decrease. Application of sodium nitrate diminished the sap-calcium, and large applications diminished the sap-potassium also. The sap-calcium at first diminished with increasing age of the crop, increasing near maturity. The magnesium content fluctuated less than that of other elements on application of sodium nitrate.

CHEMICAL ABSTRACTS.

Artificial manures in forest management. KUHNERT (Z. Pflanz. Düng., 1930, 9B, 385—392).—Results of fertiliser trials with several species of trees show that nitrogenous fertilisers, used in appropriate complete fertiliser combinations, increase both the height and diameter of the trees.

A. G. POLLARD.

Preparation of stable arsenical insecticidal suspensions slightly soluble in water. A. N. KRESTOVNIKOV and G. F. LYUTRINGSIAUZEN (Min. Svir. Tzvet. Met., 1929, 4, 431—437).—Suspensions of the compounds $3\text{Cu}(\text{AsO}_2)_2$, $\text{Cu}(\text{OAc})_2$, and $\text{Cu}(\text{AsO}_2)_2$ are prepared by passing through a colloid mill 1—5 g. of the suspension in 1 litre of water and 1% of starch or 10% of kaolin.

CHEMICAL ABSTRACTS.

Control of certain fruit diseases with "flotation sulphurs." M. A. SMITH (Phytopath., 1930, 20, 535—553).—Trials with flotation sulphur (from gas-purifying process) on a number of fungus diseases of fruit are recorded. Particle size of flotation sulphur varied from 1 μ to 5 μ in diam. as against 6—200 μ for ground sulphur and 20—130 μ for sulphur flowers. When washed free from sodium thiosulphate and thiocyanate, flotation sulphur produced no foliage injury when applied in higher concentrations than is usual in practice. Very satisfactory results with apple scab, brown rot, and cherry-leaf spot are recorded, and when combined with oil emulsion flotation sulphur satisfactorily controlled peach leaf-curl.

A. G. POLLARD.

Fruit-fly control. L. B. RIPLEY and G. A. HEPBURN (Farming in S. Africa, 1929, 4, 345—346, 357).—The strongest attractant is fermenting wheat pollard. With pure compounds as attractants, an optimal concentration exists; a 0.1% solution of linalyl acetate gives promising results. The strongest repellent is peppermint oil.

CHEMICAL ABSTRACTS.

Soil as source of infection of honey. LOCHHEAD and FARRELL.—See XIX.

PATENTS.

Manufacture of (A) a compound of urea and calcium nitrate, (B) solid urea or fertilisers containing urea. J. Y. JOHNSON. From I. G. FARBERNIND. A.-G. (B.P. 332,945 and 332,948, 29.4.29).—(A) Calcium

nitrate (1 mol. to 2–6 mols. of urea) is added to urea solutions; the compound is more stable than urea alone. (B) To avoid the increased temperatures necessary to remove the last 2–3% of water from molten urea, about 10% of diammonium hydrogen phosphate, phosphoric acid, formamide, or other substance which lowers the m.p., is added to the urea solution; the solution is then evaporated down to 0–2% of water, the pressure being adjusted to maintain the b.p. above the mixed m.p. The melt is then granulated by spraying or running into mineral oil etc. C. HOLLINS.

Sodium nitrate (B.P. 315,262). **Superphosphate** (B.P. 333,518).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Polyfructoses and their determination. I. **Determination of lævulosin for the detection of artificial invert sugar in honey.** C. I. KRUISNEER (Rec. trav. chim., 1930, 49, 841–849).—The presence of artificial invert sugar in honey is shown by the determination of the amount of lævulosin (Wohl, A., 1890, 1085) present. This is formed when a concentrated solution of lævulose is heated with a small amount of acid. The method described is as follows. Honey (10 g.) is diluted with water (75 c.c.), boiled, cooled, and the solution fermented during 48 hrs. with yeast (5 g., + 10 c.c. of sterile water). Portions of the resulting solution, after neutralisation and removal of alcohol by evaporation, are inverted by heating with hydrochloric acid for 10 min. and 3 hrs., respectively, and their lævulose contents determined by the hypiodite method. The difference (if any) in the values indicates the lævulosin content. Lævulosin, unlike other lævulose derivatives, is incompletely hydrolysed during the short treatment. H. BURTON.

Physical chemistry of starch and of bread-baking. I. Alteration of X-ray spectrum of starch in the baking and the ageing ["staling"] of bread. II. The modification of starch with V-spectrum (gelatinisation spectrum) is the equilibrium form at a high, and that with B-spectrum (retrogradation spectrum) at a low, temperature. III. First and second stages of gelatinisation. J. R. KATZ (Z. physical. Chem., 1930, 150, 37–59, 60–66, 67–80).—I. [With L. M. RIJNTSMA, T. B. VAN ITALLIE, and (Frl.) L. HEYNA.] X-Ray analysis of wheat or rye starch, of dough, and of bread in various conditions gives the following results: (a) The original flour, whether of wheat or rye, gives a characteristic interference pattern (A-spectrum) which is maintained unaltered in the "risen" dough; (b) in the crumb of freshly-baked bread the starch gives a different spectrum (gelatinisation or V-spectrum); (c) in the crumb of stale bread a third spectrum (retrogradation or B-spectrum) is obtained, closely resembling that of the original flour, but not identical with it; (d) freshly-baked crust, in so far as it is formed by rapid dehydration of the original dough, shows practically only the A-spectrum. It is inferred that the baking operation causes a profound change in the starch, probably of a chemical nature, provided sufficient water is present, as in the crumb. Staleness is the result of a reversion of the gelatinised starch to a substance very similar to the original.

Staleness is not dependent on loss of water, and does not ensue, at least for weeks, if the baked bread has been dried with 96% alcohol.

II. [With L. M. RIJNTSMA.] A true physico-chemical equilibrium subsists between the modifications of starch (called, respectively, β_n - and α -starch) which give rise to the B- and V-spectra. The former is stable below, and the latter above, 60°. The results of X-ray analysis are in agreement with those of earlier experiments on the swelling capacity of untreated, of gelatinised, and of retrograded starch.

III. [With L. M. RIJNTSMA and T. B. VAN ITALLIE.] The gelatinisation of starch takes place in two distinct stages. The first (corresponding with the condition in newly-baked bread) is completed when starch is heated with a limited quantity (50–100%) of water at 100°, or with excess of water at 62.5°; the second requires the heating of starch with excess of water at 90–100°. Although the product of the second stage (starch paste) shows a higher swelling capacity and a higher proportion of soluble amyloses, it gives, after dehydration with alcohol, the X-ray spectrum characteristic of α -starch. It also undergoes retrogradation to β_n -starch when kept at the ordinary temperature, although more slowly than does the product of the first stage. The results are discussed in relation to the microscopical appearance of gelatinised starch and the chemical reactions of starch paste. F. L. USHER.

Fermentation of honey. LOCHHEAD and FARRELL.—See XIX.

PATENTS.

Synthetic gummy materials (U.S.P. 1,746,615 and 1,746,665).—See XIII. **Methyl α -hydroxyethyl ketone and diacetyl** (B.P. 315,264).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Preservation of sweet white wines. L. MOREAU and E. VINET (Ann. Falsif., 1930, 23, 408–412).—Wines cannot with certainty be kept free from further fermentation by increasing up to the toxic limit the waste products of the life of the yeast. This was, however, accomplished by successive heatings to a temperature above the thermal death point of the yeast and subsequent re-infection after each heating. It is necessary to treat these wines with sulphurous acid to prevent recurrence of fermentation entirely, and, owing to combination, large doses of sulphurous acid are required to produce the requisite concentration of the free acid. B. W. TOWN.

Determination of ferrous iron in white wines. P. MALVEZIN (Ann. Falsif., 1930, 23, 412–414).—The wine is treated with potassium ferrieyanide and the tint compared with that given with a standard solution of ferrous iron. B. W. TOWN.

Fermentation products from cornstalks. C. S. BORUFF [with A. M. BUSWELL] (Ind. Eng. Chem., 1930, 22, 931–933; cf. B., 1930, 117).—The anaerobic fermentation of cornstalks in the presence of overflow liquor from a sewage disposal plant, which served as an inoculum and as a suitable source of nitrogen for the bacteria, was studied by determining the composition of the residue and the proportions of the products (carbon dioxide, hydrogen, methane, and volatile acids)

formed. It was shown that the gases and acids formed represent 118% of the organic matter decomposed, which is higher than the theoretical yield from either cellulose or pentosans, indicating a partial decomposition of the lignin.

F. R. ENNOS.

Fermentation of honey. LOCHHEAD and FARRELL.—See XIX.

PATENTS.

Preparation of acetylmethylcarbinol [methyl α -hydroxyethyl ketone] and diacetyl [by fermentation]. T. H. VERHAEGE, SEN. (B.P. 315,264, 25.6.29. Holl., 10.7.28).—A yield of 35% of the carbinol is obtained by intensively aerating a fermenting mash containing a carbohydrate (beet molasses, maize flour), a source of nitrogen, a phosphate, a carbonate (chalk), and a bacterial culture normally producing β -butylene glycol (*Aerobacter aerogenes*, *Clostridium polymyxa*, etc.). The aeration (e.g., 10–25 cub. m. per hr. per hectolitre) may be commenced shortly after fermentation has become evident, and may be progressively increased.

C. HOLLINS.

Active substances from hypophysis glands (B.P. 333,155).—See XX.

XIX.—FOODS.

Oxidation-reduction in milk. Methylene-blue reduction test. H. R. THORNTON and E. G. HASTINGS (J. Dairy Sci., 1930, 13, 221–245).—The chief variations in the results of the test, which depends on the removal of dissolved oxygen by bacteria followed by the reduction of the dye by the milk constituents, are due to the different rates of uptake of oxygen by different bacterial species, and the removal of bacteria in the cream. Shaking decreases the time of reduction.

CHEMICAL ABSTRACTS.

Variations in the density of the whey with the ageing of milk. F. BERNARDINI and E. A. GAUTHIER (Annali Chim. Appl., 1930, 20, 236–238).—When milk is kept either at 3° or at the ordinary temperature, the percentage of fat remains unchanged after a month. At 3° the density of the serum remains virtually constant for 5 days, falls somewhat during the succeeding 5 days, and then keeps constant again; the total fall amounts to about 0.001. At the ordinary temperature the loss in density is more rapid and more considerable, owing to attack of the proteins and sugars, not by lactic organisms, but by moulds.

T. H. POPE.

Milk of the silver fox. O. LANA Ann. Falsif., 1930, 23, 404).—A complete analysis is given.

B. W. TOWN.

Processed cheese. H. L. TEMPLETON and H. H. SOMMER (J. Dairy Sci., 1930, 13, 203–220).—Sodium citrate gave a firmer body to cheese than did sodium phosphate. The tin foil is darkened by over 2% of phosphate. Cheese body depends largely on the water content, but varies also with the pH ; an apparatus for determining cheese body is described. The water-soluble nitrogen in cheese is increased by processing.

CHEMICAL ABSTRACTS.

Fat content of Gruyère cheese. P. SAJOUS (Ann. Falsif., 1930, 23, 396–403).—It is suggested that the manufacture of Gruyère cheese should be standardised and classified as (a) "tout-gras" or "extra

gras" (45% of fat); (b) "gras" (40%); and (c) a quality containing below 40% of fat, calculated on dry material. The fat content of a cheese is roughly fourteen times that of the milk from which it is prepared.

B. W. TOWN.

Effect of processing ice-cream mixtures at different pressures when the milk solids-not-fat content is varied. W. H. E. REED and E. R. GARRISON (Missouri Agric. Exp. Sta. Res. Bull., 1929, No. 128, 41 pp.).—The temperature to which an ice-cream mixture can be frozen is determined by the composition and the pressure; the temperature is lowered by an increase in the non-fatty solids. Mixtures with a high content of non-fatty solids require a lower processing pressure than those of lower content. CHEMICAL ABSTRACTS.

Vitamin-G [vitamin- B_2] in certain meats and meat by-products. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1930, 41, 205–220).—Feeding experiments with young albino rats have been carried out to determine the relative amounts of vitamin- B_2 in certain meats, compared on the air-dry, fat-free basis. From 15 to 25% of the ration, dried beef, pork, or lamb, furnished sufficient vitamin- B_2 for excellent growth. Beef spleen contained about the same amount as beef itself, whilst beef and pork liver and beef kidney contained 5–8 times as much vitamin as did beef, pork, or lamb; 3% of the ration of beef liver, 3.05% of pork liver, and 2.77% of beef kidney furnished an ample supply of the vitamin.

E. HOLMES.

Soil as a source of infection of honey by sugar-tolerant yeasts. A. G. LOCHHEAD and L. FARRELL (Canad. J. Res., 1930, 3, 51–64).—Sugar-tolerant yeasts, capable of fermenting honey, are regularly found only in apiary soils; they are not members of the normal field-soil microflora. These yeasts, which remain viable during the winter in frozen soil, reduce to seven types, five being classified as *Zygosaccharomyces* and the others as species of *Torula* and *Mycotorula*, respectively.

T. H. MORTON.

Effect of preservatives on fermentation by sugar-tolerant yeasts from honey. A. G. LOCHHEAD and L. FARRELL (Canad. J. Res., 1930, 3, 95–103).—With the object of finding a suitable preservative for honey, the rates of fermentation have been measured of an 80% honey solution inoculated with a mixed culture of yeasts in presence of various antiseptics. Sodium hypochlorite and chloramine-T did not prevent fermentation in a concentration of 100 pts. of available chlorine per million. Fermentation was inhibited by the following substances in the concentrations indicated: hydrogen peroxide 0.5%, hexylresorcinol 0.01%, sodium salicylate 0.06%, sodium borate 0.1%, sodium sulphite 0.01%, sodium hydrogen sulphite 0.025%. Considerations such as taste and legal limits indicate that the last three substances are suitable preservatives.

R. K. CALLOW.

Does sugar inversion affect pectin jelly formation? G. M. COLE, R. E. COX, and G. H. JOSEPH (Food Ind., 1930, 2, 219–221).—Invert sugar and dextrose give satisfactory jellies with the correct amounts of pectin and acid. Sucrose in jellies continues to invert during storage and does not affect the quality of the jelly. Jellies can be made when glycerol, ethyl alcohol,

or glacial acetic acid are used instead of sucrose. Premature gelation is discussed. CHEMICAL ABSTRACTS.

Detection of artificial invert sugar in honey. KRUISHEER. **Starch and bread-baking.** KATZ.—See XVII.

PATENTS.

Treatment of wheat germ for use as an edible food substance. K. P. WANKLYN and F. STACEY (B.P. 332,847, 31.10.29).—Wheat germ will keep indefinitely if dried at 57° under reduced pressure for 8 hrs. and milled. E. B. HUGHES.

Food product. E. B. BROWN, C. N. FREY, and H. M. HARKINS, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,755,864, 22.4.30. Appl., 2.9.27).—A bread improver is prepared by heating under pressure a mixture of starch and yeast. After conversion of the starch into sugar is complete the product is neutralised and then reacidified with lactic acid. E. B. HUGHES.

Treatment of milk. H. E. MORTLAND, Assr. to PEVELY DAIRY Co. (U.S.P. 1,757,646, 6.5.30. Appl., 6.2.28).—To skimmed milk (1000 pts.) at 62.8° is added gradually a cold mixture of cane sugar (35 pts.), lime (7 pts.), and water (50 pts.), and the product after being condensed in the vacuum pan is suitable for use as a filler in ice-cream etc. E. B. HUGHES.

Meat-curing method. R. E. JORDAN, Assr. to ALBRIGHT-NELL Co. (U.S.P. 1,755,992, 22.4.30. Appl., 13.5.27).—The meats or meat products are circulated on a conveyor in a vertical smoke-house. The method is claimed to avoid the formation of an impervious crust of coagulated albumins, accelerate penetration of curing gases, and minimise shrinkage. E. B. HUGHES.

Sterilisation of food. O. H. HANSEN, Assr. to HANSEN CANNING MACHINERY CORP. (U.S.P. 1,756,549 and 1,756,550, 29.4.30. Appl., 15.6.29).—(A) The material, e.g., peas, is sterilised by heating in a relatively dry condition, partly cooled, and after addition of cool sterilised brine the product is canned. (B) Soft fruits, requiring surface sterilisation only, before being canned are immersed in a relatively small quantity of the liquid at a suitable temperature. E. B. HUGHES.

Manufacture of [concentrated] vegetable foods. E. H. MILES and G. REILLY (U.S.P. 1,775,966—7, 16.9.30. Appl., 8.7.26. U.K., 15.7.25).—See B.P. 256,765 and 274,051; B., 1928, 425.

Disintegration of cellular matter (U.S.P. 1,746,731).—See I. **Fat from carcasses etc.** (B.P. 310,542). **Edible cottonseed oil** (U.S.P. 1,747,675).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Castor oil soaps in spirituous [medicinal] preparations. W. MEYER (Arch. Pharm., 1930, 268, 358—371).—The author has examined the possibility of detecting adulteration of pharmaceutical preparations, especially those containing olive and linseed oil soaps, by the substitution for pure alcohol of a product to which castor oil (1%) and sodium or potassium hydroxide (0.4%) had been added. Indications of the presence of ricinoleic acid in the mixtures of fatty acids isolated from such products are afforded by (a) determination of constants, especially the acetyl value, (b) incomplete

miscibility with petroleum fractions (15%), (c) formation of sec.-octyl alcohol when the sodium salts are fused with potassium hydroxide (5%), and (d) appearance of a bluish-green colour when the mixture is treated with antimony trichloride and then with chloral hydrate (8%), the numbers given in parenthesis being the smallest percentages of ricinoleic acid which can be identified. The reactions enabled adulterations to be detected in eleven official preparations, but failed with three others. A comprehensive scheme for the detection of denaturants in spirituous products is given.

H. E. F. NOTTON.

Liquor plumbi subacetatis fortis: its preparation and assay. P. A. BERRY (Chem. Eng. Min. Rev., 1930, 22, 421—423).—The use of insufficient litharge in the preparation of this liquor results in a solution low in basicity, whilst an excess, although increasing the basicity, results in the crystallisation of a basic acetate, $\text{Pb}(\text{OAc})_2 \cdot 2\text{PbO} \cdot 4\text{H}_2\text{O}$, with an attendant reduced lead and acetate content. The white deposit normally formed when making this solution is composed of this basic acetate together with basic lead carbonate. The following method for the preparation is suggested: 320 g. of litharge are triturated with 100 c.c. of water and the paste is transferred to a bottle with a further 650 c.c. of water. After shaking, 240 g. of acetic acid are added and the mixture is shaken for 30 min. and then occasionally for 30 min. After setting aside for 24 hrs. the mixture is filtered and the filtrate made up to 1 litre with water. In a proposed method of assay, the lead is determined by the addition of a known excess of standard oxalic acid solution followed by its determination in the filtrate with 0.1N-potassium permanganate, and the acetic acid is calculated from the result of a determination of the total acidity of the filtrate, the oxalic acid content being known from the first titration. This method is equally suited for lead acetate.

E. H. SHARPLES.

Detection of arsenic in medicinal substances and comparison of the sensitiveness of Bettendorff's and Bougault's reagents. A. GRIPPA (Annali Chim. Appl., 1930, 20, 249—257).—Examination of a large number of substances shows that, whereas Bougault's reagent (20 g. of sodium hypophosphite, 40 c.c. of water, and 180 c.c. of concentrated hydrochloric acid) is capable of detecting 0.01 mg. of arsenic per c.c. or g. of material, Bettendorff's reagent is unable to detect less than 0.02 mg. of arsenic. Contrary to statements made, the addition of potassium iodide to Bougault's reagent does not increase the sensitivity and is useful only with ferric compounds to reduce these to the ferrous state.

T. H. POPE.

Assay of ipomea. L. E. WARREN (J. Assoc. Off. Agric. Chem., 1930, 13, 377—383).—Comparison is made of the United States and French official methods with that of Jenkins (B., 1914, 885) for the analysis of ipomea (the dried root of *Ipomea orizabensis*, Ledenois). The French method gives lower values than the others. The modified procedure recommended is as follows: 10 g. of the sample are boiled under reflux for 30 min. with 50 c.c. of alcohol and the mixture is then percolated with warm alcohol to obtain about 95 c.c. of percolate (or more if the last runnings give more than a faint cloudiness when poured into cold water). The volume of

the percolate is adjusted to 100 c.c. Of this 25 c.c. are evaporated on a water-bath to remove alcohol, the residue being washed with water (with kneading) and finally dissolved in alcohol together with portions filtered from the wash-water. The solution is evaporated, dried at 100°, and weighed. A. G. POLLARD.

Bio-assay of capsicum, U.S.P. X. J. C. MUNCH (J. Assoc. Off. Agric. Chem., 1930, 13, 383—385).—Comparison is made of the response of the individual to piperine, with the sensation of pungency in the throat produced by swallowing standard proportions of extract. A. G. POLLARD.

Influence of the pulping of fresh gentian root on its content of glucides. C. BÉGUIN (J. Pharm. Chim., 1930, 12, [viii], 213—219).—During the pulping of fresh gentian root, hydrolysis of the osides is accelerated by the soluble ferments: 21.3% of the holosides hydrolysable by invertin, 19.7% of those hydrolysable by emulsin, and 48.2% of the gentiopicroside are hydrolysed. On keeping the pulp, hydrolysis proceeds regularly, except that due to the emulsin, which seems to be most rapid during the first hour. In 2 hrs. all the gentiopicroside is destroyed, no reaction for gentiogenol is obtained, and there remains only a small amount of product hydrolysable by emulsin. E. H. SHARPLES.

Preservation of gentian root by alcohol vapour. C. BÉGUIN (J. Pharm. Chim., 1930, 12, [viii], 49—63).—Roots sterilised by means of alcohol vapour give on subsequent treatment tinctures similar to, but somewhat weaker than, those obtained directly from fresh roots. The vapour treatment extracts some proportion of the active principles, 6—8% of the content when the roots are whole, and 20—30% when they are cut. S. I. LEVY.

Peppermint oil of the Saratov district. O. V. SOBOLEVSKAYA (Trans. Sci. Chem.-Pharm. Inst., Moscow, 1928, No. 19, 186—194).—The oil of plants screened or unscreened from sunlight contained 47.7—53.05, 51.38—55.38% of menthol and 11.76, 2.96—5.06% of menthone, respectively. In a wet climate the yield of oil is higher, but the quality inferior.

CHEMICAL ABSTRACTS.

Rumanian peppermint oil and the position of the peppermint oil industry in Rumania. E. KOPP (Pharm. Zentr., 1930, 71, 577—582).—The yearly production and the quality of the oils from peppermint plants imported from England and Italy and grown in Rumania since 1926 and the economics of the industry are described. The commercial oils produced at present, in a yield of 30 kg. per hectare, have: yields from green plant 0.20—0.24%, d_{20}^{15} 0.9042—0.9113, α_D $-21^\circ 10'$ to $-27^\circ 9'$, n_D^{20} 1.4602—1.4618, solubility in 70% alcohol 1 in 2.7—3.0 pts., acid value nil, ester value 25.2—44.8, acetyl value 179.2—200.7, ester menthol 3.3—12.5%, free menthol 46.7—50.3%.

E. H. SHARPLES.

Soaps containing salicylic acid. SCHLENKER.—See XII.

PATENTS.

Manufacture of therapeutic agents [against carcinoma, spirochaetes, and trypanosomes]. I. G. FARBERIND. A.-G., Asses. of C. F. BOEHRINGER & SÖHNE, G.M.B.H. (B.P. 318,556, 5.9.29. Ger., 5.9.28).—

The calcium or other alkaline-earth salts of 3- ω -sulphomethylamino-3'-amino- and 3 : 3'-di- ω -sulphomethylamino-4 : 4'-dihydroxyarsenobenzenes are suitable agents. Other known salts [cf. B.P. 3615 of 1912 (B., 1912, 844) and B.P. 214,237 (B., 1925, 691)] are ineffective against carcinoma. C. HOLLINS.

Manufacture of aralkyl esters of the *Hydnocarpus* fatty acids. I. G. FARBERIND. A.-G. (B.P. 311,236, 6.5.29. Ger., 7.5.28).—The fatty acids from varieties of *Hydnocarpus*, obtained by saponification, are converted by usual methods into aralkyl esters, which are stable, odourless, and pleasant-tasting oils valuable against leprosy etc. The benzyl esters, b.p. 180—230°/1 mm., of the acids of chaulmoogra oil, and the phenylethyl esters, b.p. 190—240°/1 mm., of the acids of *H. wightiana*, are described. C. HOLLINS.

Rendering irradiated ergosterol stable. A. HOME-MORTON. From F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 334,002, 1.7.29).—Deterioration of irradiated ergosterol, as measured by the decrease in optical activity, is prevented by the addition of small amounts of phenols to the ergosterol or ergosterol solution before or after irradiation. E. H. SHARPLES.

Extracts of animal or vegetable organs or parts. SOC. CHEM. IND. in BASLE, W. MERKI, and P. SCHEIDEGGER (B.P. 333,159, 2.4.29. Addn. to B.P. 285,856; B., 1929, 150).—Animal organs generally (other than sex organs) or vegetable parts are cooled in solid carbon dioxide to -70° and pulverised before extraction with solvents. C. HOLLINS.

Preparation of active substances from hypophysis glands. I. G. FARBERIND. A.-G. (B.P. 305,475 and Addn. B.P. 333,155, [A] 4.2.29, Ger., 4.2.28, [B] 5.2.29).—(A) The whole gland, without separation of posterior and anterior lobes, is treated below 70° with acid (hydrochloric acid at 15° for 30 min.) or alkali (0.5N-sodium hydroxide at 35 — 40° for 20 min.), the duration of treatment being varied according to the concentration of the reagent and the temperature; the hydrolytic agent is then neutralised, albuminates and globulinates are removed by centrifuging, and a solution of active substances is obtained which may be further purified by precipitation of ballast substances with acetone, the latter being then removed in a vacuum. The albuminates and globulinates yield a further quantity of active substances on repeating the hydrolytic treatment. (B) The whole gland is hydrolysed by fermentation, e.g., with papain or by autolysis, at a determined p_H , which is adjusted, if necessary, to precipitate ballast substances during or after hydrolysis. C. HOLLINS.

Surgical pads or the like. JOHNSON & JOHNSON (GREAT BRITAIN), LTD. From JOHNSON & JOHNSON (B.P. 333,980, 7.6.29).—Substances are incorporated with the pad, which, on exposure to radiant energy, cast a distinctive shadow or image on a radiograph or screen. E. H. SHARPLES.

Manufacture of inactive menthol. K. SCHÖLLKOPF and A. SERINI, Assrs. to RHEINISCHE KAMPFER FABR. GES.M.B.H. (U.S.P. 1,776,087—8, 16.9.30. Appl., [A] 29.6.27, [B] 30.6.27. Ger., [A] 15.12.26, [B] 15.2.27).—See B.P. 285,403 and 285,394; B., 1928, 873, 691.

2-Chlorobenzthiazoles (B.P. 310,815). Hydroxy-

1' : 8'-naphthoylenenaphthiminazoles and derivatives (B.P. 316,143).—See III. Bismuth hydroxide (B.P. 332,504).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Amount of metallic silver in unexposed photographic emulsions, and formation of nuclei. H. ARENS and J. EGGERT (*Z. wiss. Phot.*, 1930, **28**, 178—190).—Repetition of Weigert and Lühr's experiments (B., 1930, 303) on the determination of the amount of metallic silver in an unexposed emulsion has yielded results suggesting that the method of analysis used is incapable of effecting a complete separation of primary silver from silver thiosulphate complex formed in development, the values obtained for the amount of the former thus being too high. The same conclusion is reached from a consideration of the amount of photo-silver obtained by extrapolating the mass of the latent image, and of the amounts of such substances as thio-sinamine and methylene-blue required to affect the sensitivity. Finally, comparison of the rate of growth of the nuclei in the physical development of an ordinary photographic emulsion with that of an emulsion of colloidal silver of approximately the same degree of dispersity indicates that the amount of primary silver is much less than Weigert and Lühr's value.

R. CUTHILL.

Gases produced by the decomposition of nitro-cellulose and cellulose acetate photographic films. J. C. OLSEN, A. S. BRUNJES, and V. J. SABETTA (*Ind. Eng. Chem.*, 1930, **22**, 860—863).—The nature and quantity of gases evolved in the decomposition by heat of cellulose films in atmospheres of nitrogen and oxygen were studied. Details of analytical methods and results are given. Three highly toxic gases (carbon monoxide, nitrous fumes, and hydrocyanic acid) may be present. The origin and variation in amount of these with experimental conditions is discussed. The safety storage conditions for these materials are indicated. S. S. WOOLF.

Effect of temperature on the photographic plate. N. BARABASHEV and B. SEMEJKIN (*Z. wiss. Phot.*, 1930, **28**, 221—228).—Density measurements on plates exposed at temperatures between 2° and —21° have shown that as the temperature of exposure falls the gradation of the plate becomes less. R. CUTHILL.

PATENTS.

Film-forming element. H. E. VAN DERHOFF, ASST. to EASTMAN KODAK CO. (U.S.P. 1,746,751, 11.2.30. Appl., 21.8.25).—For the manufacture of films from dopes containing derivatives of cellulose a cylinder comprising a supporting metallic backing covered with a polished layer of nickel and an outer adherent layer of chromium is used. The chromium is polished to the degree required for the film surface. C. A. KING.

Matting a cellulose ester film for photographic purposes. E. LOMBERG (B.P. 308,358, 21.3.29. Ger., 22.3.28).—The rear side of the film is treated with cellulose ester solution containing starch as the matting agent. J. W. GLASSETT.

Treatment of cellulose and like films with gas. CELLOPHANE SOC. ANON. (B.P. 332,631, 28.3.29. Fr., 21.2.29).—A band of gas-permeable paper or fabric

is wound up with the film in contact with the surface, thereby permitting treatment of the film surface with gas to be carried out in the rolled state, as, for example, in the development of diazotypes with ammonia gas.

J. W. GLASSETT.

Manufacture of photographic paper and films. FILM OZAPHANE (B.P. 318,511, 6.8.29. Fr., 4.9.28).—A cellulose film or a suitable support coated with a gelatin layer, which may be rendered matt by the addition of kaolin, is treated with a highly volatile solution of a light-sensitive diazo compound. The rapid volatilisation of the solvent prevents the penetration of the sensitive compound, which is thereby concentrated at the surface. A formula for a suitable sensitising solution is given. J. W. GLASSETT.

Manufacture of sensitised velvet paper for photographic uses. D. FRIEDLÄNDER, ASST. to A. MEURSING and E. J. GRATAMA (U.S.P. 1,752,665, 1.4.30. Appl., 7.7.27. Ger., 7.7.26).—A well-sized paper support is covered with an adhesive medium not readily soluble in water and is then sprinkled with a fine fibrous dust, e.g., white wool powder. After drying, the excess dust is removed with a soft brush and the paper coated with the sensitive emulsion. J. W. GLASSETT.

Transfer picture and its manufacture. E. HENTSCHEL (B.P. 331,761, 16.9.29. Ger., 18.10.28).—The support is coated with a thin layer of wax upon which the pictures are printed with a pigmented solution of a cellulose derivative, or, alternatively, the pictures are stamped out from a finished film of a cellulose ester and attached to the wax surface. A coating of an adhesive resin (dammar or mastic) is then applied. J. W. GLASSETT.

Preparation of photographic diazotypes. F. VAN DER GRINTEN (B.P. 332,552, 20.4.29. Holl., 22.2.29).—The surface of the diazotype is sized during development by the incorporation of sizing agents such as sodium stearate, water-soluble casein, cows' milk, egg albumin, flour, etc. in the solution of the coupling agent. A further sizing may be given after development by treatment with a plain aqueous solution of these agents. Suitable working formulæ for the process are given. J. W. GLASSETT.

X-Ray screens. SCHERING-KAHLEAUM A.-G. (B.P. 318,152, 19.8.29. Ger., 28.8.28).—The fluorescent layer is coated directly upon a lead-glass plate. J. W. GLASSETT.

Manufacture of [light]-sensitised element, and production of photographic images thereon. E. E. E. LEHMANN (U.S.P. 1,753,708, 8.4.30. Appl., 26.4.27. Ger., 27.4.26).—Diazotised *p*-aminodiphenylamine or a derivative is deposited upon a suitable carrier, exposed, and developed by coupling with β -naphthol solution to produce a negative, or, by treatment with an oxidising solution (dilute, acidified potassium dichromate solution) to give a positive. J. W. GLASSETT.

Colour photography. W. V. D. KELLEY (U.S.P. 1,753,379, 8.4.30. Appl., 9.11.25).—One side of a doubly-coated film is exposed behind a red-selection negative, developed in acid or neutral diaminophenol, and then iron-toned to give a blue image. After washing, clearing, and drying, the same side is exposed under the green-selection negative and the reverse side under

the blue-selection negative. These are developed as before, bleached in a copper sulphate differential-hardening bath, fixed, and then dyed with magenta and yellow dyes, respectively, which are absorbed in the hardened parts. The yellow image may be obtained alternatively by an ammonium metavanadate bath.

J. W. GLASSETT.

Means for reproducing designs in metal by aid of photography. F. W. MILLER (B.P. 331,856, 9.4.29).—A glass plate coated with a mixture of dextrin and glucose or similar substances sensitised with ammonium dichromate is exposed behind a photographic positive of the design and then dusted with a metallic powder or other conducting medium which adheres to the unexposed parts of the surface only. After applying a coating of collodion and allowing it to set, the combined metallic coating and collodion film is floated off the glass plate and transferred, collodion film underneath, to a gelatin-coated support. Leads are attached to the metal picture, the whole is immersed in a plating vat, and a convenient thickness of a suitable metal is deposited on the original metal picture. The fine metal network is then stripped and pressed into a suitable support.

J. W. GLASSETT.

Preparation of light-sensitive layers. G. KÖGEL (U.S.P. 1,776,155, 16.9.30. Appl., 4.12.28. Ger., 13.12.27).—See B.P. 302,282; B., 1930, 303.

Production of [composite] photographic prints. SOC. TECH. D'OPTIQUE ET DE PHOTOGRAPHIE (S.T.O.P.) (B.P. 334,708, 3.9.29. Fr., 6.10.28).

Cellulose compositions of low inflammability (B.P. 312,609).—See V.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Nitration of wood pulp and the like. V. PLANCHON (B.P. 333,817, 31.10.29. Fr., 18.1.29).—Sheets of wood pulp etc. are immersed a small distance apart in a nitrating bath kept at about 15° for the first 8–10 hrs. and then at 30° for a further 2 hrs.; collodion of low viscosity is produced.

H. ROYAL-DAWSON.

[Non-corrosive] priming mixture. J. D. McNUTT, ASST. to WINCHESTER REPEATING ARMS CO. (U.S.P. 1,755,330, 22.4.30. Appl., 23.3.27).—A mixture of mercury fulminate, barium nitrate, lead thiocyanate, ground glass, and a basic organic compound, such as diphenylamine, is claimed.

W. J. WRIGHT.

Explosive composition. F. OLSEN (U.S.P. 1,758,169, 13.5.30. Appl., 16.10.25).—A bursting charge for projectiles, which melts below 100°, consists of trinitrotoluene mixed with guanidine nitrate or picrate. The mixture detonates readily, but is sufficiently insensitive to shock to permit of its use in armour-piercing shells.

W. J. WRIGHT.

Combustible [compositions for tracer projectiles]. S. ST. P. MEEK (U.S.P. 1,756,255, 29.4.30. Appl., 8.9.25).—The use of mercury compounds, either as oxygen carriers, *e.g.*, mercurous nitrate and mercuric oxide, or as fuel, to decrease the rate of burning and increase the brilliance, *e.g.*, mercuric thiocyanate, or as inert substances, to increase brilliance, *e.g.*, mercurous chloride, is claimed.

W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of water by electro-osmosis. E. BARTOW and R. H. JEBENS (Ind. Eng. Chem., 1930, 22, 1020–1022).—The purification of water by the removal of salts from a central section of a cell through diaphragms to anode and cathode cells has been developed in Germany. An apparatus for this purpose consists of a battery of 10 cells. The water in course of purification passes through the central compartment of each cell in turn, and the anode and cathode waters are removed by wash-water. The average voltage across the first four cells may be 27.5, increasing towards the end of the battery to 110 volts as the purification proceeds. Treatment of zeolite-softened water and regular tap water produced water of the same quality. Electrolytes were reduced from about 700 to 2.3 pts. per million, and the ash (chiefly silica) to 9 p.p.m. The cathode water contains hydroxide and is essentially lime water, and the anode water could be used as a bleaching agent. Water purified in this manner may substitute distilled water for most purposes, as the small residual content is chemically inert. The cost of production is said to be much lower than that of distilled water.

C. A. KING.

Removal of phenolic tastes in water supplies. R. D. SCOTT (Ohio Conf. Water Purif., IX Rep., 1929, 59–60).—The taste of chlorophenol was removed by addition of 3–5.7 or 1.8–3.3 pts. in 10⁶ of chlorine for 0.1 or 0.01 pt. of phenol in 10⁶, respectively. Taste probably due to vegetation etc. was not removed by 10 pts. of chlorine in 10⁶. CHEMICAL ABSTRACTS.

Control of phenol tastes [in water] by means of increased lime treatment. J. S. SCOTT (Ohio Conf. Water Purif., IX Rep., 1929, 61–63).—Excess of lime removes phenolic tastes from chlorinated water.

CHEMICAL ABSTRACTS.

Ammonia-chlorine process [for water] as a means for taste prevention and effective sterilisation. M. M. BRAIDECH (Ohio Conf. Water Purif., IX Rep., 1929, 67–83).—Pre-ammoniation prevents rather than destroys phenolic tastes and odours, chloroamine being produced.

CHEMICAL ABSTRACTS.

Maintaining chemical balance to resist corrosion, and its application to a recent development in water softening with lime. C. P. HOOVER (Ohio Conf. Water Purif., IX Rep., 1929, 96–99).—An indicator test, and a process whereby the carbonate hardness is reduced by means of lime approximately to the solubility limit of the calcium carbonate and magnesium hydroxide, are described.

CHEMICAL ABSTRACTS.

Fermentation of cornstalks. BORUFF and BUSWELL.—See XVII.

PATENTS.

Water softener. S. S. SADTLER (U.S.P. 1,761,411, 3.6.30. Appl., 10.1.27).—A small amount of an indicator, *e.g.*, phenolphthalein or a chrome-blue, is added to the softening reagent, *e.g.*, sodium bicarbonate, to indicate when the desired degree of alkalinity is reached for domestic or laundry purposes.

C. JEPSON.

De-aeration and evaporation of liquid (U.S.P. 1,758,566).—See I. **2-Chlorobenzthiazoles** (B.P. 310,815).—See III. **Base-exchange material** (U.S.P. 1,756,623–5). **Glauconite for water softening** (U.S.P. 1,757,373).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 21 and 28, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Scientific thermal principles of industrial furnaces. G. ZOROS (Rev. Mét., 1930, 27, 352—361).—An exposition of the principles of heat production and exchange in relation to the construction of furnaces.

A. R. POWELL.

Effect of surface conditions on heat transmission. S. J. KOHLI (Inst. Chem. Eng., Oct., 1930. Advance copy. 19 pp.).—The overall and film coefficients of heat transfer through a copper tube within which a current of hot air passed, and which was cooled with water outside, were studied with especial reference to variations in the character of the surface. The water flow was maintained constant, and the gas flow varied as required, being kept uniform in each case until all temperatures were constant. Temperatures were measured by thermocouples and porcelain stuffing-boxes were used to minimise conduction. Using a polished copper tube, results were obtained for the over-all coefficient in fair agreement with Joule and Ser's formula, $k = 16\sqrt{v}$ to $19\sqrt{v}$, where v is the gas velocity in metres/sec. The author's results are summarised by the equation k (over-all) $= 16.912v^{0.625}$ and k (air-copper) $= 16.116(w/a)^{0.625}$, where w/a is the mass-velocity. Other formulæ are discussed and criticised. Tests with a silvered copper surface showed lower coefficients at high velocities, but higher coefficients at velocities below 2.2 kg./m.²/sec. Below the critical velocity the heat transfer falls rapidly. Generally, similar results are reported for a surface covered with carbon, dulled with a sand-blast, or having a screw thread. Coatings with red, green, and violet enamels increased the heat transfer throughout. It is suggested that radiations of short wave-lengths may be absorbed and converted into heat energy. At high velocities convection plays an overwhelming part, and therefore the thickness of the air film is important. At lower velocities radiation and absorption are of more importance. The metal-water film coefficients calculated in a few cases show an increase with the logarithmic mean temperature difference, due no doubt to increased convection. These coefficients are markedly increased by black surfaces on the gas side.

C. IRWIN.

Trends in heat transfer. D. F. OTHMER (Ind. Eng. Chem., 1930, 22, 988—993).—Reference is made to a mercury boiler which has been in operation for several years at a U.S.A. power station, and to the use of diphenyl vapour in the distillation of lubricating oils. The Swenson forced-circulation evaporator has had its greatest success in the concentration of caustic soda. It operates by minimising the effect of the stagnant

surface film. Other recent forms of evaporator include the "vapour recompression system," in which the vapours from a single effect are recompressed to the pressure of the heating steam by a turbo-blower, and the "multiple-effect flashing system" in which the liquid is heated under pressure in a series of heaters, the last of which is supplied with boiler steam. The liquid is evaporated by flashing into chambers at progressively lower pressures, and heats the incoming liquid. The Ruths steam accumulator and the Merkel process, obtaining the same effect by passing low-pressure steam into caustic soda solution, are mentioned, together with several types of heat interchanges. C. IRWIN.

Regenerative chambers. II. Determination of the heat exchange and pressure losses with various grate arrangements. H. KISTNER (Arch.-Eisenhüttenw., 1929—1930, 3, 751—768; Stahl u. Eisen, 1930, 50, 1027—1028).—Mathematical expressions are derived for calculating the heat lost by radiation and the reduction in gas pressure in the heat-exchange chambers of various types of regenerators.

A. R. POWELL.

Pan grinding. J. W. MELLOR (Trans. Ceram. Soc., 1930, 29, 271—279).—Mathematical. The relationship between the best speed of revolution and the diameter of the runner of the pan (for grinding colours, flint, etc.) has been worked out theoretically.

R. J. CARTLIDGE.

Relation between mass transfer (absorption) and fluid friction [in gas mixtures]. A. P. COLBURN (Ind. Eng. Chem., 1930, 22, 967—970).—The absorption of soluble components from gas mixtures and the condensation of vapours are analogous to the processes of heat or momentum transference. They depend on the rate of diffusion of gas molecules through a film on the liquid surface and on eddy currents in the turbulent portion of the gas stream. The mass-transfer coefficient $k = FM_v/u_m p_{gf} M_m \phi$, where F is the frictional resistance of fluid per unit surface area, M_v the mol. wt. of diffusing material, u_m the mean velocity in the turbulent portion, p_{gf} the logarithmic mean partial pressure of the noncondensing gas in the film, M_m the mean mol. wt. of gas mixture, and ϕ is a function of gas velocities, viscosities, and densities which approximates to unity. Data obtained on the drying of moist air are in good agreement with this theoretical result. The conditions assumed are also met with in the absorption of soluble gases in scrubbing towers where the gas velocity is high compared with that of the liquid and entrance turbulence is small. One investigation of such a case showed fairly good agreement with the equation.

C. IRWIN.

* The remainder of this set of Abstracts will appear in next week's issue.

Steam and electric power plant of Imperial Chemical Industries, Ltd., at Billingham. H. A. HUMPHREY, D. M. BUIST, and J. W. BANSALL (J. Inst. Elect. Eng., 1930, 68, 1233—1290).

Rubber mills and Banbury mixers. SCHNUCK.—See XIV. Purification of boiler feed-water. MOUSCADET.—See XXIII.

PATENTS.

Heat-treatment kiln. H. G. LYKKEN (U.S.P. 1,760,244, 27.5.30. Appl., 2.1.25).—The kiln has a stationary, annular hearth with provision for the application of heating, reducing, or oxidising gases upon it, and of heating gases below. A rotating cover makes a gastight joint with the hearth by means of sand seals, and carries rabbles; it may also have dependant radial division plates so that the hearth may be divided into zones for different treatments. The cover is preferably driven by a rope wrapped round its circumference. B. M. VENABLES.

Drying of materials [e.g., timber]. F. A. SECORD (U.S.P. 1,760,444, 27.5.30. Appl., 25.1.22. Renewed 19.10.29).—The material, e.g., a stack of timber, is first subjected to a high temperature by means of saturated air, and then to localised currents of cooler air, while a blanket of heat is maintained above. B. M. VENABLES.

Disintegrating mill. A. M. MARSH, Assr. to ALLIS-CHALMERS MANUF. CO. (U.S.P. 1,762,122, 3.6.30. Appl., 12.3.28).—A disc grinder has horizontal discs, the upper one of which is fixed and provided with a feed aperture, the lower disc is firmly coupled to the shaft of a motor, the whole being adjustable vertically to adjust the grinding size; the frame of the motor is flexibly supported in the horizontal direction. B. M. VENABLES.

Grinder and disintegrator. E. W. SCHULTZ and F. D. BANHAM (U.S.P. 1,759,245, 20.5.30. Appl., 4.5.29).—A power-driven device for operating a pestle in a manner similar to the action of a human hand is described. B. M. VENABLES.

Pulverising mill. G. GALAN (U.S.P. 1,762,056, 3.6.30. Appl., 27.8.27).—A pulveriser is provided with two feeding devices either of which is sufficient to deliver the full quantity required by the mill. When the feeder in operation chokes, the other one automatically comes into operation. B. M. VENABLES.

Pulverising apparatus. W. K. LIGGETT, Assr. to JEFFREY MANUF. CO. (U.S.P. 1,761,083, 3.6.30. Appl., 11.5.25. Renewed 25.4.29).—The hammers of a disintegrator are held in the operative position by fragile means so that when an uncrushable piece is struck the hammers can swing back to an inoperative position. B. M. VENABLES.

Apparatus for pulverising materials. F. H. DANIELS, Assr. to RILEY STOKER CORP. (U.S.P. 1,759,100, 20.5.30. Appl., 21.8.26).—The casing is divided into two pulverising zones (preferably by the disc of the rotor) which communicate only at the circumference. The primary zone has a pocket for catching tramp iron, and a beater with wide radial blades which act also as a fan; the secondary zone has means such as pins for finer grinding. B. M. VENABLES.

Device for (A) reducing and feeding reduced materials, (B) reducing materials. H. G. LYKKEN (U.S.P. [A] 1,760,245, 27.5.30, and [B] 1,761,138, 3.6.30. Appl., [A] 22.3.26, [B] 23.2.26).—The devices are disintegrators working substantially by cyclonic action, there being considerable clearance between the rotor and casing. In (A) the shaft is horizontal and the feed is supplied to the lower part of the casing; in (B) the shaft is vertical and the feed central. B. M. VENABLES.

[Apparatus for] material agitation. R. J. PEPPER, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,760,374, 27.5.30. Appl., 19.2.26).—A vessel is provided with a helical blade which runs close to the wall and tends to lift the material, and any material that sticks to the blade is removed by a fixed scraper in the upper part of the vessel. B. M. VENABLES.

Bow-tube film-type evaporator. R. C. JONES, Assr. to GRISCOM RUSSELL CO. (U.S.P. 1,760,907, 3.6.30. Appl., 26.6.23).—A bundle of steam-heated tubes is substantially horizontal and the liquid is sprayed or dribbled over the tubes to form films. Scale is removed by expansion and contraction, causing changes in curvature of the tubes which are intentionally bowed and rigidly supported at their ends. B. M. VENABLES.

Centrifugal separator. H. G. KOEPKE, Assr. to STANDARD BRANDS, INC. (U.S.P. 1,760,071, 27.5.30. Appl., 17.9.26).—A centrifuge suitable for the separation of yeast from wort, having continuous discharge of both products, is described. B. M. VENABLES.

Rotary or spinning [centrifugal] extractor. W. G. PETERS (U.S.P. 1,760,775, 27.5.30. Appl., 17.8.28).—A self-balancing centrifugal extractor for laundry or other work is described. The rotor is adapted to retain a layer of water on the interior of the outer wall, and an inner container for the goods is free to slide on the bottom to a certain extent and displace water from a position where the goods happen to be charged more heavily. B. M. VENABLES.

Centrifugal scrubber. P. M. KUEHN, Assr. to BARTLETT HAYWARD CO. (U.S.P. 1,759,315, 20.5.30. Appl., 31.5.27).—An apparatus of the type in which the rotors have a combined lifting and spraying action on the washing liquid is described. B. M. VENABLES.

Carrying out reactions with gases, vapours, or liquids, in which hydrogen, oxygen, or sulphur is present in the free or combined form. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,926, 15.4.29. Addn. to B.P. 293,077; B., 1928, 644).—The vessels used for carrying out the reactions described in the prior patent are made of iron or iron alloys which have been rendered insensitive to ageing by heating above Ac3, quenching, and tempering below 721°. A. R. POWELL.

Apparatus for contacting vapours with solids. W. MILLER, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,761,270, 3.6.30. Appl., 8.11.26).—The apparatus is suitable for the polymerisation of unstable hydrocarbons produced by cracking. The solid-treating material is supported on a perforated, conical, false

bottom within a gastight shell which is heated by products of combustion from the cracking still passing spirally round a jacket; the hot gases may also be passed through the solid material to regenerate it.

B. M. VENABLES.

Gas and liquid contact apparatus. M. H. KOTZEBUE (U.S.P. 1,759,750, 20.5.30. Appl., 26.5.27).—A bubbling tower is provided with tubular heat-exchangers at some of the trays to regulate the temperature of the reflux liquid.

B. M. VENABLES.

Apparatus for treating gases. M. AURIG (U.S.P. 1,760,623, 27.5.30. Appl., 29.4.29. Ger., 26.2.29).—A method of attaching sinusoidal plates in spaced relation on a wheel is described.

B. M. VENABLES.

Manufacture of liquids suitable for hydraulic machines or apparatus. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,200, 26.4.29).—In place of water or compressor oils there is used a glycol (or its highly concentrated aqueous solution) to which a high-molecular sulphonic acid is added to increase the lubricating power. A little butyl alcohol will suppress frothing. Examples are: ethylene glycol (63 pts.), a polyglycol ether (7 pts.), water (30 pts.), with 10–20% of Turkey-red oil or sulphuricinoleic acid; ethylene glycol (30 pts.), glycerol (30 pts.), water (20 pts.), 50% aqueous sulphonated olein (30 pts.). The f.p. of such mixtures is below -45° , and the viscosity at low temperatures is much less than that of compressor oil.

C. HOLLINS.

Balance. W. C. SEYTER, Assr. to TORSION BALANCE Co. (U.S.P. 1,759,139, 20.5.30. Appl., 21.5.29).—In a chemical balance a lens for reading the position of the rider is mounted on the rider arm in such way that it will slide longitudinally, but will always remain in focus with the beam.

B. M. VENABLES.

[Tunnel] kiln. H. M. ROBERTSON (U.S.P. 1,773,011, 12.8.30. Appl., 9.5.28).—See B.P. 331,224; B., 1930, 863.

Production of cooling [refrigerating] liquids. N. DAHL (U.S.P. 1,777,913, 7.10.30. Appl., 5.4.26. Nor., 7.2.21).—See B.P. 275,849; B., 1927, 801.

Crushing machine. E. B. SYMONS, Assr. to NORDBERG MANUF. Co. (U.S.P. 1,776,454, 23.9.30. Appl., 8.10.28).—See B.P. 327,216; B., 1930, 537.

Production of uniform pulverulent mixtures. H. BOLLMANN (U.S.P. 1,776,721, 23.9.30. Appl., 21.5.29. Ger., 5.7.28).—See B.P. 314,941; B., 1930, 170.

Separation of materials of different physical characters. H. M. CHANCE (B.P. 334,877, 7.5.29).—See U.S.P. 1,730,123 and 1,730,189; B., 1929, 964.

Manufacture of absorbent materials. J. BADDILEY and E. CHAPMAN, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,777,459, 7.10.30. Appl., 14.7.27. U.K., 15.7.26).—See B.P. 280,262; B., 1928, 41.

Apparatus for treating and mixing comminuted or finely-divided materials. A. B. and C. R. SMITH (U.S.P. 1,772,640, 12.8.30. Appl., 7.9.29. U.K., 2.8.29).—See B.P. 333,051; B., 1930, 932.

Apparatus for circulation of liquids. R. F. McKAY, H. WILLSHAW, W. G. GORHAM, and R. F. LEE,

Assrs. to AMER. ANODE, INC. (U.S.P. 1,777,648, 7.10.30. Appl., 27.2.28. U.K., 2.3.27).—See B.P. 293,072; B., 1928, 658.

Pasteuriser. F. LASSEN, Assr. to A./S. DE FORENEDE BRYGGERIER (U.S.P. 1,777,208, 30.9.30. Appl., 4.9.28. Denm., 19.2.25).—See Austr.P. 105,348; B., 1927, 801.

Gas-purifying apparatus. K. BÖMCKE, Assr. to DEUTS. ERDÖL A.-G. (U.S.P. 1,777,957, 7.10.30. Appl., 25.3.27. Ger., 23.12.25).—See B.P. 271,018; B., 1927, 516.

Liquefaction of gases. C. W. P. HEYLANDT, Assr. to FLÜGA A.-G. (U.S.P. 1,777,040, 30.9.30. Appl., 29.11.27. Ger., 29.12.26).—See B.P. 282,813; B., 1929, 501.

Apparatus for determining refraction and dispersion. H. VOELLMY (U.S.P. 1,776,712, 23.9.30. Appl., 12.1.28. Ger., 17.1.27).—See B.P. 283,859; B., 1928, 698.

Furnace walls. O. NYGAARD (B.P. 318,513, 19.8.29. U.S., 4.9.28).

[Fluid-cooled] furnace side walls. AMER. ENG. Co. (B.P. 335,067, 27.9.29. U.S., 8.8.29).

Refrigerator plant. J. STONE & Co., LTD., and E. G. ROWLEDGE (B.P. 335,208, 19.6.29).

Refrigerating apparatus of the absorption type. G. MAIURI and R. F. BOSSINI (B.P. 335,199, 17.4.29).

Continuous absorption refrigerating apparatus. J. O. BOVING (B.P. 335,039, 2.9.29).

Vaporising nozzles. J. FETTER (B.P. 335,222, 19.6.29).

Atomising nozzles more particularly for emulsifying purposes. A. BERGSVIK, and VITACREAM, LTD. (B.P. 335,159, 8.6.29).

Heating of materials in moving containers (B.P. 333,227). **Distillation apparatus** (B.P. 313,876).—See II. **Grinding balls** (B.P. 319,350).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Washing of coal on the Hoyois washer. A. GROUNDS (Fuel Econ., 1930, 5, 561–564).—The Hoyois washer is based on a combination of the trough washer and the upward-current classifier. Its advantages are: automatic running irrespective of variations in the shale content of the raw coal, large capacity for small ground space, high efficiency, close screening not required, and easy starting up and shutting down. Salient points in the design of the washer and the two main types of apparatus are described. Details are given of the unit installed at Gilly (Houilleres Unies du Bassin de Charleroi) for the washing of fine coal up to $\frac{1}{2}$ -in. size.

C. B. MARSON.

Properties of coal which influence carbonisation. H. A. J. PIETERS (Chem. Weekblad, 1930, 27, 518–523).—A discussion of the factors affecting the coking power of coal.

S. I. LEVY.

Analysis of [solid] fuels. E. TERRES and H. K. KRONACHER (Gas- u. Wasserfach, 1930, 73, 645–651, 673–677, 707–711).—Determinations carried out on four bituminous coals have shown that the loss of weight

amounts only to 55–60% of the weight of water actually evolved on heating in a current of air, nitrogen, or carbon dioxide. In the presence of air, moreover, some oxidation of the coal occurs. Similar but smaller differences between the loss of weight on heating and the true moisture content are exhibited also by cokes. Accurate values can be obtained only by direct determination of the water evolved, *e.g.*, by absorption in a calcium chloride tube, the fuel being heated at 100° in a current of inert gas. At higher temperatures, and even at 100° with some coals, appreciable decomposition of the sample may occur. The influence of the temperature on the yield of coke obtained in the crucible test has been determined over the range 450–1100°, and with times of heating of 15, 30, and 60 min., respectively. The coke yield–temperature curves run almost parallel for the four gas and coking coals examined. A combustion method for determining carbon, hydrogen, and nitrogen in solid fuels in one operation has been developed. The sample is burned in a current of pure electrolytic oxygen in a combustion tube provided with an especially long column of copper oxide. After absorption of the water and carbon dioxide in the usual manner the nitrogen and excess of oxygen are collected over alkaline pyrogallol, and the volume of the residual nitrogen is measured.

A. B. MANNING.

Marshall-Bird test for determining the agglutinating value of coal. K. A. JOHNSON and H. F. YANCEY (*Fuel Econ.*, 1930, 5, 553–555).—This test consists in carbonising an intimate mixture of 10 pts. by wt. of sand (40–50-mesh) and 1 pt. of coal (to pass 100-mesh) in a porcelain crucible at a uniform temperature of 950° in a vertical, cylindrical, electrically heated furnace; after carbonisation the resultant buttons are crushed in a testing machine and the agglutinating value reported is the average breaking strength of 10 buttons. The variables involved in the test on investigation gave the following results: (1) The agglutinating values of the coals examined decreased with increase in temperature of carbonisation up to about 950°, at which point the values of most coals increased slightly; a temperature of 950° is recommended. (2) The agglutinating values decreased with increase in the period of carbonisation; a period of 20 min. is suggested. (3) As the ratio of sand to coal increased, the agglutinating value decreased; the best ratio is that of 10 to 1. (4) The addition of one drop of glycerin to the sand-coal mixture is recommended; segregation of the coal or inert material is thereby avoided. (5) Tests carried out on the same coals in different laboratories give fairly satisfactory agreement, considering the empirical character of the test.

C. B. MARSON.

Determination of coking power of coal. G. DE Vos (*Natuurwetensch. Tijds.*, 1930, 12, 163–170).—The various methods in use in coke-oven laboratories in Belgium and Northern France are briefly described and adversely criticised. It is concluded that the content of resinous and volatile matter is of less importance than are the proportion and chemical nature of the mineral constituents, which determine the quantity and m.p. of the ash. Examples are given of contradictory and unreliable results obtained by the various methods

in use, and it is proposed that where reliable tests are necessary the material should be submitted to actual carbonisation on the large scale, following determinations of humic acid and of oxidisability, and X-ray examination.

S. I. LEVY.

Method of determination of activity of coke, and results obtained. D. J. W. KREULEN (*Chem. Weekblad*, 1930, 27, 514–518).—An apparatus is described by means of which dry air freed from carbon dioxide is caused to pass at a constant rate over a definite volume of the coke of definite particle size, contained in a platinum boat in a tube heated in an electric resistance furnace, the products being passed for a definite time through suitable absorption vessels containing solid absorbents. The activity is measured by the weight of carbon dioxide formed per unit of surface per unit of time. Very great differences are observed with different cokes at moderate temperatures (500° and 600°), but these diminish as temperature rises, and at 800° the activities are the same within the limits of experimental error. The activity of graphite is much below that of coke, but is increased by intimate admixture with coke, and by presence of catalysts (iron oxide). Diamond gave anomalous results.

S. I. LEVY.

Impact hardness, abrasion hardness, and reactivity of coke. R. A. MOTT (*Gas World*, 1930, 93, Coking Sect., 97–101).—The shatter test, used for determining coke hardness, may be employed also to assess abrasability. If a coke is abrasable, its use in a blast furnace is decreased, and a coke with a $\frac{1}{2}$ -in. shatter index of less than 97 is undesirable for blast-furnace purposes. The specific reactivities of cokes towards oxygen and carbon dioxide at temperatures above 1500° do not differ very much. Non-caking materials, such as anthracite and low-temperature breeze, when blended with coking coals give the most reactive, and good caking coals the least reactive, cokes; most cokes which are highly reactive with oxygen are also highly reactive with carbon dioxide. It is usually found that cokes of high shatter index, high real sp. gr., high porosity, and low reactivity give the highest combustion temperatures. The size of a coke is of more importance than its specific reactivity in influencing combustion phenomena under conditions comparable with those of a blast furnace; decrease in coke size increases reactivity and decreases the average combustion temperature.

C. B. MARSON.

Evaluation of active charcoal. E. BERL and W. HERBERT (*Z. angew. Chem.*, 1930, 43, 904–908).—Existing methods for the evaluation of active charcoal are found on examination to be untrustworthy, depending in many cases on the nature and amount of the inorganic constituents. A method based on the adsorption of methylene-blue from a 0.15% aqueous solution (60 c.c. per 0.1 g. of carbon) is described, the change in concentration of the solution being measured colorimetrically. The active surface of the sample is defined as 1 cm.² per 0.001 g. of dye adsorbed. In the case of highly active charcoals the values obtained vary with the final concentration of dye, although the variation may be decreased by increasing the initial concentration of methylene-blue; a modified method is therefore de-

scribed, using a 0.3% solution (50 c.c. per 0.1 g. of carbon). An end-point based on the complete disappearance is impracticable, and a standard final concentration of 0.1% is suggested. The variation of the observed adsorption with final concentration is determined for a number of technical varieties of active charcoal; the logarithms of these values give a straight-line graph, from which the desired results for a final concentration of 0.1% may be obtained by interpolation. A graph and a scale for the correction of results obtained by the above two methods to this final concentration are also given. H. A. PIGGOTT.

Recovery of organic nitrogen by dry distillation of agricultural waste. V. DIEGO (*Affinidad*, 1930, 10, 97—101; *Chem. Zentr.*, 1930, i, 2790).—Ammonia was obtained in 2.7% yield, together with tar, charcoal, acetic acid, and methyl alcohol. A. A. ELDRIDGE.

Hydrogenation of fuels. M. G. LEVI, C. PADOVANI, and A. MARIOTTI (*Annali Chim. Appl.*, 1930, 20, 361—404).—A description is given of the working of the Leuna plant for the hydrogenation of coal, lignite, heavy oils, and primary tars by the Bergius process. The results of a large number of experiments on Arsa coal (exceptionally rich in organic sulphur) and on a bituminous coal from Heraclea (Asia Minor), either alone or in presence of various catalysts, show that the hydrogenation of coal exhibits three phases and may take place in three ways: (1) At temperatures below 300°, at which decomposition or transformation of the complex molecules constituting the coal just begins, without generation of gases or vapours. The absorption of hydrogen is slight and a solid or semi-solid mass of high bitumen (substances extractable by solvents) content and marked agglomerating properties is obtainable. (2) At 350—400°, when destructive distillation of the coal begins, the hydrogenation is mainly in the liquid phases and, in intermittent operations, leads to the highest yields of total liquid products. (3) At 400—500° under 200—250 atm. pressure, cracking of the more resistant molecules takes place; hydrogenation occurs principally in the gas and light vapour phases, large amounts of hydrogen being absorbed. In general, addition of nickel oxide as catalyst does not appear to displace sensibly the temperature of hydrogenation, but the rapidity and intensity of the hydrogenation are greatly increased. With both coals the best results were obtained in presence of ferric or nickel oxide. Thus hydrogenation of the Arsa coal alone gave 18% of total oil (b.p. below 300°) and about 7% of benzine, whereas in presence of nickel (ferric) oxide 30 (28) and 14 (12)% were the respective yields. Sulphur is more abundant in the oils obtained by hydrogenation in presence of a catalyst, but this sulphur is mostly present in the medium and heavy oils. Hydrogenation benzine contains at most 1.6% S, whereas distillation benzine contains an average of 8% and resists all attempts to desulphurise it. Ferric oxide seems to be a better desulphuring agent than nickel oxide, but the loss on treatment with concentrated sulphuric acid and the phenol coefficient are lower in the liquid products obtained in presence of nickel oxide. T. H. POPE.

Determination of the calorific value of coke-oven

gas with the Junker calorimeter. H. GREWE (*Arch. Eisenhüttenw.*, 1930—1, 4, 75—85; *Stahl u. Eisen*, 1930, 50, 1269—1270).—Accurate results for the calorific value of a gas are obtained by the Junker calorimeter only when the incoming gas and air and the outgoing burnt gas are completely saturated with water vapour, the burnt gas is cooled to approximately the same temperature as the original gas mixture, and an accurate measurement is made of the volume of the burnt gas and residual air. Automatic recording apparatus for ensuring the observance of these precautions and for standardising the calorimeter are described in the original article. A. R. POWELL.

Volumetric relationships of the carbon monoxide process. R. NITZSCHMANN (*Metallbörse*, 1929, 19, 2413—2415, 2525—2526, 2581—2583; *Chem. Zentr.*, 1930, i, 2497).—Tabulated data relating to the production of producer gas between 477° and 1127° for reaction pressures of 0.5, 1.0, and 2.0 atm. and for oxygen-nitrogen mixtures containing 80—0% N are given.

A. A. ELDRIDGE.

Recovery of sulphur from coal gas. The Petit process. T. P. L. PETIT (*Natuurwetensch. Tijds.*, 1930, 12, 155—160).—An account of the process (cf. B.P. 195,061; B., 1923, 929 A) and of the reactions on which it is based is given. S. I. LEVY.

Velocity of ignition of gaseous mixtures. K. BUNTE and W. LITTERSCHEIDT (*Gas- u. Wasserfach*, 1930, 73, 837—842, 871—878, 890—896).—Experiments have been made with the object of investigating the influence of the several components of technical gas mixtures on the velocity of ignition, by the method of Gouy and Michelson. Binary and ternary mixtures of hydrogen, carbon monoxide, and methane were studied, and curves are drawn showing the variation of the velocity of ignition with composition for different proportions of gas and air. There is no simple relation between velocity and composition, and the curves, particularly those for the ternary mixture, have a very complicated form. Methane exercises a relatively large influence in lowering the velocity. The effect of carbon dioxide in lowering the velocity is more pronounced than that of nitrogen, presumably owing to the larger sp. heat of the former gas. A graphic method for determining the maximum velocity of ignition for any given mixture (with respect to the proportion of air required) is worked out on the basis of the measurements, and the values so obtained agree well with observations made on technical gas mixtures. The results obtained by the static method are, in general, higher than those given by the dynamical method used in this investigation, probably on account of the production of turbulence in the former. On the other hand, there is excellent agreement between the values given by the dynamical method and those obtained from the radial propagation of flame in soap bubbles.

F. L. USHER.

New methods in the manufacture of gas by suitable combination of standard arrangements. SCHUMACHER (*Gas- u. Wasserfach*, 1930, 73, 861—867).—A lecture, in which are discussed the methods by which the various operations in a gasworks may be most

economically adapted to meet a varying demand while maintaining a substantially uniform load on the furnaces. Examples of suitable lay-out are given.

F. L. USHER.

Defects in dry gas meters. WEHRMANN (Gas- u. Wasserfach, 1930, 73, 901–902).—The discs of dry meters are frequently found with incrustations which may prevent them from revolving. Examination of the incrustation (about 1 g.) collected from 50 instruments showed it to contain (1) an insoluble dust consisting of carbon, silica, and smaller proportions of metals; and (2) a soluble, dark brown, fluorescent, and viscous oil, evidently formed by the polymerisation of hydrocarbons present in the gas and absorbed by the grease used to lubricate the discs. The high degree of purification of the gas necessary to prevent this effect is impracticable, and alternative mechanical remedies are suggested.

F. L. USHER.

Determination of phenols (acid oils) in gas liquors and carbonisation liquors. E. KRES (Brennstoff-Chem., 1930, 11, 369–371). Cf. Heiduschka and Römisch, Gesundheitsing., 1930, 53, 53).—Fifty c.c. of a 10% solution of copper sulphate are added to 100 c.c. of the filtered liquor, the mixture, after acidification with dilute sulphuric acid, is distilled until about 20 c.c. remain, and the residue is then diluted with 50 c.c. of water and again distilled until only 15 c.c. remain. The phenols in the distillate are determined by means of a standard bromide-bromate solution, or colorimetrically. By using the procedure described the impurities which would otherwise interfere with the determination are either destroyed or rendered non-volatile.

A. B. MANNING.

Preparation of sulphur-containing balsamic mass from acetic acid tar. E. PALLAS (Pharm. Zentr., 1930, 71, 609–611).—The tar remaining after distillation in superheated steam of crude pyrolygneous acid is washed several times with warm or boiling water. The brown milky extract is rendered alkaline with calcium hydroxide, saturated with sulphur dioxide, and the clear liquid evaporated to a syrup. This is soluble to the extent of 95% in water, is a strong, slightly acid reducing agent, contains phenol, pyrogallol, and derivatives of pyridine and pyrrole and considerable amounts of sulphur derivatives, but no calcium salts, and resembles Peru balsam in its antiseptic properties. If the extract is not neutralised a darker product results, whilst if sodium hydroxide is used the product contains sodium sulphite.

H. E. F. NOTTON.

Some sources of error in the determination of the b.p. or of the boiling limits of commercial benzols by the method of Krämer and Spilker. W. MELZER (Brennstoff-Chem., 1930, 11, 371–372).—In using the distillation apparatus designed by Krämer and Spilker errors of 1% or more may arise through variations in the thickness of the wall or in the diameter of the condenser tube, or by judging the rate of distillation from the rate of formation of the drops (2 drops/sec.) instead of adjusting it accurately to 5 c.c./min.

A. B. MANNING.

Must the air content of a pycnometer be taken into account in determining its tare in the de-

termination of the sp. gr. of oils? SCHLÜTER (Chem.-Ztg., 1930, 54, 698).—The error introduced into a determination of the d of an oil by omitting to allow for the weight of air contained in the pycnometer is independent of the volume of the vessel and depends only on the d of the oil, varying from 0.00012 to 0.00036 when $d = 0.9–0.7$; i.e., it is within the limits of experimental error of the method. A. R. POWELL.

Heat transfer. OTHMER.—See I. Fruit-tree sprays. WEICHERZ.—See XVI.

See also A., Oct., 1255, **Speed of flame in a closed tube** (NAGAI). 1268, **Electro-decomposition of hydrocarbons** (AUSTIN). **Decomposition of paraffin hydrocarbons** (EGLOFF and others). 1277, **Determination of metal in [anti-knock] compounds** (DOSIOS and PIERRI).

PATENTS.

Vertical retorts for the carbonisation of coal and the like. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT Co., LTD. (B.P. 333,381, 1.8.29).—Coke-extracting and -discharge means consisting of a pivotally mounted coke chamber closed at its lower end are described. A curved plate attached to the top of the chamber serves to close the base of the retort when the chamber is tilted.

A. B. MANNING.

Retort and dryer for use in low-temperature carbonising. W. A. COLLIS (B.P. 333,020, 4.7.29).—The apparatus consists of a vertical tapering chamber heated on all sides by hot gases, and surmounted by an inclined chamber within which is a series of steps or terraces increasing in capacity as they descend. Each step is divided laterally into compartments provided with revolving scrapers which propel the charge from step to step and finally discharge it into the vertical chamber below. The inclined upper chamber has heating flues at the bottom and sides only. One or more gas and vapour outlets are provided at suitable positions.

A. B. MANNING.

Heating of materials in moving containers particularly applicable for destructive distillation. WOODALL-DUCKHAM (1920), LTD., and (SIR) A. McD. DUCKHAM (B.P. 333,227, 4.5.29).—The material is charged into containers which are then placed in turn on the platform of an annular kiln in such a manner that an outlet from the lower end of each container makes sealed connexion through an opening in the platform with a collecting main beneath the latter. The containers travel with the platform first through a zone of gradually rising temperature, then through a zone of high temperature, and finally through a zone of gradually falling temperature. They are then removed from the platform and their contents discharged. An annular liquid seal permits the rotation of the platform while maintaining a gastight connexion between the outlets from the containers and the collecting main. The apparatus may be modified to permit a current of gas to pass through the containers while they are travelling through the kiln.

A. B. MANNING.

Hydrogenation of the gases [and vapours] derived from the low-temperature distillation of solid fuels. COMP. INTERNAT. POUR LA FABR. DES

ESSENCES ET PÉTROLES (B.P. 311,280, 7.5.29. Fr., 8.5.28).—Either by predrying the solid fuel or by suitable treatment of the gases the water content of the latter as they enter the catalytic reaction chamber is adjusted to correspond with only the water of constitution of the initial material. Thus the gases leaving the retort may be treated by condensation followed by separation of part of the water and revaporisation of the tar and remaining water. A. B. MANNING.

Gas generators. HUMPHREYS & GLASGOW, LTD., Assees. of C. S. CHRISMAN (B.P. 333,072, 6.9.29. U.S., 18.10.28).—The fuel bed of the generator is supported by a rotatable bosh of inverted conical shape, having a central opening through which projects a perforated body or tuyère. Arrangements for removing the ash and clinker and for crushing the latter are claimed. A. B. MANNING.

Water-gas generators for bituminous fuels and by-product coke recovery therefrom. H. HIDAKA (B.P. 333,021, 4.7.29).—A revolving coke-extraction tube projects centrally up into the generator, and is adapted to remove a part of the coke carbonised at a comparatively low temperature. The remainder of the coke is gasified, yielding the amount of water-gas necessary to maintain a definite desired calorific value of the resulting mixed gas. The extraction tube is adjustable in a vertical direction. Within the tube is an extracting screw of variable speed of revolution and possessing a hollow shaft through which steam can be passed. A variable proportion of the blow gas is passed through the superheater to the waste-heat boiler, while the remainder is passed directly to the boiler, so that any fall in temperature in the generator can be compensated by increasing the degree of superheating of the steam supplied. A. B. MANNING.

Purification of distillation gases. CHEMINOVA GES. ZUR VERWERTUNG CHEM. VERFAHREN M.B.H. (B.P. 311,404, 6.5.29. Ger., 11.5.28).—The gas is charged at the ordinary pressure with a naphthalene solvent, the concentration of which, however, is below that necessary to bring about saturation of the gas at the temperature of the cooler places of the pipe system. The gas is then compressed so that at these places solvent charged with naphthalene separates out, and the gas finally has its pressure reduced to that at which it is used. This method of purification is suitable for systems using long-distance pressure transmission. No separation of the solvent can then occur at places where it may have an injurious effect, *e.g.*, in dry gas meters. A. B. MANNING.

Purifying from naphthalene the distillation gases of coal. GEWERKSCHAFT M. STINNES (B.P. 305,545 and 333,297, 6.2.29. Ger., 6.2.28).—(A) The gas is allowed to take up the vapour of a naphthalene solvent at about 100° and is then cooled, whereupon the solvent vapours are precipitated, carrying down with them the whole of the naphthalene in the gas. The solvent may be recirculated after being freed from at least a part of the naphthalene absorbed. (B) When the gas is to be delivered under pressure, *e.g.*, in long-distance transmission, the process is carried out by first compressing the gas, then bubbling the hot compressed gas through

the solvent, and finally cooling the gas charged with solvent in order to precipitate the latter with the naphthalene. [Stat. ref. to (B).] A. B. MANNING.

Extraction of ethylene from ethylene-containing gaseous mixtures. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 308,687, 23.3.29. Fr., 26.3.28).—Ethylene-containing gas such as coke-oven gas is partly liquefied and the liquid so produced is used to scrub the unliquefied portion free from such impurities as acetylene and oxides of nitrogen. The liquid is then distilled, the uncooled gases before the liquefaction being used as a source of heat. T. A. SMITH.

Removal of organic sulphur compounds from gases. O. W. LUSBY (B.P. 305,027, 25.1.29. U.S., 28.1.28).—The gases are treated with a catalyst containing one or more metals of group VI, *e.g.*, uranium, chromium, and/or molybdenum, with one or more metals of groups I—V, *e.g.*, cerium, copper, lead, etc., the process being carried out at a temperature below the m.p. of any component of the catalyst, and preferably between 200° and 500°. The organic sulphur compounds are thereby converted into hydrogen sulphide, which is then removed from the gases in known manner. [Stat. ref.] A. B. MANNING.

Method and apparatus for distilling [hydrocarbon oil]. E. B. BADGER & SONS Co., Assees. of W. T. HALL (B.P. 313,876, 7.6.29. U.S., 18.6.28).—Oil is fed on the plates of fractionating columns after passing through preheaters, in which it is heated by means of the hot residual oil from the columns. Since insufficient heat for the vaporisation of the desired fractions is introduced in this way, residue from the columns is passed through a heating tube and then returned to the plates of the fractionating column. The burner of the heating element is thermostatically controlled by the temperature of the oil entering the fractionating column. A cracking unit may be arranged to consist of a fractionating column at the top of which cracking stock is fed so that it comes in contact with the vapours entering from a reaction chamber at a point lower in the column. Oil from the bottom of the column is passed through a tube heater and delivered to the reaction chamber under thermostatic control. Part of the heated oil is returned to the lower portion of the fractionating column to control the evaporation in the column. T. A. SMITH.

Distillation of hydrocarbon oils. R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,758,590, 13.5.30. Appl., 4.4.25).—Oil is vaporised by superheated steam into a series of evacuated, superposed, frusto-conical conduits fitted with baffle plates which prevent unatomised oil being carried forward. Any spray caught by the baffles and walls of the conduits is reatomised by the steam jet. The oil is preferably preheated and the steam is used at 530—650°. The distillation is carried out at 25—100 mm. T. A. SMITH.

Conversion of hydrocarbon oils. G. F. BENHOFF (U.S.P. 1,748,201, 25.2.30. Appl., 1.4.24).—Crude oil is heated to distillation temperature and the vapours are passed to a dephlegmator. The residual oil is passed

through a cracking tube to an evaporator, the vapours produced being scrubbed by the condensate from the first towers in a second set. Part of the reflux from the dephlegmators is returned to the cracking coil for further cracking. The oil and carbon residues from the cracking evaporator are used as fuel.

T. A. SMITH.

Cracking of hydrocarbon oils. E. C. HERTHEL and T. DE C. TIFFT, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,747,437, 18.2.30. Appl., 12.8.27).—Oil, heated to cracking temperature under pressure by being passed once through a heating coil, is transferred to a dephlegmator, where it is maintained under pressure. Vapours containing gasoline are removed and the reflux is added to the raw cracking stock. The unvaporised oil from the first operation is passed to a second dephlegmator under a lower pressure, where part is volatilised, some of which is obtained as reflux. This reflux is heated to cracking temperature in a second heating coil and returned to the first dephlegmator.

T. A. SMITH.

Cracking of hydrocarbon oils. J. E. BELL and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,758,818, 13.5.30. Appl., 22.1.25).—A horizontal cracking drum is divided by partitions into a number of sections, arrangements being made for oil to pass from section to section as required. Oil from each section is pumped through a heating coil designed to suit the heat requirements of that part of the circuit. The coils of each section are placed in flues through which the combustion gases pass in succession, such gases passing first through the flue containing the coil through which the least cracked oil is being pumped. The heating surface of the coils is increased and the speed of circulation decreased from section to section. The furnace gases are "tempered" by returning a portion of them from the last flue to the first by an injector device. Raw stock is preheated by bringing it in direct contact with the vapours from the drum in a reflux tower, the reflux and raw oil being delivered into the first section of the cracking drum. A portion of the oil containing tar is continuously removed from the last section of the drum to prevent its accumulation in the system.

T. A. SMITH.

Cracking of hydrocarbons. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 334,241, 30.5.29).—Hydrocarbons are cracked by passing them through baths of molten salts which have high b.p., so that little or no salt is vaporised during the operation. An equimolecular mixture of barium and calcium chlorides, m.p. about 500°, is suitable for the cracking of butane at 750°. The products consisted of 18.5% of propylene and 18.7% of ethylene. A number of other suitable salt mixtures the m.p. of which range from 426° to 663° are given. Molten tellurium may also be used and has a beneficial catalytic effect. In working with tellurium it is necessary to use aluminium-lined steel vessels or other material not attacked by tellurium.

T. A. SMITH.

[Cracking] treatment of [hydrocarbon] oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,018, 29.4.30. Appl., 10.2.22. Renewed 2.11.25).—A mixture of oil and aluminium chloride is fed into a dephlegmator in which vapours from the cracking opera-

tion are being treated. The reflux from the dephlegmator column is passed through a heating coil and delivered into a drum from which vapours are taken to the dephlegmator. A certain amount of residue is continuously withdrawn from the drum and the aluminium chloride is recovered. The aluminium chloride does not escape from the system with the cracked vapours, and it is not necessary to add it continuously.

T. A. SMITH.

Cracking of petroleum and mineral oils and their different fractions. P. NACHTSHEIM (U.S.P. 1,758,618, 13.5.30. Appl., 8.5.26).—Oil is heated in cracking coils and delivered into towers fitted with inclined baffles in which the evolved vapours are repeatedly caused to meet countercurrentwise the unvaporised and reheated fractions. The vapours are then taken through a dephlegmator to a condenser. Separators for carbon are placed in the oil stream and are periodically cleaned.

T. A. SMITH.

Cracking of heavy hydrocarbons to produce lighter hydrocarbons and coke. H. L. PELZER and E. C. HERTHEL, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,757,843, 6.5.30. Appl., 16.9.22).—Oil is cracked under pressure by circulating through it superheated gases until the oil has been reduced to a coke residue. Carbon formed in the gas-heating coils is removed by "burning out." External heating of the pressure coke still is avoided.

T. A. SMITH.

Production of lower-boiling hydrocarbons. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,019, 29.4.30. Appl., 14.1.25. Renewed 22.1.29).—Oil is circulated under pressure through a heating coil and a horizontal vaporising chamber, the volume of oil and free space in this chamber being adjusted by the height of the intake to the circulating pump. A portion of the heavy carbon-containing residue is continuously withdrawn from the bottom of the vaporising chamber. The diameter of the heating tubes is 2—4 in. and of the vaporising chamber 20—48 in.

T. A. SMITH.

Manufacture of acetylene and other [unsaturated hydrocarbon] products. R. G. WULFF (B.P. 334,178, 25.2.29).—Hydrocarbons such as casing-head gasoline, benzene, and gas oil are heated under a partial pressure of 0.5—75 mm. Hg to above 720° in a carbundum tube. The low partial pressure is attained by mixing the vapour with a diluent such as steam, mercury vapour, nitrogen, etc. The duration of heating is 0.002—5 sec., after which the gases are immediately cooled. Acetylene, ethylene, etc. are then separated and the residual gas is used for heating the reaction tube. Suitable apparatus is described.

T. A. SMITH.

Manufacture of viscous oils and hydrocarbon products of low b.p. I. G. FARBERIND. A.-G. (B.P. 313,879, 11.6.29. Ger., 18.6.28).—Hydrogenation of oils etc. is carried out in two stages, the first at the lower temperature so that asphaltic materials are not converted into coke. The lighter constituents are removed after the first treatment and the residue is then cracked or hydrogenated. Catalysts may be employed in both stages. The first stage should be effected in the liquid phase at below 420°. Solid paraffins may be separated

from the distillate after the first hydrogenation. The liquid fractions contain good lubricating oils.

T. A. SMITH.

Refining of mineral oils and like carbonaceous materials. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 332,944, 27.4. and 21.11.29).—The oils are heated with hydrogen under pressure in the presence of a colloidal molybdenum compound, *e.g.*, the oxide, which is adsorbed on a finely-divided solid such as active carbon. The conditions are so chosen that no considerable cracking of the oil takes place.

A. B. MANNING.

Refining of [hydrocarbon] oils. L. C. HUFF, ASST. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,026, 29.4.30. Appl., 6.6.23. Renewed 20.2.28).—In the treatment of oils with refining liquids, streams of oil and liquid are passed at high velocity through tubes containing spiral baffles, whereby intimate contact is effected. The mixture is then allowed to settle and is separated.

T. A. SMITH.

Refining of hydrocarbon oils. E. C. HERTHEL, and H. L. PELZER, ASSIS. to SINCLAIR REFINING Co. (U.S.P. 1,756,154, 29.4.30. Appl., 16.6.28).—Crude oil is vaporised and the vapours are passed into a dephlegmator. The heaviest portions are removed and the vapours then passed through a supply of hot oil maintained alkaline by addition of caustic soda. The vapours are then passed through a series of fractionating columns, whereby lubricating oil fractions of good colour and stability are obtained.

T. A. SMITH.

Processing of heavy oils. A. J. SLAGTER, ASST. to TRANSCONTINENTAL OIL Co. (U.S.P. 1,757,596, 6.5.30. Appl., 28.3.24).—A pressure still is fitted with a dephlegmator into which the oil to be treated is sprayed and brought in direct contact with the vapours. The oil thus preheated enters the still through the vapour line, and is circulated in the still by means of a pump submerged in the oil. The circulating pump may be connected to an external heating coil so that oil may be circulated from the still through the heating coil and be delivered again into the still.

T. A. SMITH.

Decolorisation of hydrocarbon oils. N.V. MENBOUW-EN CULTUURMAATS. "BOETON" (B.P. 334,833, 5.4.29. Holl., 30.1.29).—The oil is treated with a solution containing at least 58% of a hygroscopic heavy-metal chloride (*e.g.*, ferric chloride solution, *d* 1.6). Less loss is obtained than with sulphuric acid. The process is suitable for cracked gasoline, kerosene, or lubricating oil. Some oils may require more than one treatment. The chloride solution may be used again after the removal of the polymerised substances.

T. A. SMITH.

Production of hydrocarbons of high b.p. range [suitable for lubricants] from coal, shale, and other bituminous substances. I. G. FARBENIND. A.-G. (B.P. 312,050, 8.4.29. Ger., 18.5.28).—The initial materials are treated with organic solvents at raised temperatures and under pressure, and the products thereby extracted are subjected to treatment with reducing gases, under such conditions of temperature, pressure, and time, that no substantial decomposition

to products of lower b.p. occurs. The extracted oil is preferably freed from paraffin wax prior to the reduction treatment, which, if desired, may be carried out in the presence of a suitable catalyst, *e.g.*, a mixture of molybdc acid and zinc oxide.

A. B. MANNING.

Manufacture of activated carbon. R. THRELFALL (U.S.P. 1,777,943, 7.10.30. Appl., 31.5.27. U.K., 4.6.26).—See B.P. 270,505; B., 1927, 515.

Reactivation of decolorising carbon. F. W. MEYER (U.S.P. 1,771,719, 29.7.30. Appl., 14.9.29. Ger., 8.9.28).—See B.P. 317,017; B., 1930, 936.

Manufacture of water-gas. F. WINKLER, ASST. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,876, 30.9.30. Appl., 7.5.28. Ger., 27.9.22).—See G.P. 437,970; B., 1927, 548.

Purification of liquid hydrocarbons. F. B. THOLE and S. T. CARD, ASSIS. to ANGLO-PERSIAN OIL Co., LTD. (U.S.P. 1,776,340, 23.9.30. Appl., 6.11.24. U.K., 11.1.24).—See B.P. 231,944; B., 1925, 436.

Deodorisation of petroleum hydrocarbon distillates. Manufacture of lubricating oils. L. EDELEANU, ASST. to EDELEANU GES.M.B.H. (U.S.P. 1,776,752—3, 23.9.30. Appl., [A] 16.1.28, [B] 28.5.28. Ger., [A] 25.6.27, U.K., [B] 14.4.28).—See B.P. 301,955 and 307,649; B., 1929, 161, 386.

Oxidising paraffins, waxes, and the like. F. HOFMANN, ASST. to I. G. FARBENIND. A.-G. (U.S.P. 1,762,688, 10.6.30. Appl., 14.3.28. Ger., 18.3.27).—See B.P. 298,704; B., 1928, 919.

Fuel burners. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 335,806, 20.11.29).

Burners for pulverised fuel. G. E. K. BLATHE (B.P. 335,275, 25.6.29).

Contacting vapours with solids (U.S.P. 1,761,270). **Liquids for hydraulic machines etc.** (B.P. 333,200).—See I. Pure naphthalene (B.P. 333,352). **Pure anthracene** (B.P. 319,762).—See III. **Hydrogen** (B.P. 311,737). **Carbon disulphide** (B.P. 314,060 and 334,856).—See VII.

III.—ORGANIC INTERMEDIATES.

Use of calcium chloride in the dehydration of alcohol. K. B. EDWARDS and R. LACEY (J.S.C.I., 1930, 49, 422 *r*).—The use of fused calcium chloride in recovering concentrated alcohol from residues is recommended. When 20% (wt./vol.) was added to 80% alcohol and rapidly fractionated, a 95% alcohol distillate was obtained. A repeat distillation after a similar addition gave 52% of 98% and 24% of 95.5%. The process is simple and economical, and miscibility tests are given for control.

Measuring and recording p_H . WULFF and KORDATZKI.—See XI.

See also A., Oct., 1270, Determination of purity of acetic anhydride (ROSENBAUM and WALTON). 1279, Methylation of α -naphthylamine (GOKHLE and MASON). 1280, Formation of diazo-oxy-compounds (BUCHERER and TAMA). 1282, Novel preparation of 3:5-dinitro-*p*-cresol (HODGSON and SMITH). Sulphonation of β -naphthol in presence

of boric acid (ENGEL). 1292, New synthesis in the 1:2-naphthanthraquinone series. Derivatives of naphthanthraquinone (WALDEMANN), 1303, Rapid determination of furfuraldehyde (NOLL and others).

PATENTS.

Production of acetaldehyde from acetylene. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 334,427, 21.10.29).—In the conversion of acetylene into acetaldehyde by action of steam a catalyst is used comprising zinc oxide activated with a molybdate or molybdic acid. C. HOLLINS.

Manufacture of acetic anhydride. W. W. GROVES. From E. B. BADGER & SONS Co. (B.P. 333,991, 17.6.29).—During addition of sulphur chloride and chlorine (or sulphuryl chloride and chlorine) to sodium acetate pasted with acetic anhydride, the reaction mixture is cooled by means of coils carrying ammonia and brine circulating through a compressor. The temperature is adjusted by means of the expansion valve, and the time of charging is materially shortened. A suitable plant is figured. C. HOLLINS.

Manufacture of acetic acid [from formaldehyde and carbon monoxide]. BRIT. CELANESE, LTD., S. J. GREEN, and R. HANDLEY (B.P. 334,207, 27.5.29).—A mixture of carbon monoxide and formaldehyde vapour is heated at 300–400° and under 100–300 atm. pressure, preferably in presence of phosphoric acid, with or without addition of copper or copper compounds. Aqueous formaldehyde or methylal may be used. C. HOLLINS.

Manufacture of aliphatic compounds [acids from alcohols etc. and carbon monoxide]. BRIT. CELANESE, LTD., S. J. GREEN, and R. HANDLEY (B.P. 334,189, 27.5.29).—The paraffin hydrocarbon by-products produced in the catalytic manufacture of acids from alcohols etc. and carbon monoxide are removed, *e.g.*, by adsorption on charcoal or silica gel, before recirculation of the reaction gases. C. HOLLINS.

Production of compounds from propylene. J. W. WOOLCOCK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 334,228, 30.4.29).—Propylene is absorbed in a mixture of sulphuric acid and acetic acid, or of isopropyl hydrogen sulphate and acetic acid, below 60°; on dilution with water a mixture of isopropyl alcohol and acetate separates, and the aqueous layer is boiled to yield more isopropyl alcohol, or the whole diluted liquor is boiled without separation in order to increase the proportion of alcohol to ester. A mixture of equal volumes of 99.5% sulphuric acid and glacial acetic acid absorbs propylene alone from admixtures with ethylene; higher olefines may be removed by pretreatment with cooled 80% sulphuric acid. C. HOLLINS.

[Production of esters by] catalytic dehydrogenation. E. I. DU PONT DE NEMOURS & Co. (B.P. 312,345, 3.4.29. U.S., 26.5.28).—An aliphatic alcohol is passed in vapour form over a catalyst comprising copper and a difficultly reducible oxide or mixture of oxides, *e.g.*, oxides of magnesium and manganese, or of magnesium and zinc, at 375° or above, and at a pressure of at least 4000 lb./in.² The product from ethyl alcohol contains 20% of ethyl acetate. C. HOLLINS.

Production of esters [of sulphonated higher fatty acids etc.]. H. T. BÖHME A.-G. (B.P. 315,832, 24.6.29. Ger., 19.7.28).—Higher fatty or other suitable acids are esterified with an alcohol of relatively low mol. wt. (other than glycerol), and the esters are sulphonated, preferably below 15°, to give wetting agents. Butyl ricinoleate, *e.g.*, is treated with 1.4 pts. of concentrated sulphuric acid below 15°. C. HOLLINS.

Preparation of alkyl cyanides. IMPERIAL CHEM. INDUSTRIES, LTD., and T. S. WHEELER (B.P. 333,989, 14.6.29).—An alkyl halide and a metal cyanide, *e.g.*, ethylene dichloride and sodium cyanide, are heated in presence of the corresponding nitrile (*e.g.*, succinic nitrile) as diluent. C. HOLLINS.

Manufacture of carbohydrate compounds [soluble hydroxyalkyl ethers]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,282, 4.6.29).—The carbohydrate (potato meal, dextrin, cotton) is treated with an alkylene oxide at 15° in presence of aqueous caustic alkali of 20% or lower concentration without application of pressure. C. HOLLINS.

Preparation of pyridylalkines and piperidylalkines. A. BOEHRINGER (B.P. 312,919, 29.5.29. Ger., 1.6.28).—Pyridines having side-chains, $R \cdot CO \cdot CH_2$, in 2- or 2:6-positions are hydrogenated in stages or otherwise in presence of nickel, platinum, etc. 2-Phenacylpyridine yields 2- β -hydroxy- β -phenylethylpyridine, m.p. 107°, and on further hydrogenation the corresponding hexahydrocarbinol, m.p. 87°, b.p. 160°/1 mm. C. HOLLINS.

Separation of mixtures of secondary and tertiary amines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,349, 28.6.29).—The mixture is treated with the calculated amount of phthalic anhydride in absence of a neutral solvent. The phthalamide acid is extracted with alkali, leaving pure tertiary amine; the secondary amine may be recovered from the phthalamide acid. The application to mixtures of mono- and diethylanilines, methylanilines, benzylanilines, of diphenylamine and diphenylethylamine, and of 4-methoxy-3- β -hydroxyethoxytoluene and dimethylaniline, is described. C. HOLLINS.

Production [purification] of 2:4:6-tribromoaniline and its acyl derivatives. BRIT. CELANESE, LTD., and B. E. M. MILLER (B.P. 334,217, 29.5.29).—Crude 2:4:6-tribromoaniline is sublimed at 160–165° and then acylated, especially diacetylated, to give colourless products suitable for incorporation with cellulose acetate etc. for non-inflammable films. C. HOLLINS.

Manufacture of benzyl chloride. IMPERIAL CHEM. INDUSTRIES, LTD., N. BENNETT, H. DODD, W. C. SPRENT, and F. HOLT (B.P. 334,260—1, 1.6.29).—(A) The crude benzyl alcohol recovered from the benzylation of cellulose is freed from iron etc. by addition of alkali (anhydrous sodium carbonate), filtered, and esterified with hydrochloric acid. (B) After esterification the product is treated with chlorine, whereby unesterifiable benzyl compounds are converted into benzyl chloride and benzaldehyde; the latter is removed by means of bisulphite, and benzyl chloride is distilled. C. HOLLINS.

Purification of phenylethyl alcohol. E. C. BRITTON, Assr. to Dow CHEM. Co. (U.S.P. 1,752,365, 1.4.30. Appl. 9.6.23).—The phenylethyl alcohol in a crude product is converted selectively into its hydrogen phthalate by heating with phthalic anhydride in benzene. The hydrogen phthalate is removed as sodium or other soluble salt and hydrolysed. C. HOLLINS.

Manufacture of aromatic [hydr]oxyaldehydes. J. D. RIEDEL—E. DE HAËN A.-G. (B.P. 333,313, 18.5.29. Addn. to B.P. 285,451 and 290,649; B., 1928, 873; 1929, 672).—The mixture of 3-hydroxy-4-methoxy-methoxy-1-propenylbenzene and methoxyvanillin obtained from safrole by the process of the prior patent is separated by fractional acidification. The former compound is converted into vanillin by methylation, hydrolysis with dilute acid, and oxidation; the latter is acylated, hydrolysed, methylated, and de-acylated to give vanillin, or is hydrolysed to protocatechualdehyde with alcoholic acid. C. HOLLINS.

Manufacture of quinaldine. L. P. KYRIDES, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,752,492, 1.4.30. Appl. 16.7.23).—Aniline is added to a mixture of hydrochloric acid in nitrobenzene and at 90–110° aldol or crotonaldehyde is slowly run in. Improved yields are obtained. C. HOLLINS.

Manufacture of monobrominated menthane. SCHERING-KAHLBAUM A.-G. (B.P. 334,474, 24.12.29. Ger., 31.12.28).—Bromomenthane, b.p. 115–120°/15 mm., is obtained by bromination of menthane in presence of iodine at 0°. C. HOLLINS.

Purification of naphthalene. GES. F. TEERVER-WERTUNG M.B.H., and A. and G. SPILKER (B.P. 333,352, 1.7.29).—Centrifuged, crude (75–80%) naphthalene is melted and treated with about 0.15 pt. of aqueous sodium hydroxide (d 1.1). The separated naphthalene is washed with water and fractionally distilled, whereby 60–65% of the material is obtained pure (m.p. above 79°) and 10% of m.p. 78–79°. C. HOLLINS.

Production of hydrocarbons from naphthalene. J. VARGA B.P. 333,453, 11.10.29. Ger., 13.10.28).—Naphthalene is hydrogenated under pressure of more than 100 atm. in presence of molybdenum or tungsten compounds and hydrogen sulphide (or substances which produce it) which serves to activate the catalyst. The temperature is maintained above or below the critical point (480–500°) at which a sudden absorption of heat occurs, followed by a rise of about 100°. For tetra- and deca-hydronaphthalenes naphthalene is heated for 1 min. at 460° with 4% of sulphur, 2% of molybdc acid, and hydrogen at 120 atm. For benzene the same proportions are used at 540° for 1 hr. C. HOLLINS.

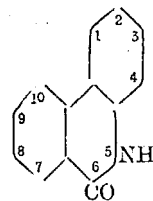
Production of pure anthracene. RÜTGERSWERKE A.-G., and L. KAHL (B.P. 319,762, 30.8.29. Ger., 29.9.28).—Crude anthracene is dissolved in 2 pts. of an organic base (or mixture of bases, e.g., pyridine, quinoline, crude coal-tar bases, etc.) and heated at 160–170° with potassium hydroxide equivalent to the carbazole content. Pure anthracene is filtered off after cooling. Part of the bases used may be replaced by solvents such as benzene, halogenated hydrocarbons, etc. C. HOLLINS.

[Synthesis of] anthraquinone derivatives. H. A. E. DRESCHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 334,166, 21.2.29).—The carbamide made by phosphogenating *o*-(4-chloro-3-aminobenzoyl)benzoic acid is cyclised with 100% sulphuric acid at 120° to give 2:1- and 3:2-chloroaminoanthraquinones, separable by fractional dilution. C. HOLLINS.

Manufacture of anthraquinone intermediates and dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and W. W. TATUM (B.P. 333,992, 18.6.29).—The mixture of 1:5- and 1:8-diaminoanthraquinones obtained by reduction of the product of nitration of anthraquinone is converted into the sulphate, which is heated at 200–220° to yield a mixture of diaminoanthraquinone-disulphonic acids. The product may be brominated, giving bromodiaminoanthraquinonesulphonic acids, from which, by replacement of bromine by a *p*-toluidino-group, a blue wool dye is obtained. C. HOLLINS.

Manufacture of nitrogen-containing derivatives of the benzanthrone series. I. G. FARBENIND. A.-G. (B.P. 333,882, 13.3.29. Addn. to B.P. 307,926; B., 1930, 810).—Under milder conditions mononitro-compounds are obtained. The nitration of 2:3:7:8-dibenzpyrene-1:6-quinone in nitrobenzene at 15° for 12 hrs., and of 1:2:7:8-dibenzpyrene-3:6-quinone in nitrobenzene at 130–135° for 5 min., is described. C. HOLLINS.

Manufacture of phenanthridone, homologues, derivatives, and substitution products thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,173, 2.5.29).—A fluorenone in sulphuric acid is treated with hydrazoic acid in benzene, whereby the oxime probably formed first undergoes the Beckmann transformation with production of a phenanthridone. Fluorenone gives a 93% yield of phenanthridone (annexed formula). The preparation of 3:8-dibromo- (m.p. 313°), 3-nitro- (m.p. 343°), 3-amino- (m.p. 285–286°), and 3-hydroxy-phenanthridones, phenanthridone-1-carboxylic acid, and 2-hydroxy-1:2-benzophenanthridone, is described. C. HOLLINS.



Manufacture of emulsions. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,587, 15.3.29).—The residue from the distillation of synthetic methyl alcohol (from carbon oxides), e.g., "isobutyl oil," b.p. 150–280°, containing ketones (45%), lactones (19%), acids and esters (15%), alcohols (15%), and phenols (6%), is mixed with an alkynaphthalenesulphonic acid, or with the sulphonation product from palm oil and phenol, to give an emulsifying agent for cresol, carbon disulphide, chlorinated hydrocarbons, etc. C. HOLLINS.

Cleaning of liquid solvents. J. H. BEAUMONT. From R. R. STOLLEY CORP. (B.P. 333,217, 6.5.29).—Solvents after use in dry-cleaning processes are purified by passing them upwards through aqueous caustic alkali, surmounted by a layer of active charcoal if desired, then through water, and finally over calcium chloride. C. HOLLINS.

Preparation of carbocyclic hydrocarbons. O. NICODEMUS and W. BERNDT, Assrs. to GEN. ANILINE

WORKS, INC. (U.S.P. 1,776,924—5, 30.9.30. Appl., [A] 20.4.26, [B] 21.11.27. Ger., [A] 24.4.25, [B] 22.6.25).—See B.P. 251,270 and 253,911; B., 1927, 742, 828.

Aminoalkylation of amines. W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER, ASSTS. to WINTHROP CHEM. CO., INC. (U.S.P. 1,752,617, 1.4.30. Appl., 12.1.28. Ger., 25.1.27).—See B.P. 301,401; B., 1929, 164.

Manufacture of halogen-substituted aromatic tertiary amines. G. KALISCHER and K. KELLER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,777,266, 30.9.30. Appl., 8.7.26. Ger., 15.7.25).—See B.P. 288,665; B., 1928, 441.

Manufacture of aromatic derivatives of formamide. R. WIETZEL, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,777,777, 7.10.30. Appl., 11.3.26. Ger., 13.3.25).—See B.P. 269,302; B., 1927, 458.

Naphthalene derivatives and their applications in dye preparations. W. GIBSON, A. J. HAILWOOD, J. B. PAYMAN, and A. SHEPHERDSON, ASSTS. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,776,970, 30.9.30. Appl., 1.8.28. U.K., 29.8.27).—See B.P. 300,800; B., 1929, 124.

Application of naphthalene derivatives to dye preparations. W. GIBSON, A. J. HAILWOOD, J. B. PAYMAN, and A. SHEPHERDSON, ASSTS. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,777,038, 30.9.30. Appl., 21.10.29. U.K., 29.8.27).—See B.P. 300,800; B., 1929, 124.

Manufacture of anthraquinone intermediates. W. W. TATUM, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,869, 30.9.30. Appl., 10.2.26. U.K., 11.1.26).—See B.P. 268,891; B., 1927, 437.

Manufacture of halogenated anthraquinones. A. SHEPHERDSON, W. W. TATUM, and H. M. BUNBURY, ASSTS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,932, 30.9.30. Appl., 10.2.26. U.K., 11.1.26).—See B.P. 271,023; B., 1927, 518.

Ethylene from gaseous mixtures (B.P. 308,687). **Naphthalene from coal gas** (B.P. 305,545 and 333,297). **Acetylene and other unsaturated hydrocarbons** (B.P. 334,178).—See II. **Soluble leuco-esters** (B.P. 333,146—7).—See IV. **Aluminium alcoholates** (B.P. 334,820).—See VII. **Vulcanisation accelerators** (U.S.P. 1,747,187—8).—See XIV. **2:3 [βγ]-Butylene glycol by fermentation** (B.P. 315,263).—See XVIII.

IV.—DYESTUFFS.

See A., Oct., 1260, Effect of light on dyes (KRAIS). 1280, Mechanism of azo-dye coupling (BUCHERER and TAMA). 1291, Reactions of quinol (EVANS and DEHN). 1303, Combination between dyes and gelatin granules (RAWLINS and SCHMIDT).

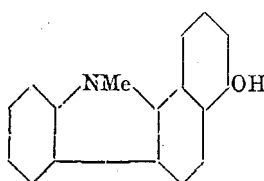
PATENTS.

Production of colouring matters and intermediates [soluble leuco-esters]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,146, 25.1.29).—The sodium salt of the disulphuric ester of leuco-β-aminoanthraquinone is oxidised with ferricyanide in feebly acid solution to form the disul-

phuric ester of leucoindanthrone, together with a more soluble dark green compound which can be diazotised and coupled with β-naphthol; the azo compound couples with *p*-nitrodiazobenzene in alkaline solution. Similar products are obtained from leuco-esters of β-acetamidoanthraquinone, or of 3-chloro-2-aminoanthraquinone. C. HOLLINS.

Vat dye derivatives [soluble leuco-esters]. L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,147, 29.1.29).—The leuco-ester of a vat dye is mixed in paste form with a dispersing agent (sulphite-cellulose liquor, aminated or oxidised sulphite-cellulose liquor, soaps, formaldehyde-naphthalenesulphonic acids, molasses, saponin, alkyl naphthalenesulphonic acids, naphthalene-di- or -tri-sulphonic acid), and dried. C. HOLLINS.

Manufacture of vat dyes [from isatin and hydroxynaphthacarbazoles] G. FARBENIND. A.-G.

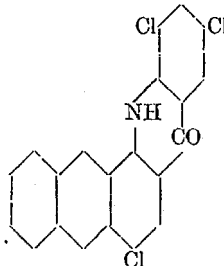


(B.P. 316,099, 22.7.29. Ger., 21.7.28).—Isatin-α-anil is condensed in acetic anhydride with 4-hydroxy-11-methyl-α-naphthacarbazole (annexed formula) to give a deep black vat dye. C. HOLLINS.

Manufacture of [indigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 318,107, 16.8.29. Ger., 27.8.28).—A 5-halogeno-7-alkoxy-4-methylisatin α-chloride (etc.) is condensed with a 4-halogeno-α-naphthol to give blue vat dyes fast to light. 5-Chloro-7-methoxy-4-methylisatin α-chloride and 4-chloro-α-naphthol give a greenish-blue. C. HOLLINS.

[Anthraquinone vat] dyes. W. G. WOODCOCK, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,191, 31.1.29).—Vat dyes are purified or improved by treatment with hypochlorite and boric acid, sodium hydrogen carbonate, or similar weakly acidic substances. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone-acridone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,240, 30.5.29).—Trichloroanthraquinoneacridone (annexed formula) is condensed with



amides or sulphonamides, whereby the α-chlorine is replaced by an acylated amino-group, yielding violet to blue vat dyes which may be hydrolysed to give greenish-blue vat dyes. The latter may be re-acylated. Condensations with toluenesulphonamide and benzamide, hydrolysis, and re-acylation with 1-chloroanthraquinone-2-carboxylic chloride, are described. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. SOC. CHEM. IND. IN BASLE (B.P. 319,365, 20.9.29. Switz., 21.9.28).—3:3'-Dibenzanthronyl is nitrated at 10—20° with mixed acid, and the product is fused with alkali to give a blue-grey vat dye. Nitration of this yields a blackish-green vat dye which produces deep black shades on oxidation on the fibre with hypochlorite. C. HOLLINS.

Manufacture of monoazo dyes [for wool etc.]. CHEM. FABR. VORM. SANDOZ (B.P. 312,582, 27.5.29. Ger., 26.5.28).—*m*-Aminobenzenesulphonamides of the type, $\text{NH}_2\text{-Ar-SO}_2\text{NRR'}$, in which Ar carries no nitro-group to the amino-group, and no carboxyl in *o*-position, and R and R' are alkyl, aryl, or aralkyl groups, are diazotised and coupled with 1-sulphoaryl-5-pyrazolones or in acid medium with γ -acid. Examples are: aniline-*m*-sulphonethylanilide, m.p. 78°, $\rightarrow \gamma$ -acid (yellowish-red); *p*-toluidine-2-sulphonethylanilide \rightarrow 1-(2:5-dichloro-4-sulphophenyl)-3-methyl-5-pyrazolone (greenish-yellow). C. HOLLINS.

Manufacture of [insoluble] azo dyes dyeing black tints [pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 311,400, 22.4.29. Ger., 11.5.28).—2:3-Hydroxynaphthoic *p*-2-cymidine (1-methyl-4-isopropyl-2-anilide) is coupled in substance or on the fibre with diazotised *o*-phenetidine $\rightarrow \alpha$ -naphthylamine or *p*-nitroaniline \rightarrow 2:5-dimethoxyaniline to give black dyes. C. HOLLINS.

Manufacture of polyazo dyes. CHEM. FABR. VORM. SANDOZ (B.P. 313,562, 4.6.29. Ger., 14.6.28).—Tetrazo-tised benzidine etc. is coupled with 1 mol. of H-acid in acid medium, and then in alkaline medium with 1 mol. of a diazo compound and 1 mol. of an *N*-alkylated (etc.) *m*-aminophenol, to give blacks for cotton, viscose silk, wool, silk, leather, and mixtures. An example is: aniline \rightarrow H-acid \leftarrow benzidine \rightarrow 3-hydroxy-2'-methyl-diphenylamine. C. HOLLINS.

Solubilisation of a perylenetetracarboxylic diimide dye and dyes produced thereby. A. J. HAILWOOD, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,971, 30.9.30. Appl., 25.7.27. U.K., 19.8.26).—See B.P. 280,647; B., 1928, 83.

Manufacture of aminoanthraquinone derivatives [dyes for cellulose acetate]. O. BALLY and P. GROSSMANN, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,777,953, 7.10.30. Appl., 16.7.28. Switz., 4.8.27).—See B.P. 295,257; B., 1929, 710.

Primary disazo dyes. J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,801, 30.9.30. Appl., 19.4.28. U.K., 28.4.27).—See B.P. 294,922; B., 1928, 741.

Manufacture of colour lakes. L. RUDOLF and B. ZSCHIMMER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,776,931, 30.9.30. Appl., 16.7.26. Ger., 22.7.25).—See B.P. 274,627; B., 1927, 742.

Anthraquinone dyes (B.P. 333,992). Mononitrated benzanthone derivatives (B.P. 333,882).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Down of *Apocynum venetum*. N. L. KARAVAEV and P. N. ODINTZOV (J. Appl. Chem., Russia, 1930, 3, 91—97).—The cellulose content approaches that of straw. The down might serve as a thermal insulator, but is unsuitable for the manufacture of nitrated products. CHEMICAL ABSTRACTS.

Determination of dry weight of wood pulp. B. WIESLER (Papier-Fabr., 1930, 28, 616—618).—A discussion of the difficulties attending the accurate

determination of moisture content in pulp deliveries, and the various commercial customs relating to the expression of "absolute dry" and "air-dry" weights.

T. T. PORTS.

Rosin size manufacture. RIDLEY.—See XIII.

See also A., Oct., 1275, **Lignin and related compounds** (HIBBERT and others). 1303, **Rapid determination of furfuraldehyde** (NOLL and others). 1322, **Sinkage of pulp-wood** (GIBBS). 1325, **Hemicelluloses of jute fibre** (CHOUDHURY and SAHA). **Hemicelluloses of maize cobs** (PREECE).

PATENTS.

Effecting continuous digestion in the extraction of cellulose. J. HOLMES and H. A. KINGCOME (B.P. 333,426, 20.9.29).—A continuous, fractional-digestion process is described, which works on the countercurrent principle and uses, if desired, liquors of different composition in the various pressure stages. The apparatus consists of a number of vertical tubular members, the pressure in each stage being maintained by a balancing column of liquor. The digestion liquor is heated indirectly by steam and the condensate is used in the countercurrent washing of the digested material.

D. J. NORMAN.

Cellulosic compositions. BRIT. CELANESE, LTD. (B.P. 313,134, 7.6.29. U.S., 7.6.28).—Materials containing organic esters of cellulose are rendered less inflammable or non-inflammable by incorporation therein at any convenient stage of one or more aromatic compounds containing bromo-substituted aliphatic side-chains, e.g., benzyl bromide, benzylidene bromide, in the proportion of 2—40 pts. to 100 pts. of the cellulose derivative. F. R. ENNOS.

Manufacture of pulp and paper. L. M. BOOTH (U.S.P. 1,761,069, 3.6.30. Appl., 22.10.27).—After the production of pulp by an alkaline digestion, the usual washing is either dispensed with or is discontinued while an appreciable amount of alkali still remains, and addition is made of an acid coagulating salt (ferrous sulphate, ferric chloride) and of chlorine.

F. R. ENNOS.

Preparation of electrolytic writing and drawing paper and the like. R. HORN (B.P. 313,561, 4.6.29. Ger., 14.6.28).—Paper is impregnated, in some cases with addition of a carrier electrolyte (sodium chloride), with an aqueous or alcoholic, neutral or alkaline solution either of an organic nitroso-, nitro-, amino-, hydroxyl-amino-, or quinone compound, e.g., dimethylglyoxime, or of an organic colouring matter (sodium alizarin-monosulphonate), together with a mordant if required, so that on passage of a current with a soluble anode of silver, copper, lead, iron, nickel, aluminium, or alloys of these metals with each other or with zinc or tin, in the absence of a catalyst, either a sparingly soluble organic metal compound or a lake is produced, whereby the paper is marked. Alternatively, the paper, after treatment with a carrier electrolyte, a catalyst (vanadium pentoxide), and a mordant, is impregnated with an aqueous or alcoholic solution of an organic substance, e.g., aniline, which produces a sparingly soluble colouring matter or lake when oxidised with an insoluble anode or an anode rendered passive. F. R. ENNOS.

Coating paper. W. W. TRIGGS. From CHAMPION COATED PAPER Co. (B.P. 333,226, 3.5.29).—The binding agent consists of raw starch applied to the paper in aqueous suspension in admixture with the other constituents of the coating mixture, gelatinisation being subsequently effected by exposing the wet coated paper to substantially saturated steam. A dispersing agent (gum arabic) is preferably added to the coating mixture.

D. J. NORMAN.

Films or sheets for adhesive purposes. R. ARNOT (B.P. 333,194, 27.2. and 15.3.29).—Synthetic resins in a reactive state are applied to a porous carrier such as paper, fabric, or the like, or are mixed with film-forming solutions, *e.g.*, viscose solution or cellulose ester solutions, without the addition of any volatile solvents other than those which normally result from manufacturing processes, *e.g.*, those present in cellulose lacquers. Softening agents, metal powders, fireproofing agents, or other adhesives may be incorporated.

D. J. NORMAN.

[Spinning pot for] production or treatment of artificial threads or other textile yarns, *e.g.*, rayon, by the centrifugal spinning process. J. L. RUSHTON, H. HILL, and D. L. PELLATT (B.P. 335,049, 11.9.29).

Spinning centrifuges for artificial silk. A. E. O'DELL. From BERLIN-KARLSRUHER INDUSTRIEWERKE A.-G. (B.P. 335,307, 16.7.29).

Paper pulp and like beating engines. S. MILNE (B.P. 335,671, 26.7.29. Addn. to B.P. 207,716).

Carbohydrate compounds (B.P. 334,282).—See III. **Rubber goods etc.** (B.P. 335,271). **Rubber-paper products** (U.S.P. 1,756,035).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Kinetics of chlorine bleaching. H. KAUFFMANN (Z. angew. Chem., 1930, 43, 840—843).—Assuming that the active substance in bleaching liquors is free hypochlorous acid formed by the hydrolysis of hypochlorite, the velocity coefficient of the transformation of the ClO' ion (a reaction of the second order) is a measure of the bleaching power of the solution. It is shown that this coefficient must be inversely proportional to the hydroxyl-ion concentration, and this is in accord with the observation that bleaching power increases with increasing acidity. Since the destructive effect on cotton fibre increases with the alkalinity, it follows that neither hypochlorous acid nor chlorine monoxide can be responsible. It is assumed that the fibre is attacked by an unstable complex ion $[\text{ClOH}, \text{ClO}]'$, and a calculation involving the equilibrium constants of the dissociation of hypochlorous acid and of the hydrolysis of chlorine shows that the destructive effect should be at a maximum when the ratio $\text{Cl}_2 : \text{NaOH}$ is 1 : 1.5. This is in agreement with observation. F. L. USHER.

Liability of dyed wool to mildew with special reference to the resistance resulting from chroming. R. BURGESS (J. Text. Inst., 1930, 21, T441—452).—Samples of wool dyed under various conditions with and without addition of chromate were inoculated with mould fungi, maintained under conditions favourable

to mould growth, and their liability to mildew was examined. The solubility of chromed and unchromed wool in boiling water, and in an Alizarin Red dye-bath, the effects of various concentrations of potassium dichromate on mildew development on unscoured yarn, and enzyme activity on chromed and unchromed wool have also been investigated. From the results obtained it is concluded that the process of chroming imparts to wool a considerable resistance to the growth of mould fungi, which is due to impoverishment of the wool as a source of the nutriment required by the organisms. Thorough washing-off after dyeing or scouring reduces the liability to mildew.

B. P. RIDGE.

Potentiometric control of the degree of mercerisation of cellulose. S. N. USHAKOV and A. D. SOKOLOV (J. Appl. Chem., Russia, 1930, 3, 31—46).—Electrometric titration of the liquors indicates that the rate of circulation is important in shortening the reaction time.

CHEMICAL ABSTRACTS.

Colloid chemistry of laundering. L. ZAKARIAS (Rev. gén. Colloid., 1930, 8, 112—120).—A review of published work on the protective effect, surface tension, and foaming power of soaps and their relation to the detergent effect. The detergent action of soap is due to the peptisation and swelling of the colloidal impurities, which combine with the soap to form a mixed colloid, soluble in water and readily removed by rinsing. The addition of fats, sulphonated oils, or polysaccharides does not increase the detergent action of soap. The presence of polysaccharides protects soap from the action of calcium salts, so that soaps containing polysaccharides have simultaneously a softening and a detergent effect.

E. S. HEDGES.

Detergent power of soaps. BOSSHARD and STURM.—See XII.

See also A., Oct., 1260, Effect of light on dyes (KRAIS). 1309, Fastness of dyes to human perspiration (VASS and McSWINEY).

PATENTS.

Dyeing of animal fibres. DURAND & HUGUENIN Soc. ANON. (B.P. 334,508, 27.5.29. Addn. to B.P. 218,649; B., 1925, 708).—Dyeings fast to rubbing are obtained with esters of leuco-vat dyes on animal fibres by adding to the acid oxidising bath used for development an emulsifying agent which remains soluble in an acid medium ("Supamines," or a sulphonated fatty acid).

F. R. ENNOS.

Application of vat dyes to textile fibres. A. R. THOMPSON, JUN., ASSR. to ROHM & HAAS Co. (U.S.P. 1,759,071, 20.5.30. Appl., 27.2.26).—Streaky or uneven dyeing is prevented by addition of sodium formaldehyde-sulphoxylate to the alkaline dye bath reduced with sodium hyposulphite.

F. R. ENNOS.

Coloration of textile materials. BRIT. CELANESE, LTD. (B.P. 308,173, 18.3.29. U.S., 17.3.28).—Yarns, particularly those containing cellulose derivatives, are tinted with fugitive colours by contact with a wick or pad moistened with a solution of a dye in di- or polyhydric alcohols or compounds having a di- or polyhydric alcohol base of b.p. above 100°. Suitable solvents include ethylene glycol, diethylene glycol, and their

mono- or di-ethyl ethers. Since these compounds are hygroscopic it is preferred to add an amount of water equal to that which would be absorbed from the air during use; this addition prevents gradual subsequent changes in viscosity and volume. A. J. HALL.

Coloration of textile materials. BRIT. CELANESE, LTD. (B.P. 308,733, 27.3.29. U.S., 27.3.28).—Filaments, yarns, etc. are temporarily coloured, continuously with their production, by passing over a wick, pad, or roller supplied with a tinting fluid consisting of a fugitive colouring matter, a non-drying animal, vegetable, or mineral oil, and a sulphonated oil.

F. R. ENNOS.

Coloration of [artificial silk] textile filaments, yarns, threads, etc. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,565, 11.1.29. Cf. B.P. 332,263; B., 1930, 944).—Artificial silk yarns composed of cellulose or its esters or ethers are coloured intermittently with fast dyes at regular or irregular spaced intervals along their length by means of suitable devices which rotate or oscillate, or reciprocate in contact with the travelling yarn.

A. J. HALL.

Production of coloured resists under Aniline Black. I. G. FARBENIND. A.-G. (B.P. 316,316, 26.7.29. Ger., 28.7.28).—The fabric, padded as for Aniline Black, is printed with a paste containing zinc oxide, large excess of caustic alkali, and a mixture of a non-nitrated nitroso-amine alkali salt and a coupling component such as a 2:3-hydroxynaphthoic or acetoacetic arylamide. The fabric is steamed, whereby the Aniline Black is developed but not the azo colour, and then passed successively through an acetic acid bath and a chromate bath to develop the coloured resist. The production of yellow, orange, scarlet, and red resists free from aureole is described.

C. HOLLINS.

Production of thickening materials for use in printing [of textile fabrics]. H. BOLLMANN and B. REWALD (B.P. 333,959, 28.5.29).—A mixture of one or more of thickening agents, such as dextrin, gum tragacanth, starch, or gum arabic, with a solution of vegetable or animal phosphatides in a mineral oil is claimed. Suitable phosphatides are extracted from ground soya beans by means of benzene-alcohol (9:1), the solvent being afterwards removed by distillation and the phosphatides precipitated from the residual oil by blowing in steam; soya-bean oil adhering to the precipitated phosphatides is largely removed by centrifuging.

A. J. HALL.

Printing on wool [with leuco-vat dye esters]. I. G. FARBENIND. A.-G. (B.P. 334,419, 11.10.29. Ger., 11.10.28. Addn. to B.P. 298,648; B., 1929, 1012).—Wool is dyed with a dischargeable colour, and is then printed with leuco-vat dye ester and a reducing agent (formaldehydesulphoxylate), steamed, and developed with acid and persulphate. Coloured discharge effects are obtained.

C. HOLLINS.

Manufacture of artificial silk [of matte lustre] from viscose. I. G. FARBENIND. A.-G. (B.P. 312,687, 6.5.29. Ger., 31.5.28).—Subdued lustre is imparted to viscose silk by emulsifying with the viscose solution not less than 0.5% (calc. on the viscose) of one or more

organic bases (excluding aniline) which are more or less insoluble in water, e.g., quinoline, dimethylaniline, toluidine.

D. J. NORMAN.

Production or treatment of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., W. I. TAYLOR, R. P. ROBERTS, and L. W. GREGORY (B.P. 334,563, 28.3.29).—Materials of reduced or subdued lustre are produced by dry-spinning solutions of cellulose esters or ethers containing a metallic (barium) salt, and subsequently treating the formed filaments with a fixing solution to precipitate therein an opaque, insoluble metallic compound (barium sulphate or phosphate).

F. R. ENNOS.

Improvement of fabrics made from or containing vegetable fibre. HEBERLEIN & Co. A.-G. (B.P. 334,756, 14.10.29. Ger., 12.10.28).—A linen-like appearance is imparted to the fabric, without loss of its natural softness, by pressing the fabric above 100° and then treating with a swelling agent (mineral acids, concentrated alkali lye with or without cooling, concentrated zinc chloride or ammoniacal copper oxide solutions).

F. R. ENNOS.

Waterproofing of fabrics. J. W. MACKENZIE (B.P. 334,020, 16.7.29).—A waterproofing composition, applicable to canvas fabric by painting is prepared by heating together a mixture of flour, water, sodium carbonate, and tallow or of a heavy mineral oil, and adding rosin; a sizing agent such as gum arabic may be added before use. The composition dries on the fabric within 1 day.

A. J. HALL.

Treating and ageing of fabrics. E. S. CHAPIN and A. H. JACOBY (B.P. 334,069, 29.8.29).—The steam supplied to the ageing chamber is admitted at successive stages in such manner that although heat is generated locally within the fabric (printed with vat dyes) due to chemical reactions, its temperature is maintained constant within satisfactory limits.

A. J. HALL.

Treatment of casein-varnish-finished coated fabrics or articles. M. N. NICKOWITZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,746,162, 4.2.30. Appl., 23.11.27).—Sheeting or fabric surfaced with rubber (or even with pyroxylin or oxidised oil) is treated with a casein varnish. After vulcanisation by dry heat the surface is treated with a 4% solution of bromine in carbon tetrachloride, the varnish thereby being softened and rendered smooth without affecting the under layer of rubber.

D. F. TWISS.

Dyeing [of regenerated cellulose]. J. BADDILEY, R. BRIGHTMAN, and P. CHORLEY, Assrs. to BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,776,802, 30.9.30. Appl., 4.6.28. U.K., 7.6.27).—See B.P. 296,485; B., 1928, 855.

[Dyeing of "mixed" cellulose acetate] threads, cords, and fabrics. C. W. PALMER and S. M. FULTON, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,777,445. 7.10.30. Appl., 16.6.27. U.K., 21.7.26).—See B.P. 278,116; B., 1927, 905.

Apparatus for cleaning liquid solvents used for dry-cleaning and other purposes. J. H. BEAUMONT. From R. R. STOLLEY CORP. (B.P. 334,208, 27.5.29).—See U.S.P. 1,711,829; B., 1929, 895.

Bleaching or otherwise treating fibrous material. B. P. VON EHRENTHAL and K. SCHOLZ (B.P. 316,151, 23.7.29).

Washing machines. E. A. ALLIOTT, and MANLOVE, ALLIOTT & Co., LTD. (B.P. 335,028, 22.8.29). R. S. LANGE (B.P. 334,906, 13.4.29).

Centrifugal extractor (U.S.P. 1,760,775).—See I. Fatty acid esters [as wetting agents] (B.P. 315,832). Cleaning solvents (B.P. 333,217).—See III.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Chemistry of leaching chalcocite. J. D. SULLIVAN (U.S. Bur. Mines, Tech. Paper 473, 1930, 24 pp.).—Chalcocite (Cu_2S) can be leached with ferric sulphate with the production of copper sulphate and ferrous sulphate. Neither air with water alone nor ferrous sulphate dissolves the mineral, but sulphuric acid with access of air will dissolve it at a slower rate than ferric sulphate solution. The rate of dissolution of chalcocite is independent both of the concentration of ferric sulphate, provided enough reagent is present, and of the acid concentration, though a slight excess of acid is desirable as it prevents the formation of basic iron compounds. Increase in temperature greatly accelerates the reaction; *e.g.*, with a solution containing 0.5% Fe as ferric sulphate and 0.5% H_2SO_4 , 73% of the copper was dissolved in 1 day at 50°, in 5 days at 35°, and in 15 days at 23°. Variation in size of particles from 10- to < 200-mesh had little influence on the rate of dissolution if the particles were freely in contact with the solution. Products of the reaction do not interfere until large quantities of ferrous sulphate appear in the solution. When leached with ferric sulphate one half of the copper dissolves rapidly, leaving the residue with an approximate formula, CuS , but of entirely different properties from those of covellite. This compound is attacked at a much slower rate. (Cf. following abstract.) C. A. KING.

Innovations in copper leaching employing ferric sulphate-sulphuric acid. H. E. KEYES (U.S. Bur. Mines, Bull. 321, 1930, 67 pp.).—The leaching solution containing ferric sulphate and sulphuric acid is produced by autoxidation of ferrous sulphate and sulphur dioxide in the presence of air. The first reaction is to oxidise the iron and then to produce sulphuric acid, but intimate contact of air and solution is desirable. Most copper minerals, *e.g.*, chalcocite, bornite, and the oxidised forms of mineral, are amenable to leaching with acidified ferric sulphate, exceptions being chalcopyrite and certain silicates. Copper is precipitated from solution by means of sponge iron. Results of plant tests on different ores by open drainage and flood-leaching are described. (Cf. preceding abstract.) C. A. KING.

Heat transfer. OTHMER.—See I. Organic nitrogen from agricultural waste. DIEGO. Sulphur from coal gas. PETT.—See II. Action of acids on non-rusting steels. SAUVAGEOT and LAUPRÉTE. Titration of potassium cyanide. SANIGAR.—See X. Measuring and recording p_{H} . WULFF and KOR-

DATZKI.—See XI. Ammonia from beet juice. ZAMARON.—See XVII. Base-exchange materials. MARTIN.—See XXIII.

See also A., Oct., 1258, Sputtered nickel films and the synthesis of ammonia (INGERSOLL). 1277, Determination of metal in organic compounds incapable of electrolysis (DOSIOS and PIERRI).

PATENTS.

Production of chemically pure phosphoric acid. I. G. FARBERIND. A.-G. (B.P. 334,790, 18.11.29. Ger., 20.2.29. Addn. to B.P. 319,656; B., 1930, 660).—The efficiency of the prior process is improved by electrolysis at 50° or above. When the crude acid contains insufficient heavy-metal salt small amounts of a copper salt, preferably copper phosphate, are added in order to facilitate the deposition of arsenic. The solution may contain 1 pt. of copper to at least 2 pts. (preferably 2.5–4 pts.) of arsenic. S. K. TWEEDY.

Digestion of phosphate rock. W. H. LOHMANN, ASSR. to GEN. CHEM. Co. (U.S.P. 1,747,588, 18.2.30. Appl., 17.5.27).—In the digestion of phosphate rock with liquor obtained by washing mud produced in a previous digestion and adding sulphuric acid and nitre cake to the resulting liquid, the precipitation of glauconite (calcium sodium sulphate) is prevented by keeping the liquor below 66° throughout the digestion. S. K. TWEEDY.

[Continuous] production of dicalcium phosphate. A. HOLZ and T. V. D. BERDELL (B.P. 335,007, 30.7.29).—A solution of an alkali chloride is electrolysed, the liberated hydrogen is collected separately, and the chlorine passed into water into which an equivalent amount of sulphur dioxide is simultaneously introduced. The mixture of hydrochloric and sulphuric acids obtained is caused to react with crude phosphate rock, the solution, after filtration, being treated with the alkali hydroxide, derived from the electrolysis, and calcium hydroxide, and the precipitated dicalcium phosphate removed by filtration. The mother-liquor containing alkali chloride is utilised for the electrolysis. Alternatively, after treatment of the phosphate rock and filtration, the filtrate may be concentrated and caused to react with additional phosphate rock, and the solution of monocalcium phosphate precipitated with alkali hydroxide alone. W. J. WRIGHT.

Manufacture of dithiophosphates. C. T. ROMIEUX and H. P. WOHNSEDLER, ASSRS. to AMER. CYANAMID Co. (U.S.P. 1,748,619, 25.2.30. Appl., 14.11.27).—Phosphorus pentasulphide is treated with an organic hydroxy-compound (*e.g.*, an aliphatic alcohol such as isopropyl alcohol). A solvent, such as solvent naphtha or other hydrocarbon, is then added to the crude product, after which an amine (*e.g.*, aniline or ammonia) is added and the resulting pure dithiophosphate of the amine is filtered off. Alternatively, the crude product is dissolved in an alkaline substance (*e.g.*, sodium carbonate solution), the aqueous layer formed being removed and treated with acid in order to regenerate the dithiophosphoric acid in the pure state. The acid is then dissolved in a solvent, such as solvent

naphtha or water, and a dithiophosphate precipitated therefrom by addition of an amine, a base, or a metal salt.

S. K. TWEEDY.

Manufacture of bleached white barytes. W. J. O'BRIEN (U.S.P. 1,758,009, 13.5.30. Appl., 28.5.27).—The ground barytes (1–10 pts.) is fused and roasted with nitre cake (1 pt.) in an oxidising atmosphere, up to 0.1 pt. of an oxidising agent (*e.g.*, sodium nitrate, potassium chlorate) being added if desired; the molten mass when passed into water preferably containing a reducing agent (sodium sulphite) yields a precipitate of bleached barytes. The nitre cake may be replaced by sodium chloride up to the extent of 50%.

S. K. TWEEDY.

Manufacture of barium carbonate. B. LAPORTE, LTD., I. E. WEBER, and V. W. SLATER (B.P. 334,709, 5.9.29).—A barium compound containing sulphur (*e.g.*, the sulphide or hydrogen sulphide) is added to a solution of carbon dioxide (under pressure, if desired) or barium hydrogen carbonate in such proportion that carbonic acid or barium hydrogen carbonate is always present, or that the p_H value of the solution is always less than 7. The process may be made continuous by simultaneously adding carbon dioxide and barium sulphide to water; the gas need not be free from oxygen.

S. K. TWEEDY.

Production of gels. Preparation of zeolites. W. W. TRIGGS. From GEN. ZEOLITE Co. (B.P. 334,676—7, 27.7.29).—(A) Small proportionate amounts of two concentrated reacting solutions are mixed in coalescing flow; the flow is continued in order to effect thorough admixture and the fluid is then collected in a receiving tank where the gel is allowed to develop. Preferably the two solutions come into contact as baffled jets. The method may be employed to produce gel zeolites, and is particularly adapted for use with solutions which react rapidly to form thick gelatinous precipitates. The operations may be carried out at normal temperature. (B) An alkali silicate solution containing about 0.25–2 mols. of silica per mol. of alkali oxide is combined with a solution of an amphoteric metal hydroxide containing 1–1.25 acid equivalents to each equivalent of amphoteric metal oxide. The gel formed is dehydrated, preferably below 100°, and thoroughly washed. The silicate solution may be obtained by mixing, during or before the zeolite-forming reaction, a soluble silicate and a non-zeolite-forming alkaline substance; or the amphoteric oxide solution by mixing a soluble amphoteric metal compound and a non-zeolite-forming acidic substance. In an example, sodium silicate, aluminium sulphate, sodium hydroxide, and sulphuric acid are caused to interact in admixture.

S. K. TWEEDY.

Manufacture of aluminium alcoholates containing aluminium chloride. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 334,820, 7.1.30. Ger., 13.6.29).—A mixture of aluminium and an alcohol is treated, if desired, at elevated temperature, and preferably in presence of an indifferent solvent such as ethyl acetate, with dry chlorine, or with a chlorine compound (*e.g.*, hydrochloric acid) capable of imparting chlorine to aluminium. Excess of aluminium is essential if the alkoxide produced is to be free from

alcohol of crystallisation. The product actively catalyses the conversion of acetaldehyde into ethyl acetate.

S. K. TWEEDY.

Manufacture of metal carbonyls. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,976, 24.6.29 and 10.3.30).—Carbon monoxide is passed in succession through a series of reaction vessels, the carbonyl formed in any vessel being condensed from the gas stream before it enters the next vessel. Fresh supplies of carbon monoxide are admitted first to the vessel in which the reacting material is almost exhausted and the gas stream almost denuded of carbon monoxide issues from the vessel containing the freshest reaction materials.

A. R. POWELL.

Manufacture of hydrogen. A. T. LARSON, ASSR. to LAZOTE, INC. (B.P. 311,737, 14.5.29. U.S., 15.5.28).—The reaction of steam with carbon monoxide at temperatures above 300° is accelerated by employing catalysts containing copper and an oxide of zinc, tungsten, molybdenum, cerium, vanadium, manganese, uranium, chromium, or magnesium.

W. J. WRIGHT.

[Electrolytic] manufacture of chlorine. F. S. LOW, ASSR. to WESTVACO CHLORINE PRODUCTS, INC. (U.S.P. 1,746,542, 11.2.30. Appl., 25.10.28).—Hydrochloric acid is electrolysed between submerged electrodes, the hydrogen chloride content of the solution being replenished, *e.g.*, by introducing gas below the surface of the electrolyte, preferably at a point which is so much below the surface that the gas introduced is almost completely absorbed. A cell for carrying out the process is described.

S. K. TWEEDY.

Vanadium catalysts for use in the oxidation of oxidisable gases [sulphur dioxide]. S. ROBSON and P. S. LEWIS (B.P. 334,862, 8.6.29).—A mixture of dried silica gel with 5–10% of vanadium pentoxide, ammonium vanadate, or an alkali vanadate and a small quantity of a promoter such as stannous chloride is bonded with sodium hydroxide or silicate and with a vegetable gum to form a thick paste which after partial drying is made into pellets. These are then treated cold with a stream of sulphur dioxide until they show an acid reaction, heated to 400° in a stream of hot air, and transferred to the catalyst chamber for the manufacture of sulphuric acid by the contact process.

A. R. POWELL.

Manufacture of carbon disulphide. H. OEHME, and CHEM. FABR. KALK G.M.B.H. (B.P. 314,060, 5.6.29. Ger., 23.6.28).—Hydrogen sulphide, absorbed in ammonia liquor from coal gases and liberated by heat or by a current of carbon dioxide, is passed over charcoal at 900–1000° at such a rate that 70% of the sulphur is converted into carbon disulphide; the unused hydrogen sulphide is absorbed in ammonia liquor and returned to the circuit.

C. HOLLINS.

Manufacture of carbon disulphide. H. OEHME, and CHEM. FABR. KALK G.M.B.H. (B.P. 334,856, 5.6.29. Ger., 2.10.28).—In the process described in B.P. 314,060 (preceding abstract), after removal of the reaction products when only partial decomposition of the hydrogen sulphide has taken place, the remaining hydrogen sulphide, separated from the hydrogen, if desired, is used for producing sulphur or its compounds

other than carbon disulphide. Or the mixture of hydrogen and hydrogen sulphide may be returned to the crude gas, so that recovery of undecomposed hydrogen sulphide is effected at the same time as the recovery of the hydrogen sulphide from the crude gas itself.

W. J. WRIGHT.

Preparation of phosgene [carbonyl chloride].

R. WILLIAMS, ASSR. to DU PONT AMMONIA CORP. (U.S.P. 1,746,506, 11.2.30. Appl., 4.10.27).—Carbon monoxide and nitrosyl chloride are allowed to interact, preferably in presence of a catalyst comprising activated carbon and within the temperature range of 100–250°.

S. K. TWEEDY.

Manufacture of phosphoric acid. S. G. NORDEN-GREN, ASSR. to AKTIEB. KEMISKA PATENTER (U.S.P. 1,776,595, 23.9.30. Appl., 12.4.29. Ger., 7.7.28).—See B.P. 314,976; B., 1930, 861.

Manufacture of ammonium chloride. J. W. MOORE and W. G. POLACK, ASSRS. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,776,698, 23.9.30. Appl., 1.6.27. U.K., 25.6.26).—See B.P. 274,263; B., 1927, 701.

Manufacture of iron carbonyl compositions. A. MITTASCH and M. MÜLLER-CUNRADI, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,920, 30.9.30. Appl., 22.4.25. Ger., 5.12.24).—See Can.P. 262,601; B., 1927, 403.

Fixation of atmospheric nitrogen. J. BRIGHT-MORE (U.S.P. 1,776,308, 23.9.30. Appl., 23.6.27. U.K., 6.7.26).—See B.P. 283,232; B., 1928, 192.

Sulphur compounds from gases (B.P. 305,027).—See II. Abrasive (U.S.P. 1,747,579).—See VIII. Extracting iron from ores (U.S.P. 1,759,456). Metal netting as catalyst (B.P. 334,466).—See X. Water-purifying material (U.S.P. 1,757,372).—See XXIII.

VIII.—GLASS; CERAMICS.

Chemical and mineralogical character of stalactites from two glass works. D. S. BELIAKIN (Bull. Acad. Sci. U.S.S.R., 1930, 257–265).—Stalactites formed in the furnaces of two glass works have been examined and the chemical natures of dinas and glass compared. Chemically the two stalactites differ only in their lime and magnesia contents. Mineralogically, whilst one appears to consist of tridymite, cristobalite, and glass, the other consists of tridymite and pseudowollastonite.

A. FREIMAN.

Translucency and particle size in white enamels containing stannic and zirconium oxides. C. J. VAN NIEUWENBURG and J. SCHOTSMAN (Trans. Ceram. Soc., 1930, 29, 342–351).—To test the truth of the supposition that the opacity of enamels increases with decreasing particle size, provided that the latter remains about one half to one fourth of the wave-length of the kind of light used, the opacifying power was measured and curves for the complete cumulative distribution of three brands of tin oxide and two of zirconium oxide were obtained by the use of an ordinary visual photometer. Results show the above hypothesis to

be incorrect. It is concluded that a test of particle size is by no means sufficient as a means of predicting opacifying power.

R. J. CARTLIDGE.

Earthenware glaze preparation. A. TEICHFELD (Trans. Ceram. Soc., 1930, 29, 339–341).—In Poland, and, in general, on the Continent, the frit in the mill mixture of earthenware glazes varies between 65% and 80%. This necessitates a fritting operation once in every six weeks, the kiln being run for a whole week. When the content of frit in the mixture was reduced from 76% to 48%, fritting was then only necessary once in every three months, and the melted frit ran so easily that for trials the well-known perforated crucible was used.

R. J. CARTLIDGE.

Tenriuji yellow Celadon glaze. T. ISHII (Trans. Ceram. Soc., 1930, 29, 352–359).—Nine base glazes within the range $0.2K_2O$, $0.8CaO$, $0.3-0.5Al_2O_3$, $3.0-4.0SiO_2$ were prepared to find some which would be similar to the Tenriuji Celadon in lustre, transparency, and colour. The colorants used contained 3.0–3.5 pts. of ferrous silicate and 0.01–0.07 pt. of chromic oxide, added to 100 pts. of the batches. Experiments were conducted on glazes containing rutile and manganous carbonate as auxiliary colorants. The secret of producing this glaze is shown to lie in the composition of the base glaze and of the body, and in the nature of the kiln atmosphere during firing.

R. J. CARTLIDGE.

Kinuta blue Celadon glaze. T. ISHII (Trans. Ceram. Soc., 1930, 29, 360–387).—Firing is most important in conducting experiments on Celadon glazes, since the colours are strongly affected by the nature of kiln atmosphere. Test pieces were fired in a wood-fired laboratory kiln with reducing flame to about 1300° for 18–26 hrs. Results show the best base glazes for Kinuta Celadon ware are those which are more siliceous than porcelain glazes and tend to be devitrified. The felspar content should be high enough to prevent rapid melting, but not so high that crazing results. The best proportion for the bases is $0.5K_2O$ to $0.5CaO$, or $0.6K_2O$ to $0.4CaO$.

R. J. CARTLIDGE.

X-Ray examination of kaolins and clays. F. KÄSTNER and F. K. MAYER (Sprechsaal, 1930, 63, 118–120; Chem. Zentr., 1930, i, 2466).—Chemical and X-ray analysis showed that kaolinisation of granite buried for 1 year in garden soil had occurred. The clays and kaolins examined contained the same mineral (kaolin).

A. A. ELDRIDGE.

Firing of ceramics and faults due to firing. P. BREMOND (Trans. Ceram. Soc., 1930, 29, 330–338).—The bodies examined were fired slowly to given temperatures, and after each firing the vitrification was determined. Curves show that the hard porcelains are not readily vitrified until about 1200°, whereas felspar porcelains begin to vitrify at about 1050–1100°. Carburisation by carbon monoxide, methane, ethylene, and town's gas is discussed. Faults in all porcelain and stoneware bodies may be created or removed by carburising and decarburising these bodies at a stage in the firing below the 5% porosity point or above this limit. (Cf. B., 1927, 141.)

R. J. CARTLIDGE.

Influence of oxidising and reducing atmospheres on refractory materials. III. Experiments with a refractory mixture containing added amounts of colloidal ferric hydroxide. A. E. J. VICKERS (Trans. Ceram. Soc., 1930, 29, 290—298; cf. B., 1928, 570).—Refractory cones containing carbon and colloidal ferric hydroxide were heated in atmospheres of nitrogen, oxygen, carbon monoxide, carbon dioxide, sulphur dioxide, hydrogen, air, and water vapour and the effects were noted. An inspection of the graph shows that water vapour produces the greatest decrease in refractoriness, but such effect for any gas depends on the amount of ferric oxide added.

R. J. CARTLIDGE.

Formation of mullite in refractories. P. P. BUDNIKOV and B. A. KHIZH (J. Appl. Chem., Russia, 1930, 3, 21—24).—A furnace lining originally containing SiO_2 57—58, Al_2O_3 36.4—37.3, Fe_2O_3 1.5—2, CaO 1.5—1.9%, after six months' service in a coal-fired furnace contained SiO_2 49.38, Al_2O_3 28.54, Fe_2O_3 20.82, CaO 0.91%. Crystals of mullite with embedded crystals of ferrous oxide were present. Under certain conditions the formation of mullite is favoured by the presence of iron compounds.

CHEMICAL ABSTRACTS.

Advances in the decorative arts as applied to ceramics. F. H. RHEAD (Trans. Ceram. Soc., 1930, 29, 388—400).

Pan grinding. MELLOR.—See I.

See also A., Oct., 1241. **Silicate system based on crystal structure** (VON NÁRAY-SZABÓ).

PATENTS.

Colouring of clay. R. T. VANDERBILT, Assr. to R. T. VANDERBILT CO., INC. (U.S.P. 1,755,616, 22.4.30. Appl., 5.5.27).—The clay, which may be previously dried, is sprayed, in lump or granule form, and while hot from the drying operation, if desired, with a solution of a dye, and is then pulverised and thoroughly mixed.

S. K. TWEEDY.

Manufacture of [non-efflorescing] bricks. A. BONNINGTON, Assr. to ALTON BARIUM PRODUCTS CO. (U.S.P. 1,758,026, 13.5.30. Appl., 14.7.27).—As raw materials brick clay together with natural barium sulphate, or the residue from roasted or reduced barytes ore from which, if desired, the water-soluble ingredients have been extracted, are employed. The proportions of the admixture are preferably such that the water-soluble salts contained in the clay are converted into insoluble salts.

S. K. TWEEDY.

Manufacture of [abrasive] material comprising anhydrous calcium sulphate and calcium pyrophosphate. C. B. DURGIN and P. LOGUE, Assrs. to FEDERAL PHOSPHORUS CO. (U.S.P. 1,747,579, 18.2.30. Appl., 16.11.25).—To hot sulphuric acid (d 1.06—1.07) is added crushed phosphate [of lime] (e.g., calcined, steamed bone), and, after stirring for several hours and adding milk of lime if desired, the precipitate of calcium sulphate and dicalcium phosphate is collected and calcined. The product (consisting of interlaced crystals) preferably contains about 15% P_2O_5 .

S. K. TWEEDY.

Bleaching or decolorising of clay or other

minerals. T. W. PARKER, Assr. to DARTMOOR CHINA CLAY CO., LTD. (U.S.P. 1,777,446, 7.10.30. Appl., 25.2.28. U.K., 28.2.27).—See B.P. 279,694; B., 1928, 15.

Manufacture of non-splintering laminated glass sheets, other compound inter-adherent laminated sheets, and the like. W. C. DOUGAN (B.P. 334,858, 6.6.29).

Manufacture of pottery ware. W. J. MILLER (B.P. 310,961, 22.4.29. U.S., 5.5.28).

IX.—BUILDING MATERIALS.

Fundamental synthesis of calcium aluminates and their hydration. VII. S. NAGAI and R. NAITO (J. Soc. Chem. Ind., Japan, 1930, 33, 315—318 B).—Heating and compressive strength tests have been carried out in continuation of previous work (A., 1930, 436).

C. W. GIBBY.

PATENTS.

Manufacture of cementitious material of cellular structure. G. B. HINTON (U.S.P. 1,756,789, 29.4.30. Appl., 24.10.27).—A mixture of cement pulp and a frothing agent is aerated and agitated to form a froth of small uniform bubbles coated with cement (cf. U.S.P. 1,657,716; B., 1928, 216); bubbles of larger size are then incorporated in the froth so that the product contains bubbles of air coated with a film of cement having smaller bubbles of air embedded therein.

S. K. TWEEDY.

Seasoning of wood [prior to impregnation]. F. S. SHINN (U.S.P. 1,763,070, 10.6.30. Appl., 15.10.27).—The wood, contained in a retort, is successively treated with steam under raised pressure, e.g., at 20 lb./in.², treated with compressed air at, e.g., 80 lb./in.², and subjected to a high vacuum. The first and second treatments may be reversed and the various processes repeated if desired.

L. A. COLES.

Preservation of wood. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,202, 28.5.29).—Wood is impregnated with a hydroxyalkylamine salt of a nitrated phenol, e.g., mono- or di- β -hydroxyethylaniline, hydroxy-ethylamine or -propylamine, etc. salts of 2:4-dinitrophenol or 3:5-dinitro-*o*-cresol. The salts may be applied together with fluorides, hexamethylenetetramine, etc.

C. HOLLINS.

Production of [heat- and sound-insulating] organic material. E. C. BAYER (U.S.P. 1,777,247, 30.9.30. Appl., 31.1.27. Denm., 9.2.26).—See B.P. 265,968; B., 1928, 299.

Cold bituminous paving composition. O. H. BERGER, Assr. to W. P. McDONALD CONSTRUCTION CO. (U.S.P. 1,776,379, 23.9.30. Appl., 13.7.27).—See B.P. 293,830; B., 1928, 750.

[Concrete] mixing machine. P. P. YONE, Assr. to KOEHRING CO. (U.S.P. 1,760,101, 27.5.30. Appl., 9.4.27).

Fireproof or soundproof floors or ceilings. RHEOCRETE PUMICE STONE SLAB CO., LTD., and D. WHITAKER (B.P. 334,914—5, 12.6.29).

Drying of timber (U.S.P. 1,760,444).—See I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

High-frequency steel furnaces. D. F. CAMPBELL (Iron and Steel Inst., Sept., 1930. Advance copy. 10 pp.).—At present the largest furnaces of the Ajax-Northrup type are of 20–25 cwt. capacity per heat, giving an output of 20 tons of steel per day; there appears no technical difficulty in constructing larger units of 3–5 tons, or even 25 tons, though the latter might not be economically practicable. The rapidity of melting and absence of contamination are discussed with reference to the higher-melting alloys, and costs have been reduced by the greater efficiency of motor-generator sets. C. A. KING.

Open-hearth furnace steelworks. Comparison of British and Continental installations and practice. H. C. WOOD (Iron and Steel Inst., Sept., 1930. Advance copy. 32 pp.).—Production of steel in Great Britain is almost entirely due to the Siemens-Martin process, whilst on the Continent this process has a strong competitor in the basic Bessemer converter, which results in every incentive to improve practice to be applied. The outputs in Continental practice are generally in advance of those obtaining in Great Britain, and great importance has been placed on large tonnage outputs per unit at low fuel consumption, low repair and labour costs, and the general ease of administrative and scientific control. Modern mechanical equipment is almost universally employed, and every encouragement is offered to the introduction of improvements in design or method. C. A. KING.

What reasons compelled the Prague Ironworks Company to introduce thin-walled blast-furnaces? J. SARE (Iron and Steel Inst., Sept., 1930. Advance copy. 33 pp.).—The theory of blast-furnace working is discussed and in connexion with the irregular working and severe corrosion of the walls it was observed that places where the lining had been melted off to only a few cm. thick would last well for a comparatively long time. Five furnaces of the thin-walled type have been erected on practically the original lines, and have been eminently successful, as the lining has a long life, troubles in operation have been decreased, and the consumption of coke has been reduced contrary to general conceptions. The original thickness of the lining of the latest furnace was 250 mm., and after operating for 18 months the lining was 180–200 mm. thick in the middle of the shaft and 120–140 mm. in the belly. It was expected that after attaining this equilibrium this thickness would probably remain for a long period undergoing merely mechanical wear. The only disadvantage is the increased consumption of cooling water. Construction costs are not higher than those of the older type, as the decreased firebrick covers the extra cost of armouring the furnace. C. A. KING.

Stock distribution and gas-solid contact in the blast furnace. C. C. FURNAS and T. L. JOSEPH (U.S. Bur. Mines, Tech. Paper 476, 1930, 73 pp.).—The relative inefficiency of the blast furnace is due in part to poor contact between the gas stream and the ore, and this in turn depends largely on the difference in size of the

material in different parts of the furnace. Size segregation in a furnace is due to the greater ore-coke ratio at the centre caused by the greater angle of repose of the ore. The average particle size at the centre may be decreased by reducing the time taken to deposit the materials on the bed, *e.g.*, by keeping the stock line close to the bed and by alteration in the bell discharge. Conversely, the size at the centre is increased by reversed filling, alternating skips of ore and coke, and by decreasing the bell clearance. Material finer than 14-mesh is potential flue dust, and as about 25% of this is lost it would be more economical to separate the fine portions and sinter it before introduction into the blast furnace. It is also desirable to crush all material to pass 2-in. mesh. At one plant about 20% of fuel has been saved by crushing and grading the charge into three different sizes, and charging each size separately. C. A. KING.

Desulphurisation [of pig iron] in the basic open-hearth furnace. K. KÖHLER (Stahl u. Eisen, 1930, 50, 1257–1264).—The effect of lime and manganese in the slag on the desulphurisation of iron in the basic open-hearth furnace has been studied. The sulphur content of the finished steel is inversely proportional and that of the slag directly proportional to the lime content of the slag. The rate of removal of the sulphur is also a linear function of the lime content of the slag between 25 and 40% CaO. A further increase in lime increases the viscosity of the slag, and therefore reduces its reactivity. No definite relation between the manganese content of the slag and its efficiency as a scavenger for sulphur could be established. Slags with a low lime and a high manganese content have no desulphurising action, and a deficiency of manganese in no way impairs the desulphurising action of the lime. Addition of a large excess of manganese does, however, appear to have some accelerating action on the removal of sulphur from the metal bath. A. R. POWELL.

Influence of nickel and of chromium on the properties of cast iron. L. THIERY (Compt. rend., 1930, 191, 47–49).—As regards its influence on the transition temperature of cast iron, replacement of silicon by nickel yields a fine graphite more highly disseminated throughout the metal; nickel and chromium when simultaneously present give rise to an even finer structure. The cementite normally present in cast iron is replaced by pearlite as the nickel percentage increases, and by ferrite when the nickel content is increased to 1.40–2.46%. Nickel up to about 2% increases the tensile strength, extension, and elasticity, but diminishes the hardness (Brinell), whilst the subsequent addition of chromium increases the hardness and decreases considerably the extension and elasticity. H. F. GILLBE.

Nitrogen in technical iron. III. Precipitation of nitrogen and carbon from α -iron as an example of the decomposition of a doubly supersaturated solid solution. W. KÖSTER (Arch. Eisenhüttenw., 1930–1, 4, 145–150; Stahl u. Eisen, 1930, 50, 1401–1402; cf. B., 1930, 715).—The effect of ageing on the magnetic properties of mild steels containing nitrogen and quenched from just below the A1 point has been

studied in an attempt to explain the mechanism of the decomposition of a doubly supersaturated solid solution of the duralumin type. On ageing at 20°, only the effect of carbide precipitation on the magnetic properties can be detected; at 110–150°, however, the effect of nitride separation preponderates, but again above 150° the action of carbide precipitation is more marked. Measurements of the change of coercivity during ageing show that the presence of carbide in supersaturated solid solution retards the separation of nitride therefrom, especially at 110°. For any constant carbon content of the solid solution there is a linear relation between the total nitrogen content of the steel and the increase in coercivity on ageing. By plotting these curves for steels with varying nitrogen and carbon contents the effect of carbon on the amount of nitrogen which can be retained in solid solution has been determined; this varies from 0.002% N with 0.01% C to 0.02% N with 0.035% C. These results indicate that the isothermal precipitation of a constituent from a solid solution may be greatly influenced by the presence of a second constituent, and that in the case of steel the separation of nitrogen from solid solution at 100° comes to an end, independent of the total nitrogen content, when the amount still in solid solution reaches a definite value dependent on the carbon content of the solid solution.

A. R. POWELL.

Effect of contamination by nitrogen on the structure of electric welds. L. W. SCHUSTER (Iron and Steel Inst., Sept., 1930. Advance copy. 30 pp.).—It has been suggested from time to time that the needles found in welds are caused by oxygen, but in all the welds made with a Swedish iron electrode and also an electrode from which gases had been removed by heating in hydrogen, no needles were evident unless an appreciable amount of nitrogen was present. Welds made in an atmosphere of carbon dioxide were also free from needles, but in every weld made in nitrogen needles occurred either before or after heat-treatment in an atmosphere free from oxygen. In all cases in which needles have been formed as a result of a normalising treatment the original structure approached that known as a Widmanstätten structure, from which the needle structure develops. Freedom from needles by no means implies an absence of nitrogen, as the conditions may prevent nitride needles from coming out of solution, or a high nitrogen content may form braunite. C. A. KING.

Copper steel for dynamo and transformer sheets. A. KUSSMANN, B. SCHARNOV, and V. S. MESSKIN (Stahl u. Eisen, 1930, 50, 1194–1197).—Addition of up to 0.7% Cu to steel with 1.5 and 4% Si has practically no effect on the magnetic properties, but a larger proportion of copper has a deleterious effect. Less than 0.7% Cu improves the hardness and tensile strength and the resistance to corrosion of these steels, and is therefore a desirable addition.

A. R. POWELL.

Determination of chromium in special steels. P. KLINGER (Arch. Eisenhüttenw., 1930—1, 4, 7–15; Stahl u. Eisen, 1930, 50, 1166–1167).—The chromium content of 12 special steels with 1.5–12% Cr has been determined by nine different methods. Methods involv-

ing alkali fusions give the best results when the chromium is determined iodometrically, and of those based on dissolution of the steel in acids followed by oxidation to chromic acid the persulphate-silver nitrate procedure is the most satisfactory. The effect of other metals on the results given by the various procedures is briefly discussed.

A. R. POWELL.

Determination of manganese in cobalt steels. P. KRÜGER (Z. angew. Chem., 1930, 43, 687).—The method previously described (cf. Hallbauer and Krüger, B., 1927, 447) is modified to prevent the interference of organic substances derived from the carbon in the steel. The filtrate from the cobaltinitrite is boiled with a slight excess of nitric acid to destroy nitrites, neutralised with zinc oxide, filtered, and titrated directly with permanganate by Volhard's method. A. R. POWELL.

Diffusion of aluminium into iron. N. W. AGEEV and O. I. VIER (Inst. Metals, Sept., 1930. Advance copy. 13 pp.).—The reaction of iron with molten aluminium takes place in two stages: in the first of these the iron dissolves in the liquid until the solution is in equilibrium with the solid phase at a given temperature; in the second the alloy thus formed diffuses into the iron, forming the δ -solid solution of aluminium in iron and at temperatures above 1100° the ϵ -phase also. The rate of diffusion increases with the temperature according to Weiss' equation, but falls rapidly with the time of contact. These results are in agreement with the accepted equilibrium diagram of the system.

A. R. POWELL.

Gravimetric determination of vanadium in high-speed steel. S. CINBERG (J. Russ. Met. Soc., 1926, 173–176).—The steel is digested with hot 17% sulphuric acid, the residue (tungsten, vanadium, and carbides of iron and chromium) is collected and boiled with nitric acid (1 : 2) until all the tungsten is converted into trioxide. The liquid is evaporated to small volume and treated with alkali hydroxide, the residual chromium being reduced with alcohol and precipitated with ammonia. After removal of ammonia by boiling, the liquid is treated with a slightly acid solution of mercurous nitrate, the orange-yellow double salt which is precipitated being ignited in platinum to vanadium pentoxide. In steels containing no chromium, but 1.5–4.5% V, the vanadium can be determined by digesting with dilute sulphuric acid, igniting the insoluble residue in platinum, and weighing as V_2O_5 .

CHEMICAL ABSTRACTS.

Effect of titanium on the transformation points of steels. A. MICHEL and P. BÉNAZET (Rev. Mét., 1930, 27, 326–333).—Addition of titanium to mild steel raises the temperature of the A3 transformation and lowers that of the A4, so that it appears that the γ -field of the iron-titanium system is bounded by a closed curve extending outwards to about 1.5% Ti. Thus the system is somewhat similar to the iron-chromium system. In the iron-chromium alloy with 13% Cr addition of only 0.6% Ti is sufficient to suppress the $\alpha \rightarrow \gamma$ transformation.

A. R. POWELL.

Secondary tempering of over-tempered steels and stability of austenite. P. CHEVENARD and A. PORTEVIN (Compt. rend., 1930, 191, 523–525).—The

inferences that under conditions (*i.e.*, such that no point of transformation occurs) for over-tempering a hyper-eutectoid steel there should, as the percentage of carbon decreases, successively appear the points Ar₂ and Ar₁, and further, that between these points austenite should be specially stable, have been verified, and a case of secondary tempering was examined. A steel (1.5% C, 2% Cr) brought to the austenitic condition by over-tempering at 1180° in water was examined in the way previously described (*cf.* B., 1930, 950). It was heated to varying temperatures, θ_r , for 7 hrs., and the dilatation, hardness, and electrical and magnetic properties were determined and plotted against θ_r . The results for values of θ_r below 330° and above 500° are quite similar to those obtained (*loc. cit.*) for the corresponding manganese steel; but in the interval an entirely different phenomenon, a secondary tempering, is observed. This is discussed with the aid of the (amplitude of anomalies)/ θ_r curves (*cf.* B., 1921, 698 A), and the results are shown to be consistent with the following changes: with $\theta_r = 350^\circ$ austenite begins to change into martensite (Ar₂), the amount of cementite formed increasing as θ_r increases and the remaining austenite becoming more and more decarbonised. At 430° Ar₁ appears with formation of troostite. The double maximum of the hardness- θ_r curve indicates two causes of hardening: (a) production of the aggregate $\alpha +$ cementite, and (b) production of secondary martensite.

C. A. SILBERRAD.

Thermal hardening of grey castings. L. GUILLET, J. GALIBOURG, and M. BALLAY (*Compt. rend.*, 1930, 191, 538—541).—Austenitic grey castings acquire a martensitic structure as a result, *e.g.*, of prolonged heating at 400—600°, or of cooling in liquid air, and the change is accompanied by an increase in hardness. It is more usual in practice, however, to produce this effect by means of martensitic tempering followed sometimes by recovery (*cf.* B., 1928, 607). Data are given for the effects of varying temperature and conditions of recovery on the hardnesses of the castings after tempering in oil (700—950°), in a current of air (700—950°), and in still air (900°). The transformation points were determined dilatometrically. Simple tempering without recovery gave fragile castings with low resistance to shearing, bending, and compression. Recovery at 300—350°, though not greatly reducing the hardness, increased the above resistance by 20—25%.

J. GRANT.

Hardening of some metals [steel and monel metal] by cold-rolling. E. GREULICH (*Stahl u. Eisen*, 1930, 50, 1397—1401).—The hardness, yield point, and elongation of carbon and alloy steels begin to change after only a 1% reduction in thickness by cold-rolling, whereas the ultimate strength and reduction in area in the tensile test first show signs of a change after a 6% reduction. The hardness, yield point, and ultimate strength increase with the reduction in thickness by cold-rolling according to the logarithmic equation, $y = a + b \cdot \log \phi$, where a and b are constants and ϕ is the percentage reduction. The constant, a , depends on the original tensile strength and hardness, whereas b is characteristic of the capacity of the material for hardening by cold-work. For plain carbon steels,

5% nickel steel, and rustless chromium steels, $b = (9.5 \text{ kg./mm.}^2)/\phi$ and is independent of the carbon content, provided that the structure is pearlitic: austenitic steels and monel metal harden more rapidly, however.

A. R. POWELL.

Problem of ageing phenomena on the basis of recent research on iron alloys. W. KÖSTER (*Z. Metallk.*, 1930, 22, 289—296).—A summarised account is given of recent work of the author and others on the age-hardening of alloys of iron with carbon, nitrogen, and copper, especially of iron supersaturated with both carbon and nitrogen, and the bearing of the results on modern theories of age-hardening is discussed.

A. R. POWELL.

Heterogeneity of an ingot made by the Harmet process. A. KRÍŽ (*Iron and Steel Inst.*, Sept., 1930. Advance copy. 16 pp.).—Results of a complete macro-graphic and chemical examination of an ingot pressed by the Harmet method are given. The typical V-segregations formed in the central zone of ingots cast in the usual manner were not formed, the funnel-like contraction of the residual molten steel being counteracted by the artificial compression. A thick horizontal segregation under the head, evidently due to the compression, is typical in ingots cast by this method. C. A. KING.

Permanence of dimensions [of steel] under stress at elevated temperatures. W. H. HATFIELD (*Iron and Steel Inst.*, Sept., 1930. Advance copy. 18 pp.).—With the view of bringing the comparative permanence of dimensions of steel within the scope of a test practicable in point of time, the method adopted consists in determining, by static loading, the stress within which, at the given temperature, stability of dimensions is attained within 24 hrs. for a further period of 48 hrs. with an extension not exceeding the elastic deformation of 0.5% on the gauge length, and with limitation of measurement of the order of 0.000001 in. per in. per hr. This value is termed the "time yield," and a single test for a given temperature is obtainable in 3 days.

C. A. KING.

Evaluation of the quality of railway rails. A. N. MIRINSKY (*Rev. Mét.*, 1930, 27, 370—380).—Creep of the metal on the upper surface of a rail is due to the steel having too low an elastic limit, which is usually caused by rolling at too high a temperature. A sorbitic structure is the most resistant to wear and surface crushing, but a troostite structure is almost as satisfactory, especially as regards resistance to abrasion. Good rails may be made by rolling at a relatively low temperature or by rolling at a high temperature, cooling, reheating at 700°, cooling in a current of cold air to below 600°, and then allowing the rails to cool normally in still air.

A. R. POWELL.

Chemical methods of testing zinc coatings on sheet and wire. E. H. SCHULZ (*Stahl u. Eisen*, 1930, 50, 1017—1022).—A critical review of the various methods which have been proposed for determining the regularity of zinc coatings on iron and steel, the amount of zinc per unit area, and the resistance of zinc coatings to corrosion. Methods of testing for porosity and for differentiating between hot-galvanised and electrolytic deposits are also discussed.

A. R. POWELL.

Determination of manganese in cobalt steels.

J. KASSLER (Chem.-Ztg., 1930, 54, 733—734).—The steel (2.5 g.) is dissolved in 30 c.c. of nitric acid (d 1.2), the solution diluted, and the iron precipitated with zinc oxide. An aliquot part of the solution is filtered, treated with 3 g. of ammonium chloride, and boiled for 2 min. with 1 g. of ammonium persulphate. The precipitated manganic hydroxide, which contains only traces of cobalt, is redissolved in dilute nitric or sulphuric acid with the aid of a little hydrogen peroxide and oxidised to permanganic acid by the persulphate-silver nitrate method, and the analysis is finished in the usual way by titration with arsenite. A. R. POWELL.

Principles governing the testing of steel for its resistance to corrosion. E. H. SCHULZ (Stahl u. Eisen, 1930, 50, 1266—1269).—Standardised procedures and apparatus for making the usual accelerated and time corrosion tests on steels are briefly described.

A. R. POWELL.

Action of the common acids on various kinds of non-rusting steels.

M. SAUVAGEOT and (MLLE.) L. LAUPRÈTE (Rev. Mét., 1930, 27, 362—367).—The rates of dissolution in sulphuric, hydrochloric, and nitric acids of varying concentration of a 13% chromium steel, an austenitic nickel-chromium steel, an austenitic nickel-chromium-molybdenum steel, and a high-nickel steel have been determined at 15° and at 80°. The three steels containing chromium were all practically unattacked by nitric acid of any concentration at any temperature, but the nickel steel rapidly dissolved in hot 1—25% nitric acid and slowly in cold acid of the same concentration, although acid of a higher concentration than 30% was without action at any temperature. The nickel steel was by far the most resistant to the action of hydrochloric acid, but all the samples were badly corroded by hot 20% acid. Sulphuric acid had practically no action on the nickel steel whatever the temperature (up to 100°) or concentration; the other steels showed a maximum rate of dissolution in 40—45% acid. A. R. POWELL.

Attack of insects on metals. O. BAUER and O. VOLLENBRUCK (Z. Metallk., 1930, 22, 230—233).—Perforations in a lead water-pipe covered with felt were traced to the gnawing action of the beetles, *Dermestes peruvianus* and *D. larvatus*. Experiments with glass test tubes closed with thin sheet lead, tin, aluminium, zinc, and brass showed that the beetles could penetrate the two first-named metals, but were unable to make even the slightest impression on the others.

A. R. POWELL.

Reactions in blowing of cupriferous nickel matt.

V. TAFEL and F. KLEWETA (Metall u. Erz, 1930, 27, 85—88; Chem. Zentr., 1930, i, 2618).—The reaction $\text{Cu}_2\text{S} + 2\text{NiO} = 2\text{Cu} + 2\text{Ni} + \text{SO}_2$ takes place above 1300°. When formed in the converter, nickel oxide may react more readily.

A. A. ELDRIDGE.

New method of preparing copper wire-bars.

M. TAMA (Z. Metallk., 1930, 22, 207—209).—Direct melting of carefully washed copper cathodes in an Ajax-Wyatt induction furnace followed by casting into vertical moulds produces excellent copper ingots for drawing into wire. Owing to the neutral atmosphere

of the furnace, the oxygen content of the bars is extremely low and the other impurities should not exceed 0.002%. Wire 3.56 mm. in diam. drawn from these bars has a conductivity of 58 m./ohm-mm.², a tensile strength of 25 kg./mm.², and an elongation of over 35%.

A. R. POWELL.

Tensile properties of electrical conductor wires at low temperatures. F. PESTER (Z. Metallk., 1930, 22, 261—263).—The tensile strength of copper wire increases with fall of temperature from 20° to —60° by 9%, that of bronze wire by 6.3—8.9%, that of aluminium wire by 10.5%, and that of alloy wire by 10.2%. The elongation in all cases increases considerably on cooling, but the bending test for copper, bronze, and alloy shows minimum values at —20°.

A. R. POWELL.

Properties of extruded rods of electrolytic copper, smelter copper, arsenical copper, and 4% aluminium bronze, and their variation with the extruding conditions. P. SIEBE and G. ELSNER (Z. Metallk., 1930, 22, 203—206, 238—245).—The tensile properties, resistance to impact shock, and grain size of extruded rods of electrolytic copper (99.86% Cu, 0.05% O), refined copper (99.66% Cu, 0.06% O), arsenical copper (99.49% Cu, 0.39% As, 0.09% P), and aluminium bronze (4.04% Al) have been determined immediately after extrusion at 750—850° and at 990—1020°, after subsequent drawing to reduce the diameter by 15%, and after annealing for various periods at 650—950°. All of the copper specimens had a higher tensile strength but lower ductility when extruded at 800° than when extruded at 1000°, but the differences were somewhat reduced by subsequent drawing; of the three varieties of copper, that containing arsenic had the highest strength and ductility in all cases. The tensile strength of the aluminium bronze remained the same after extrusion at 800° or at 1000°, but the higher temperature increased the elongation; in this case the drawn metal had better tensile properties at the lower extrusion temperature. Photomicrographs and a tabulated description of the structural changes which occur during extrusion, drawing, and annealing are included.

A. R. POWELL.

Manufacture application of "Y" and "R.R." alloys. W. C. DEVEREUX (Nickel Bull., 1930, 3, 286—293).—Details are given of the methods of preparing copper and nickel hardener alloys for use in making "Y" and "R.R." aluminium alloys; then follows an account of the methods of melting, casting, heat-treating, and working these alloys and a brief outline of their properties.

A. R. POWELL.

Electric melting of aluminium. E. F. RUSS (Z. Metallk., 1930, 22, 273—276).—Crucible, kettle, and hearth furnaces of the resistance type for the melting of aluminium are described and illustrated. They have the advantage over gas-, oil-, or coke-fired furnaces of a stationary atmosphere which reduces the loss by oxidation and prevents gas adsorption to a large extent. By means of automatic temperature regulators a steady temperature gradient may be maintained and all danger of overheating the metal is avoided. A tilting-hearth furnace with a capacity of 1500—2000 kg. will melt

8–10 tons of aluminium in 24 hrs. with a current consumption of 250 kw./hr. at 220 volts, equivalent to 600–700 kw.-hrs. per ton. The loss by oxidation should not exceed 1%. A. R. POWELL.

Remelted aluminium and aluminium alloys and their uses. R. STERNER-RAINER (Z. Metallk., 1930, 22, 269–272).—In remelting scrap aluminium alloys the charge should be made up to contain 5–7% Zn and 3–4% Cu for sand-castings, 2–3.5% Zn and 6–8% Cu for chill-castings, and not more than 2.5% Zn and 6–8% Cu for hollow castings. In no case should the impurities exceed 1.2% Fe + Si, 0.3% Pb, 0.3% Sn, 0.2% Ni, 0.1% Mn, and 0.1% Mg; the minimum aluminium content in all cases is 87%. The alloys are capable of improvement by heat treatment and are rather more easily worked on the lathe than are similar alloys made from virgin metal, as the turnings are shorter and there is less tendency for the tool to tear the metal surface. A. R. POWELL.

Improvement of an aluminium alloy as shown by X-rays. VON GÖLER and G. SACHS (Mitt. Materialprüf., 1930, 117; cf. A., 1929, 743).—X-Ray examination of a 5% Cu aluminium alloy after quenching from 525° and ageing at various temperatures shows that no change in the structure takes place below 100°; at 150° about 20% of the copper is precipitated out of solid solution in 24 hrs., at 200° 40% is precipitated in 30 min., and at 250° the whole of the copper separates within a short time. On the other hand the mechanical properties improve with ageing at below 100°; this improvement is characterised by an increase in the yield point and tensile strength without a corresponding reduction in the ductility. Precipitation of the copper is accompanied by a further increase in tensile strength but a reduction in the ductility. With single-crystal specimens precipitation of the copper causes considerable variations in the mechanical behaviour, so that the crystal behaves like fine-grained metal, although it has an X-ray structure similar to that of a single crystal, but single crystals aged at the ordinary temperature behave in the tensile test in a normal way. A. R. POWELL.

Gas removal and grain refinement of aluminium alloys. W. ROSENHAIN, J. D. GROGAN, and T. H. SCHOFIELD (Inst. Metals, Sept., 1930. Advance copy. 14 pp.).—By passing through molten aluminium or its alloys the vapour of carbon, silicon, titanium, or tin tetrachloride, aluminium chloride, ferric chloride, or tetrachloroethane, gas occlusions are almost completely removed from the metal. Ferric and tin chlorides cause the metal to be contaminated with the corresponding metals, as also does, to a very small extent, titanium tetrachloride, but in this case the titanium acts beneficially in causing the development of a very fine grain which persists after several re-meltings. This grain refinement can also be produced by adding small quantities of titanium-aluminium alloy to the metal, but this treatment does not remove dissolved gases. The chloride treatment of modified silicon-aluminium alloys results in their complete reversion to the normal state. Treatment of "Y" alloy with 2 c.c. of titanium tetrachloride per lb. provides a method of producing high-quality castings in sand or chill moulds and appreciably improves the forging, rolling, and drawing properties of the metal. A. R. POWELL.

ciably improves the forging, rolling, and drawing properties of the metal. A. R. POWELL.

Eutectoid transformation of aluminium bronze. II. Effect of quenching velocity. I. OGINATA (Mem. Ryojun Coll. Eng., 1930, 3, 87–94; cf. B., 1930, 105).—Quenching in water from above 500° of the aluminium bronze with 12.5% Al does not completely suppress the β - β' transformation, which can still be detected by an evolution of heat as the alloy cools from 500° to 400°. The acicular structure obtained by quenching in water is therefore regarded as a heterogeneous mixture of β and β' . Quenching in toluene at –95° produces a homogeneous structure which is assumed to be that of pure β , and the apparently homogeneous structure produced by cooling at a somewhat slower rate than obtains in water-quenching is considered to be pure β' , which is much harder than β . A. R. POWELL.

Nickel-aluminium bronze. T. ISHIKAWA (J. Soc. Mech. Eng. Japan, 1928, 31, 215–233).—The hardness of forged bars of nickel (10%)–aluminium (2%) bronze rapidly increases by tempering at 600° after quenching in water or liquid bath below 400°.

CHEMICAL ABSTRACTS.

Hardening of beryllium-aluminium and magnesium-aluminium alloys. M. HAAS and D. UNO (Z. Metallk., 1930, 22, 277–279; cf. B., 1930, 716).—Dilatometric tests and micrographical examination of beryllium-aluminium alloys have confirmed the composition of the eutectic as 1.4% Be, 98.6% Al, m.p. 644°. The solid solubility of beryllium in aluminium increases from 0.2% at 20° to 0.8% at 640°. Schmid and Spitaler's equilibrium diagram of the aluminium-magnesium system has been confirmed by dilatometric analysis. A. R. POWELL.

Notched-bar impact strength of some aluminium alloys, especially at low temperatures. W. A. GÜLDNER (Z. Metallk., 1930, 22, 257–260).—The notched-bar impact strength of aluminium alloys is of the order of 1–4 m.-kg./cm.², its actual value in any specific case being a linear function of the speed of the blow. The slope of the straight line showing this relation is a definite property of the metal and is not affected by heat treatment although it varies with the mechanical treatment. An increase in the weight of the hammer has no influence on the impact strength. A. R. POWELL.

Corrosion of light and ultra-light metals and alloys. R. CAZAUD. (A) Critical studies of corrosion tests. E. HERZOG and G. CHAUDRON. (B) Reduction of mechanical properties by corrosion. COURNOT and MOLNAR. (C) Development of the zone of action of galvanic couples. COURNOT and MOLNAR. (D) Method of [testing corrosion by] the E.M.F. of dissolution. AUBERT and PROT. (E) Relative tests on the rise of temperature of activated aluminium alloys. QUILLARD. (F) Effect of arsenic on the corrosion resistance of duralumin in sea-water. R. CAZAUD. (G) Use of fatty substances in the protection of aluminium against chemical action. A. TRILLAT. (H) Application of the "phenol method" to the protection of light aluminium alloys. AUBERT and A. PIGNOT. (I) Resistance to sea-water of aluminium sheet with various protective coatings. QUILLARD.

(J) Resistance to sea-water of special light aluminium alloys. LEGENDRE. (K) Practical corrosion tests on caissons. LECŒUVRE (Rev. Mét., 1930, 27, 285—289, 290, 291—296, 296—299, 299—300, 337—341, 341—343, 343—344, 344—347, 347—350, 350—351).—(A) Experiments with various light-metal alloys in numerous corrosive liquids show that the loss in weight is not a reliable method of measuring the resistance to corrosion; more reliable comparative figures are obtained by determining the diminution in the percentage elongation in the tensile test. (B) The salt-spray test is a more rapid method of determining the resistance of aluminium alloys to sea-water than is the total immersion test. In the spray test the reductions in tensile strength and elastic limit do not appear to be proportional to the progress of corrosion. (C) Cadmium and zinc protect aluminium from corrosion by sea-water, whereas iron, copper, and nickel accelerate the corrosion. For aeronautical purposes aluminium alloys may be rivetted with cadmium-plated duralumin rivets, which act as a protection against corrosion, causing the surrounding metal to become covered with a thin protective film of alumina. Aluminium and duralumin rivets act as protective agents in magnesium alloy structures. (D) The effect of inclusions in duralumin in promoting corrosion by the formation of local couples is discussed; incorrect thermal treatment may result in accelerated corrosion, owing to the segregation of more electropositive constituents. (E) The rate at which the temperature of the metal rises after dipping it in mercuric chloride solution and exposing it to the air is a measure of the resistance of an aluminium alloy to corrosion. This test shows that addition of 1—1.5% Mn to aluminium greatly reduces its rate of corrosion, but that 2% Mg increases the rate considerably. (F) Arsenic may be added to duralumin in the form of copper or manganese arsenides, but losses of arsenic by volatilisation occur during heat-treatment. In the microstructure arsenic appears as needles of arsenide which have little effect on the mechanical properties or resistance to corrosion. (G) Coating aluminium with various fatty substances affords efficient protection against corrosion; a water jet under 9—12 kg. pressure does not remove the greasy coating. (H) Aluminium alloys may be protected from corrosion by painting with a mixture of 30% of pitch, 5% of phenol, and 65% of light petroleum; two coats are required, the metal being allowed to dry for 48 hrs. after each application. A final application of aluminium paint is given to restore the original aluminium appearance of the surface. (I) Comparative tests on the protective action of films produced by treatment of aluminium with sodium silicate and of films of paraffin and varnish are recorded. (J) The effect of protective plates of zinc attached to structures in which duralumin is in contact with another metal or alloy under sea-water is illustrated, and the behaviour in sea-water of certain special aluminium alloys of unstated composition is briefly described. (K) Aluminium structures may be protected from corrosion by covering them with a resistant varnish or with cadmium by the Schoop spray method or by electrolysis. Good protection is also afforded by applying bands of zinc by the Schoop method to various parts of the structure. A. R. POWELL.

Transformation points in metals [zinc, bismuth, thallium, and cobalt]. A. SCHULZE (Z. Metallk., 1930, 22, 194—197, 308—311).—The electrical resistance, thermoelectric power, and thermal expansion of the above four metals have been determined at temperatures up to the m.p. The curves for "spectroscopically pure" zinc show no points of inflexion, whereas those for zinc containing cadmium, lead, and iron show one or more such points corresponding with the m.p. of the eutectic of zinc and the impurity. It would thus appear that zinc undergoes no allotropic transformation, previous observations to the contrary being due to the presence of impurities. Similarly, pure bismuth undergoes no allotropic transformation between -190° and 270° , but the presence of impurities may cause inflexions in the curves of some of its physical properties. Thallium shows abrupt changes in the thermal expansion and electrical resistance curves at 225.2° on heating, due to the change $\alpha \rightarrow \beta$ -thallium; on cooling, the reverse change takes place a few degrees lower. β -Thallium has a lower resistance and greater coefficient of expansion than α -thallium. Cobalt undergoes an allotropic transformation on heating at 444° , but the reverse change is subject to considerable hysteresis effects the magnitude of which is enhanced by the presence of impurities. A second change occurs at 1128° , when the metal loses its magnetic properties.

A. R. POWELL.

Diffusion of zinc and lead into liquid tin. Kinetics of soldering. A. MERZ and E. BRENNECKE (Z. Metallk., 1930, 22, 185—189, 234—237).—The diffusion constants of zinc and lead in molten tin have been determined at 260° , 310° , and 370° by the Graham-Stefan method. The constant for zinc at 260° is $2.66 \text{ cm.}^2/\text{day}$ and the temperature coefficient $0.0119 \text{ cm.}^2/\text{day}/^{\circ}\text{C.}$ between 260° and 370° . For lead the diffusion constant is $1.23 \text{ cm.}^2/\text{day}$ at 260° and the temperature coefficient $0.0066 \text{ cm.}^2/\text{day}/^{\circ}\text{C.}$ Within the above temperature range the rate of diffusion in both cases is independent of the concentration of the metals. When copper is dipped into molten tin a layer of Cu_3Sn is formed below 420° and this layer has a retarding effect on the rate of dissolution of copper in liquid tin. The above results are discussed in the light of Nernst's theory of reaction velocity in heterogeneous systems and their bearing on the theory of the mechanism of soldering is indicated.

A. R. POWELL.

Recovery of fine gold and silver from old gold and silver alloys. E. ROSENBAUM (Chem.-Ztg., 1930, 54, 743—744).—The method of refining gold-silver alloys by inquartation and parting with nitric acid is described and a detailed account given of the usual ammonium chloride-sulphur dioxide method for recovering platinum and gold from solutions obtained by dissolving alloys of these metals in aqua regia.

A. R. POWELL.

Electrodeposition of gold alloys. G. GRUBE (Heraeus Festschr., 1930, 34—44; Chem. Zentr., 1930, i, 2475).—The electrodeposition of alloys of gold with copper, silver, and with both metals, is described. The concentration of the electrolyte must be kept constant and air excluded.

A. A. ELDRIDGE.

Titration of potassium cyanide, and of free cyanide in silver-plating solutions, by means of silver nitrate. E. B. SANIGAR (Trans. Amer. Electrochem. Soc., 1930, 58, 49—68).—The effects of certain impurities (especially carbonates) on the sharpness and accuracy of the end-point in the titration of 0.5*N*-potassium cyanide and of a silver-plating solution (20.5 g. Ag per litre, 0.4*N* free cyanide) with silver nitrate, using potassium iodide as indicator, have been investigated. The presence of potassium carbonate in the simple cyanide solution did not interfere with the titration unless its concentration was as high as 0.18*N*, when precipitate formation began to mask the end-point. In the argenticyanide solution, however, this limiting concentration of carbonate was only 0.03*N*. Addition of 0.1*N*-sodium hydroxide is without effect on the sharpness of the end-point or on precipitate formation. Addition of ammonia raises the limiting carbonate concentration considerably, but tends to cause high results unless the concentration of potassium iodide is increased. It is found, however, that precipitate formation due to carbonates can be prevented without affecting the accuracy of the end-point, merely by diluting the argenticyanide solution sufficiently. With an average plating solution containing carbonates, a 35—50-fold dilution is recommended before titration with 0.1*N*-silver nitrate. This method is preferred to that in which carbonates are removed by precipitation with barium or calcium salts before titration, since any excess of these salts leads to the formation of precipitates which mask the end-point, or if the solution is sufficiently diluted to avoid this, the titration values obtained are somewhat low. H. J. T. ELLINGHAM.

Scientific industrial control of electrolytic deposits, especially of nickel and chromium. M. BALLAY (Rev. Mét., 1930, 27, 316—325).—A review of modern scientific methods of controlling the p_H of nickel-plating baths and the current yields in nickel- and chromium-plating baths, and of determining the adherence and resistance to corrosion of electrolytic deposits. A. R. POWELL.

Electrodeposition of cobalt-nickel alloys. I. S. GLASSTONE and J. C. SPEAKMAN (Trans. Faraday Soc., 1930, 26, 565—574).—The composition of the alloys deposited from solutions of definite p_H between 3.2 and 5.2, buffered by addition of acetic acid as required, and containing nickel and cobalt sulphates at concentrations varying from 2 to 0.41 g.-equiv. per litre has been determined for current densities ranging from 0.0028 to 5—6 amp./dm.² The deposit always contains a relatively greater proportion of cobalt than the solution from which it is deposited, on account of the retarding influence of the cobalt ion on nickel. The composition of the alloy at low current density is not influenced by changes of current density, by dilution, or by stirring the electrolyte. At current densities above about 1 amp./dm.², the cobalt content of the deposit decreases with the current density, as a result of the decrease of the cobalt-ion concentration caused by the preferential deposition of that metal; the effect becomes more marked on dilution, but is slightly diminished by stirring.

With increase of the p_H of the electrolyte the composition of the initial deposit remains unchanged, but the effect of the impoverishment in cobalt ions becomes operative at lower current densities, and the current efficiency increases. Added anions or cations do not appear to influence the composition of the alloy deposited. The results obtained are compared with those found for the simultaneous deposition of iron and nickel.

H. F. GILLBE.

"Pendulum" hardness tests of commercially pure metals. D. A. N. SANDIFER (Inst. Metals, Sept., 1930. Advance copy. 29 pp.).—The time and scale hardnesses and work-hardening capacities of 23 commercially pure metals have been determined with the Herbert pendulum tester. Time work-hardening capacity is measured by plotting induced hardness against the number of passes of the pendulum ball and subtracting the original time hardness from the maximum induced hardness read from the curve. There is no definite relation between the time and scale hardnesses of pure metals. The time work-hardening capacity of soft metals is usually low; it increases as the time hardness increases, then decreases again, finally becoming negative for very hard metals, e.g., manganese. The relation between the scale hardness (*S*) and the scale work-hardening capacity (*SC*) is expressed by the equation: $(S-29)^2 + 66(SC-51.1) = 0$ for a pendulum length of 0.21 mm. The time hardness (*T*) is a linear function of Young's modulus; for pure metals $E = 8.1 \times 10^{10} T$ dynes/cm.² for a pendulum length of 0.1 mm. For all metals for which $T < 30$ the relation $TV^{1.87} = 818.5$ (where *V* is the at. vol.) holds good, the sole exception being antimony. The gradual increase in hardness produced by cold-working a pure metal is accompanied by a slow decrease in the work-hardening capacity.

A. R. POWELL.

Effects of two years' atmospheric exposure on the breaking load of hard-drawn non-ferrous wires. J. C. HUDSON (Inst. Metals, Sept., 1930. Advance copy. 14 pp.).—Samples of wire (diam. $\frac{1}{8}$ in.) were exposed to the London atmosphere for 2 years and their tensile strength was then compared with their original strength and with the strength of similar wires kept indoors for the same period. The wires subjected to atmospheric corrosion showed an average loss in strength of 2—3%, except in the case of 70:30 brass, for which the loss in strength was much greater. The absolute values for the corrosion deduced from these results are of the same order of magnitude as those obtained in more prolonged field tests. The poor behaviour of brass wire is attributed to secondary action of the brass on the copper corrosion products first formed, resulting in the redeposition of copper and dezincification of the outer brass layers. The corrosion products which adhere to nickel-copper alloys contain a much higher proportion of copper than that in the original alloy; this is due to selective dissolution of the nickel in the rain water and removal of the soluble nickel salts, and is especially marked when the atmosphere contains a high proportion of sulphur compounds. A. R. POWELL.

Brinell, Rockwell, and scleroscope hardness of non-ferrous metals. O. SCHWARZ (Z. Metallk., 1930,

22, 198—202).—Hardness measurements of annealed and hard-worked copper, brass, aluminium, and light aluminium alloys have been made with the Brinell, Rockwell, and scleroscope hardness testers. The relation between the Rockwell *B* hardness ($\frac{1}{16}$ -in. ball, 100-kg. load) and the Brinell hardness (*H*) (load in kg. = $10 \times$ [diam. of ball]²) is $H = 8300/(140 - B)$ for values of *H* between 50 and 180, and between the Rockwell *C* hardness (2.5-mm. ball, 62.5-kg. load) and the Brinell hardness $H = 2900/(100 - C)$. The Brinell hardness number is roughly 4.5 times the scleroscope number for non-ferrous metals (mean deviation $\pm 5\%$), but the scleroscope does not measure exactly the same property of a material as the Brinell and Rockwell machines; the scleroscope number is a function of the product of the elastic limit and elongation. A. R. POWELL.

Treatment of accumulated concentrates from the Oriental Consolidated Mining Co., Hokuchin, Korea. W. G. CLARKE and B. H. MOORE (Bull. Sch. Min. W. Australia, 1930, 5, 31—38).—Fine-grinding and cyanidation by agitation of the raw concentrates (4.5 dwt. Au per ton) was preferred to roasting and cyanidation. CHEMICAL ABSTRACTS.

Leaching of chalcocite. SULLIVAN. **Copper leaching.** KEYES.—See VII.

See also A., Oct., 1245, **System nickel-chromium** (SEKITO and MATSUNAGA). **System iron-tungsten** (TAKEDA). **System iron-nickel-cobalt** (KÜHLEWEIN). 1253, **Electrolysis of amalgams, fused silver-lead alloys, and bronzes** (KREMAN and others). 1258, **"Explosive" antimony** (VON STEINWEHR and SCHULZE; COHEN and COFFIN).

PATENTS.

Production of metals [iron] from ores. E. A. A. GRÖNWALL and H. J. H. NATHORST (B.P. 334,500, 26.4.29).—The ore is smelted in the usual way in a shaft furnace, but the gases issuing from the shaft are cooled for the removal of carbon dioxide by condensation and/or washing and the residual gases are mixed with a proportion of oxygen insufficient for their complete combustion and returned to the lower part of the furnace, preferably after carburetting the mixture by passing it through a mass of glowing coal. A. R. POWELL.

Low-temperature reduction of iron ores. E. D. NEWKIRK and A. J. BRIGGS (U.S.P. 1,760,078, 27.5.30. Appl., 3.10.27).—A mixture of finely-ground iron ore and coke is passed downwards through a cylindrical, rotating tube furnace, inclined slightly to the horizontal, and a stream of highly reducing gases is passed counter-current to the ore stream. At intervals along the tube a limited supply of air is admitted, so that the gases are fractionally and progressively burned as they pass towards the feed hopper. The continuous production of a good grade of sponge iron in one operation is thus ensured without external heating of the furnace. A. R. POWELL.

Extraction of useful elements [iron] from their ores by the aid of gases. B. HOFSETH (U.S.P. 1,759,456, 20.5.30. Appl., 25.8.27. Norw., 10.9.26).—Iron ore is treated, *in situ*, with hot gases to preheat a section to 350—500°, and currents of hydrogen and

chlorine from separate pipes are allowed to impinge on the heated zone, whereby ferric chloride volatilises and may be collected in suitable chambers through which the issuing gases are drawn directly from the ore body. The heat evolved in the reaction is sufficient to maintain it continuously as long as hydrogen and chlorine are supplied in sufficient quantity. A. R. POWELL.

Melting of metal or heating molten metal in an electrical furnace, and a furnace therefor. K. G. WENNERSTRÖM (B.P. 334,201, 28.5.29).—The metal is melted above a layer of hot slag, through which an electric current is passed. The slag is contained in a series of narrow heating chambers in the lower part of the furnace, and the electrodes conducting the current to these chambers are internally water-cooled. A. R. POWELL.

Furnace for casting iron and other difficultly fusible metals under pressure. E. PETERSSON (U.S.P. 1,758,463, 13.5.30. Appl., 5.11.26. Swed., 9.1.25).—The furnace is provided with a casting pipe which terminates in a nozzle situated in a hole in an upper side-wall of the furnace so that it is exposed to the heat of the molten metal. The mould is pressed against this nozzle by means of a self-adjustment device which ensures a vacuum-tight connexion with the mould. A. R. POWELL.

Casting of readily oxidisable metals. W. W. SMITH (U.S.P. 1,761,498, 3.6.30. Appl., 18.4.28).—The casting mould is made from a mixture containing 50—90% of pulverised carbon, 2—15% of powdered soap-stone, and the remainder magnesium oxide, the mixture being bound with a light mineral oil. The surface of the mould is dusted with magnesium oxide. C. A. KING.

Ingot mould. E. J. KAUFFMAN, Assr. to VALLEY MOULD & IRON CORP. (U.S.P. 1,762,098, 3.6.30. Appl., 19.7.29).—In casting an ingot mould, a mixture of cast iron and at least 5% of mild steel is used. A mould having a gradation from steel at the inner face to iron at the outer surface is made by casting first the iron centrifugally and then the mild steel. C. A. KING.

[Production of] metal [cast iron]. C. F. LAUENSTEIN, Assr. to LINK-BELT CO. (U.S.P. 1,760,241, 27.5.30. Appl., 15.10.28).—Cast iron having a structure consisting of grains of ferrite and graphite surrounded by an intercrystalline bond of sorbite or pearlite is claimed. This structure is obtained by annealing white cast iron at 800°, quenching, and annealing at 500—650°. A. R. POWELL.

Manufacture of cast-iron grinding balls. F. KRUPP GRUSONWERK A.-G. (B.P. 319,350, 18.7.29. Ger., 22.9.28).—The cast iron used contains 0.3—1.8% Si, 2.7—3.8% C, and 0.3—1.2% Cr, the actual proportion of these constituents being varied according to the diameter of the ball, so that the sand-cast metal has a ledeburitic structure without an exceptionally hard skin. This is obtained if $D = 250 + 250\text{Cr}/3 - 50(\text{Si} + \text{C})$, where *D* is the diam. in mm. and Cr, Si, and C are the percentages of the alloying elements. A. R. POWELL.

Iron refining. G. S. EVANS, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,759,347, 20.5.30. Appl., 18.2.28).—After melting down the metal in an air

furnace, the slag is removed and a soda ash refining agent is introduced into the firing end of the furnace, allowed to flood the surface of the metal, and thereafter removed from the opposite end. The original slag may be further fluxed by an addition of the same reagent.

C. A. KING.

Metal [case-]hardening process. G. A. FLECKENSTEIN, E. M. HANF, and M. L. WATERMAN, Assrs. to SINGER MANUF. CO. (U.S.P. 1,759,690, 20.5.30. Appl., 23.11.28).—The parts of an article which are to be kept soft during any of the usual case-hardening processes are first plated with a coating of chromium 0.00006 in. thick.

A. R. POWELL.

Treatment of ferrous metal articles. M. C. BAKER and W. A. DINGMAN, Assrs. to PARKER RUST PROOF CO. (U.S.P. 1,761,186, 3.6.30. Appl., 30.6.28).—The articles are pickled, washed, heated at 260°, quenched in 33% phosphoric acid solution to which, if desired, metal phosphates may be added, and dried at 160—230° without washing.

A. R. POWELL.

[Inhibitor for use in the pickling] treatment of iron or steel. K. B. THEWS and J. S. CARLITZ (U.S.P. 1,759,634, 20.5.30. Appl., 7.12.29).—Substances of the general formula $CX(NR_2)_2$, where X is selenium or tellurium, and R is an alkyl, aryl, or acyl group, are claimed.

A. R. POWELL.

Metallurgical process [for treatment of copper sulphide ores]. R. D. PIKE (U.S.P. 1,761,641, 3.6.30. Appl., 11.2.26).—The ore is leached with a hot solution of ferric chloride and the filtered liquor is treated with sponge iron to remove copper. The ferrous chloride solution thus obtained is treated with calcium chloride to reduce its sulphate content to a value below that corresponding with the solubility of calcium sulphate, filtered, and electrolysed to obtain pure iron. The tailings from the leaching vats are dried, heated to remove sulphur by distillation, roasted, and leached with the spent electrolyte from the iron cells before it is returned to the main leaching vats.

A. R. POWELL.

Smelting of copper. A. D. WILKINSON, Assr. to CANANEA CONSOL. COPPER CO., S.A. (U.S.P. 1,759,078, 20.5.30. Appl., 26.3.26).—A mixture of 25% of flotation concentrates, 66—70% of crushed copper ore, and 9—5% of converter chips, the whole containing 9—10% of moisture, is banked at a steep angle in a reverberatory and smelted under conditions which result in the formation of a rich copper matte. The presence of water in the charge eliminates a large proportion of the sulphur, and oxidises most of the iron into the slag.

A. R. POWELL.

Reducing the quantity of sulphurous acid gas set free from copper glance during roasting. M. MIYAKE (B.P. 334,761, 18.10.29).—Copper sulphide ore is mixed with a small proportion of bone ash and red-earth prior to roasting.

A. R. POWELL.

[Nickel-iron-copper] alloy and the process of forming the same. J. J. OLSEN (U.S.P. 1,759,169, 20.5.30. Appl., 24.8.27).—An alloy suitable for use in welding or brazing is made by melting 140—150 lb. of copper, adding 4—5 oz. of 85% ferrosilicon, 3.5—7 lb. of carbon-free iron, and 1.75—3.5 lb. of nickel, stirring

under a sodium nickel borate flux, and casting in a reducing atmosphere at just above the m.p.

A. R. POWELL.

Manufacture of [silicon-copper-tungsten or -molybdenum] alloys. J. GRAY. From A. PACZ (B.P. 335,081, 7.10.29).—An alloy for the manufacture of electrical contact terminals comprises 50—95% Mo and/or W, 5—50% Cu, and 0.5—5% Si. Part of the copper may be replaced by silver and 0.5—2% Fe, Co, or Ni may be added. The alloy is produced by precipitating a mixture of alkali silicate, tungstate, or molybdate with an acid, reducing the oxides in hydrogen, heating the resulting powder with magnesium in an inert atmosphere, extracting the magnesia with acid, again reducing the metals in hydrogen, and heating the sponge so formed with molten copper or a copper-silver alloy until the latter soaks into the mass.

A. R. POWELL.

[Electric] furnace for producing aluminium. P. E. FRØLAND (U.S.P. 1,761,442, 3.6.30. Appl., 1.3.28. Norw., 12.3.27).—The bottom of an electric furnace is of tamped carbon in which is embedded a more solid bar of carbon, e.g., an electrode, which extends outside the furnace to form the cathode connexion. As an auxiliary contact, bars of aluminium preferably are let into recesses in the underside of the bed and connected also to the current supply.

C. A. KING.

[Zinc-aluminium alloy. T. S. FULLER and D. BASCH, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,760,549, 27.5.30. Appl., 13.12.23).—The alloy contains at least 80% Al, more than 6% Zn, 0.2—2.5% Mg, and 1—3% Fe. It is annealed at 450—575°, quenched, and aged at 150° for 1—3 hrs. After this treatment an alloy of 80.75% Al, 6% Zn, 2% Fe, and 1.25% Mg has a hardness of 65 Brinell and a tensile strength of 30,000 lb./in.²

A. R. POWELL.

Aluminium alloys. METALLGES. A.-G. (B.P. 335,105, 7.11.29. Ger., 7.12.28).—Alloys of aluminium with 1—6% Cu, 2—14% Zn, up to 1% Mg, and up to 0.5% Si, with or without up to 1% Cd and Mn, are claimed. The preferred range of composition is 3—4% Cu, 8—10% Zn, 0.1—0.5% Mg, 0.05—0.1% Li, 0.4—0.8% Mn, and 0—1% Cd.

A. R. POWELL.

Casting light metals, especially aluminium and aluminium alloys. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 334,435, 29.10.29. Ger., 29.10.28).—A rectangular melting furnace is provided with a number of small delivery chambers arranged in a row on one side of the furnace and connected therewith by means of small channels. These chambers are closed at the top by means of a cooling supporting plate on which the chill moulds are situated and which is provided with supply tubes which project into the molten metal in the delivery chambers. In this way the metal may be drawn up into the mould without disturbance of the metal surface by connecting the upper part of the mould to a vacuum pump.

A. R. POWELL.

[Silicon-aluminium alloys [for pistons]. K. SCHMIDT GES.M.B.H. (B.P. 334,777, 6.11.29. Ger., 15.8.29).—An aluminium alloy consisting of more than 70% Al with 15—25% Si, 0.2—3% Cr, 0.5—3% Mn, 0.5—3% Co, 0.5—3% Ni, 0.5—3% Sb, and 0.5—1% Ti and/or Mo is claimed.

A. R. POWELL.

Removal of carbonyl-forming metals or carbides thereof from soot. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,190, 27.5.29).—The material is heated at 400–500° in hydrogen, an inert gas, or a gas mixture containing a small proportion of oxygen, and then at 50–210° in a current of carbon monoxide under elevated pressure and in the presence of a small amount of sulphur as a catalyst. A. R. POWELL.

Metal [lead] foil. W. J. HAWKINS, Assr. to AMER. MACHINE & FOUNDRY Co. (U.S.P. 1,760,645, 27.5.30. Appl., 16.5.28).—Lead containing 0.25–0.5% Mg may be rolled to foil having a thickness of only 0.0005 in. A. R. POWELL.

Lead alloy for electric cable sheaths etc. STANDARD TELEPHONES & CABLES, LTD., Assees. of R. S. DEAN (B.P. 314,522, 31.5.29. U.S., 29.6.28).—An alloy of lead with 0.02–0.07% Ca quenched from 310–325° and aged at below 200° is claimed. [Stat. ref.]

A. R. POWELL.

Electrolytic recovery of tin in the form of compact plates of any desired thickness, from alkaline lyes containing alkali stannate. SIEMENS & HALSKE A.-G. (B.P. 334,738, 1.10.29. Ger., 28.3.29. Addn. to B.P. 286,673; B., 1928, 452).—The addition of colloids to the bath as specified in the prior patent is unnecessary when lead and bismuth salts are absent.

A. R. POWELL.

[Nickel]-electroplating process. R. J. WIRSHING and H. R. FAAS, Assrs. to GEN. MOTORS RES. CORP. (U.S.P. 1,761,948, 3.6.30. Appl., 10.1.27).—Nickel may be plated from a bath containing 200 g. of nickel acetate and 20 g. of sodium chloride per litre, using a current density of 15–100 amp./ft.² at 35–70°.

A. R. POWELL.

Metal netting for use as catalyst in contact units [e.g., for ammonia oxidation]. BAMAG-MEQUIN A.-G. (B.P. 334,466, 12.12.29. Ger., 8.2.29).—Platinum and/or rhodium wires are inserted as warp and/or weft threads in a netting composed of platinum-rhodium alloy. J. S. G. THOMAS.

Soldering flux. F. E. HARRIS (U.S.P. 1,757,118, 6.5.30. Appl., 2.3.29).—A flux, suitable for use in soldering aluminium, copper, pewter, etc., comprises zinc stearate mixed with an oil (olive oil) and a light petroleum derivative such as benzol, or benzine admixed with carbon tetrachloride to destroy inflammability.

S. K. TWEEDY.

Reduction of iron ores. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,771,971, 29.7.30. Appl., 16.6.28).—See B.P. 313,597; B., 1930, 994.

Reduction of ores, oxides, and the like. H. E. COLEY (U.S.P. 1,777,993, 7.10.30. Appl., 2.11.23. U.K., 10.11.22).—See B.P. 215,400; B., 1924, 601.

Manufacture of steel. E. H. GRAF, Assr. to LUSIFER PRODUCTS Co. (U.S.P. 1,777,081, 30.9.30. Appl., 23.11.28).—See B.P. 332,890; B., 1930, 952.

Manufacture of [sand] moulds [for casting iron]. W. KLEPSCH (U.S.P. 1,777,975, 7.10.30. Appl., 15.9.27. Ger., 29.6.26).—See B.P. 302,254; B., 1929, 133.

[Refractory materials for] refining industrial metals in the molten state. J. A. JACK (U.S.P.

1,777,617, 7.10.30. Appl., 31.3.28. U.K., 6.3.28).—See B.P. 312,700; B., 1929, 645.

Refining of copper. H. H. ALEXANDER (U.S.P. 1,776,826, 30.9.30. Appl., 5.7.28).—See B.P. 310,356; B., 1929, 525.

Protection of metal bodies from corrosive agencies. P. F. SCHADE, Assr. to CHEMIEPROD. GES. M.B.H. (U.S.P. 1,777,107, 30.9.30. Appl., 11.11.27. Ger., 27.7.27).—See B.P. 293,835; B., 1929, 856.

Manufacture of bimetallic strips, plates, or the like. F. W. MILLER (U.S.P. 1,776,364, 23.9.30. Appl., 11.11.27. U.K., 26.11.26).—See B.P. 266,645; B., 1927, 338.

Etching of printing forms. J. BEKK, Assr. to BEKK & KAULEN CHEM. FABR. G.M.B.H. (U.S.P. 1,776,535, 23.9.30. Appl., 18.1.29. Ger., 24.1.28).—See B.P. 304,685; B., 1929, 687.

Apparatus for cleaning articles of metal etc. [by solvents]. CHEM. WERK ZÜRICH A.-G. (B.P. 313,599, 23.5.29. Switz., 15.6.28).

[Die]-casting of metals or alloys [with an aluminium base]. METAL CASTINGS, LTD., and A. H. NICHOLSON (B.P. 334,714, 12.9.29).

Welding [apparatus]. BUDD WHEEL Co., Assees. of J. P. TARBOX, A. F. HANSON, and G. B. REED (B.P. 335,491, 24.1.30. U.S., 26.1.29).

Fusing coatings to metal sheets. AJAX METAL Co., Assees. of H. NEUHAUSS (B.P. 335,769, 24.10.29. U.S., 24.10.28).

Carrying out reactions with gases etc. (B.P. 334,926).—See I.

XI.—ELECTROTECHNICS.

Instrument ["stato-ionometer"] for measuring and recording the concentration of hydrogen and other ions, employing the thermionic valve. P. WULFF and W. KORDATZKI (Chem. Fabr., 1930, 329–330, 342–345, 353–355).—The use of the emission valve in the measurement of, e.g., p_H offers the possibility of automatic recording of results as variations in the anode current. This is not the case with any form of bridge potentiometer. For a practical instrument it is essential that the zero point shall be reasonably constant and the current consumption should be as small as possible. Arrangements proposed by various investigators are discussed. The authors' apparatus employs three batteries (one for compensating current) and one valve, but the batteries may be very small in size. The voltmeter is placed in circuit with the compensating battery, which is so adjusted that no current passes. As, however, the *E.M.F.* of the heating current gradually falls and the emission is also reduced, but not linearly, the zero position changes slowly and should be adjusted daily. The apparatus is provided with alternative connexions by which the complete galvanometer scale may represent from 250 to 1000 millivolts, or it may be graduated in p_H values for a given temperature. The apparatus should not be exposed to sudden temperature changes, but a temperature fall of a few degrees alters the zero by only 1 or 2 millivolts. A suitable

hydrogen electrode for use with the instrument is illustrated and curves obtained in the hydrolysis of methyl acetate with caustic soda and in the ferrocyanide titration of zinc are given. C. IRWIN.

Steel furnaces. CAMPBELL. **Welding.** SCHUSTER. **Tensile properties of conductor wires.** PESTER. **Melting of aluminium.** RUSS. **Test for corrosion.** AUBERT and PROT. **Transformation points in metals.** SCHULZE. **Electrodeposition of gold alloys.** GRUBE. **Silver plating.** SANIGAR. **Control of electrodeposition.** BALLAY. **Cobalt-nickel deposits.** GLASS-TONE and SPEAKMAN.—See X. **Ash determinations of cane and beet products.** FARNELL.—See XVII.

See also A., Oct., 1230, **Sensitising alkali metal photo-electric cells** (OLPIN). 1253, **Electrolysis of amalgams, fused silver-lead alloys, and bronzes** (KREMANN and others). 1258, "Explosive" **antimony** (STEINWEHR and SCHULZE; COHEN and COFFIN). 1265, **Coreless induction furnace** (ADCOCK). **Electric muffle with chromium-nickel wire heaters** (SEBERT). 1268, **Decomposition of hydrocarbons in the electrodeless discharge** (AUSTIN).

PATENTS.

Regeneration of [sulphated] lead plates of electric accumulators. E. FEIGELSON (B.P. 334,393, 18.9.29).—The sulphated plates are charged and discharged for a number of short periods, the charging current density being about one fifth normal, until the density of the electrolyte ceases to increase and the plates appear normal. J. S. G. THOMAS.

Electric dry cell. SIEMENS BROS. & CO., LTD., and C. F. EDWIN (B.P. 334,420, 12.10.29).—A carbon electrode surrounded with manganese dioxide is arranged in an electrolyte consisting of a paste composed of lead chloride, flour, and gum, and contained in a lead tube forming the other electrode. J. S. G. THOMAS.

Photosensitive cells. ARCTURUS RADIO TUBE CO., ASSEES. of S. RUBEN (B.P. 334,409, 30.9.29. U.S., 16.3.29).—A cuprous oxide electrode is arranged in contact with an electrolyte, *e.g.*, hydrogen peroxide, from which nascent oxygen is evolved independently of electrolytic action. J. S. G. THOMAS.

Thermionic cathodes. WESTINGHOUSE ELECTRIC & MANUF. CO., ASSEES. of G. P. HALLIWELL (B.P. 334,460, 5.12.29. U.S., 14.12.28).—An intimate mixture of a metal of the nickel group, or its oxide, and a deoxidising agent, *e.g.*, titanium, tungsten, molybdenum, thorium, vanadium, tantalum, silicon, manganese, aluminium, chromium, or metals of the rare-earth group, is pressed into bars or slugs and heated in a non-oxidising atmosphere at a sufficiently high temperature to cause sintering and recrystallisation. J. S. G. THOMAS.

Electrically heated [tunnel] kiln. B. J. MOORE and A. J. CAMPBELL, ASSRS. to GIBBONS BROS., LTD. (U.S.P. 1,776,321, 23.9.30. Appl., 26.11.27).—See B.P. 270,035; B., 1927, 492.

Electric cell. A. HELBRONNER and E. DUTT (U.S.P. 1,777,202, 30.9.30. Appl., 19.4.28. Fr., 11.8.27).—See B.P. 295,589; B., 1930, 153.

Impregnation of dielectric materials. GEN. ENG. CO. (RADCLIFFE), LTD., and S. TAYLOR (B.P. 335,576, 25.6.29).

Electrolytic writing and drawing paper (B.P. 313,561).—See V. **Pure phosphoric acid** (B.P. 334,790). **Dicalcium phosphate** (B.P. 335,007). **Chlorine** (U.S.P. 1,746,542).—See VII. **Furnace for melting metals** (B.P. 334,201). **Treatment of copper sulphide ores** (U.S.P. 1,761,641). **Alloys** (B.P. 335,081). **Furnace for aluminium** (U.S.P. 1,761,442). **Alloy for cable sheaths** (B.P. 314,522). **Recovery of tin from alkaline lyes** (B.P. 334,738). **Nickel plating** (U.S.P. 1,761,948).—See X. **Rubber-paper products** (U.S.P. 1,756,035).—See XIV. **Ozonising-tobacco** (U.S.P. 1,757,477).—See XX.

XII.—FATS; OILS; WAXES.

Component glycerides of stillingia (Chinese vegetable) tallow. T. P. HILDITCH and J. PRIESTMAN (J.S.C.I., 1930, 49, 397—400 r).—Samples of stillingia tallow from Chinese sources and from plantations in Florida and Texas have been investigated with reference to fatty acid composition and glyceride structure. The crude American plantation sample had sap. equiv. 260.4, acid value 53.5, iodine value 20.0, and setting point 47.3°, whilst the neutralised fat had sap. equiv. 275.4, iodine value 22.1, and setting point 48.2°. The setting points are much higher than those recorded hitherto for stillingia tallow. Of two Chinese samples, I (neutralised) had sap. equiv. 260.9, iodine value 26.6, and setting point 25.6°, and II (a very crude sample) was purified by extraction with warm alcohol, the residual fat (77%) having sap. equiv. 270.6, acid value nil, iodine value 14.4, and setting point 42.4°, whilst the alcohol-soluble portion (23%) had sap. equiv. 254.0, acid value 87.6, and iodine value 29.8. The fatty acids present, determined by ester-fractionation, were mainly palmitic (57—69%) and oleic (21—34%), with small amounts of myristic (3—6%) and stearic (1—3%) acids; non-fatty matter of a semi-volatile ester nature appears to accompany the fat in small quantities. According to the relative amounts of saturated and oleic acids present in the whole fat, the fully-saturated glycerides are present to the extent of about 25—35%; in the mixed saturated-unsaturated glycerides, mono-oleodisaturated glycerides (mainly oleodipalmitins) predominate. The American sample contained 27.6% of fully-saturated glycerides (largely tripalmitin) and over 60% of mono-oleodisaturated glycerides (chiefly oleodipalmitins). Stillingia tallow should be an excellent candle- or soap-making material, but its value as a confectionery fat is uncertain in consequence of the presence of comparatively high proportions of tripalmitin.

Determination of the detergent power of soaps. E. BOSSHARD and H. STURM (Chem.-Ztg., 1930, 54, 762—763).—A standard soiled fabric is prepared by precipitating a known amount of iron oxide (from iron ammonium alum solution) on a flannel strip. The strip is washed by a standardised procedure, dried, and ashed, and the residual iron oxide in the ash is determined by the Zimmermann-Reinhardt method. The

amount of iron oxide removed by the washing operation, expressed as a percentage of the total soil, represents the detergent power of the soap used. If desired, the soiled test strip may be oiled by treating with a known amount of an ethereal solution of petroleum oil, and the fat left after washing determined by extraction. Reproducible results are obtained. The addition of perborate to a curd soap lowered the detergent value.

E. LEWKOWITSCH.

Is degreasing of raw bones with liquid solvents practicable? A comparison with oil-seed extraction. G. GÜNTHER (Chem.-Ztg., 1930, 54, 761—762).—Extraction of substances of such texture as raw bones with liquid media is impracticable; penetration and fat-extraction is incomplete, and displacement of the solvent from the relatively cold material by steam would lead to excessive condensation and spoil the material for further working up.

E. LEWKOWITSCH.

Greek tobacco-seed oil. J. D. KANDILIS and N. S. KARNIS (Praktika, 1929, 4, 475—481; Chem. Zentr., 1930, i, 2820).—The (drying) oil (36.85%) had: iodine value 117.8—137.9, d_{20}^{20} 0.9253—0.9440, n_D^{25} 1.4735—1.4828, acid value 2.25—16.93, saponif. value 186.8—201.6, Hehner value 95.21—96.34, Reichert-Meissl value 0.32—2.03, Polenske value 0.15—0.30.

A. A. ELDRIDGE.

Laundering. ZAKARIAS.—See VI. **Viscosity of linseed oil.** BARRY.—See XIII.

See also A., Oct., 1271, **Partial hydrogenation of linolenic acid** (BAUER and ERMANN). 1308, **Fat from *Cypridina*** (KOTAKE and KIMOTO). **Liver oil of thresher shark** (LOVERN). 1323, **African *Chaulmoogra* species** (PERROT and FRANÇOIS). **Wallflower-seed oil** (VAN LOON). 1326, **Determination of fats** (PINCUSSEN and KOLODNY).

PATENTS.

Removal of slime from oils and fats. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,089, 21.10.29).—The slime is precipitated by treating the fats, at temperatures from 70° to 200°, with small quantities of anhydrous sulphonic acids of aromatic hydrocarbons (e.g., β -naphthalenesulphonic acid) and, if desired, in an inert atmosphere and/or in the presence of adsorbents.

E. LEWKOWITSCH.

Treatment of [unsaturated] oils and other glycerides. W. C. ARSEM, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,760,535, 27.5.30. Appl., 22.5.29).—The polymerisation ("bodying") of drying or semi-drying oils can be greatly accelerated by the use of about 1% of an organic amine (preferably benzidine) as catalyst.

E. LEWKOWITSCH.

Fatty acid esters (B.P. 315,832).—See III. **Emulsions** (U.S.P. 1,758,651).—See XX. **Grease from garbage** (U.S.P. 1,764,390).—See XXIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Modern [lacquer] finishes. F. KOLKE (Farben-Ztg., 1930, 35, 2569—2570, 2618—2619).—An account is given of the general properties and methods of application of the following "novelty finishes":

colourless cracking lacquers, crystallising lacquers (oil and cellulose bases), crinkle, grain, and Lymnato finishes. The effects obtained are illustrated.

S. S. WOOLF.

Viscosity of lithographic varnishes and linseed oil. T. H. BARRY (J. Oil Col. Chem. Assoc., 1930, 13, 220—230).—The viscosity-temperature relations of representative grades of litho varnishes, alone and mixed with each other and with linseed oil, were observed over the temperature range 20—90°, using the falling-sphere method of determination. Even with mixtures of extreme grades no departure from regularity was noted, the curves being of the linear-logarithmic type throughout. From the absence of the relatively great increase in viscosity invariably produced when small additions of the colloid are made to colloidal systems, it is inferred that, within the limitations of the experiment described, the dispersions are of the "molecular solution" type.

S. S. WOOLF.

Rosin size manufacture. C. N. RIDLEY (Ind. Chem., 1930, 6, 369—371).—A brief summary of current methods of rosin size manufacture, including the Delthirna process. The effect of hard water on rosin size is illustrated.

T. T. POTTS.

Protection of light aluminium alloys. AUBERT and PIGNOT.—See X. **Hop resins.** GUTHRIE and PHILIP.—See XVIII.

PATENTS.

Production of lacquers and other coatings for absorptive material. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,567, 6.5.29).—Solutions of practically water-insoluble cellulose derivatives in practically water-insoluble organic solvents or solvent mixtures, which may contain difficultly volatile constituents, are emulsified in aqueous solutions of organic water-soluble, film-forming, and protective colloids which are free from albumin, e.g., polymerised vinyl alcohols, starch, water-soluble urea-formaldehyde condensation products, ammonium salts of conversion products of phenol-formaldehyde condensation products with halogen fatty acids, methylcellulose. Softening agents, other emulsifying or wetting agents, fillers, colouring materials, resins, etc. may be incorporated.

S. S. WOOLF.

Casein-containing plastic composition. W. W. CHRISTMAS, Assr. to C. C. HINES (U.S.P. 1,758,500, 13.5.30. Appl., 9.6.24).—Powdered casein (1 lb.), powdered calcium hydroxide (1 lb.), calcium carbonate (1 lb.), acetic acid ($\frac{1}{2}$ oz.), an inorganic filler (3 lb.), and an organic fibrous filler (3 pints) are mixed with water to form a flowing mass.

D. WOODROFFE.

Treating waste and low-grade [zinc oxide] pigments. H. REINHARD (U.S.P. 1,776,689, 23.9.30. Appl., 8.9.28. Ger., 9.9.27).—See B.P. 321,675; B., 1930, 204.

Black azo pigments (B.P. 311,400).—See IV. **Adhesive films etc.** (B.P. 333,194).—See V. **Coated fabrics** (U.S.P. 1,746,162).—See VI. **Nitrocellulose-rubber composition** (U.S.P. 1,746,142).—See XIV. **Impregnation of leather etc.** (B.P. 333,759).—See XV. **Light-sensitive plates** (B.P. 315,355).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber mills and Banbury mixers. C. F. SCHNUCK (Ind. Eng. Chem., 1930, 22, 1007—1010).—A description is given of the essential features of construction and of the modern working and respective advantages of the ordinary mill and the Banbury machine for the breakdown and compounding of rubber. D. F. TWISS.

Effect of vulcanising temperature on the properties of a rubber-sulphur mixture. C. R. PARK (Ind. Eng. Chem., 1930, 22, 1004—1007).—Using a mixture of rubber (100 pts.) and sulphur ($6\frac{1}{4}$ pts.) and vulcanising over a temperature range of 135.5—162°, tests on tensile strength, extensibility, tear-resistance, and ageing, whether natural or accelerated, indicate that, within the limit of experimental error, the same result is obtained at equivalent degrees of vulcanisation whatever is the temperature of vulcanisation. The temperature coefficient of vulcanisation is 2.5 per 10°. Although accelerated ageing tests are of practical value, there is a marked difference between the changes induced by ageing naturally, in a Geer oven, and in a Bierer-Davis bomb, respectively. D. F. TWISS.

Surface application of age-resisters to vulcanised rubber versus mill incorporation prior to vulcanisation. W. L. SEMON, A. W. SLOAN, and D. CRAIG (Ind. Eng. Chem., 1930, 22, 1001—1004).—Comparative ageing tests at 70°, in an oven and in oxygen under a pressure of 21.1 kg./cm.², on a mixture of high zinc oxide and sulphur content and on a non-blooming motor-tread quality, respectively, using a large number of anti-oxidants, shows that the relative effectiveness of the various anti-ageing agents varies considerably with the test applied. Surface application of the anti-oxidant in solution, however, is generally less effective than incorporation by milling (cf. Moureu, Dufraisse, and Lotte, B., 1930, 625), the main advantages of the former being the possibility of treating finished articles and the avoidance of complications from interference by the anti-oxidant with the processing or vulcanisation of the stock. Incorporation by milling is easier, safer, and more effective generally. D. F. TWISS.

PATENTS.

(A) Production of aqueous rubber emulsion. (B) Dispersion of coagulated bodies. (C) Production of aqueous rubber dispersions. W. B. PRATT, Assr. to DISPERSIONS PROCESS, INC. (U.S.P. 1,755,890—2, 22.4.30. Appl., [A] 11.4.23, [B] 10.7.24, [C] 14.9.27).—(A) Raw or compounded rubber can be converted into an aqueous emulsion or artificial latex by dissolving, together with a saponifiable substance such as oleic acid, in a volatile solvent, *e.g.*, benzene, and dispersing in water containing an alkali or amine; the resulting soap facilitates the dispersion process. The volatile solvent can be eliminated by evaporation and the bulk of the soap and non-caoutchouc constituents may subsequently be removed by centrifuging the dispersion and separating the aqueous portion, after which the cream may be rediluted. (B) Rubber (or gutta-percha), compounded or otherwise, is dispersed in water by adding up to 15% of an inorganic colloid, such as colloidal clay, and subjecting in a mixing machine to a kneading and stretching operation with the gradual addition of water.

A paste, cream, and milk are thus successively obtainable. Although these dispersions are not easily coagulated by acid, they yield a coherent film on removal of water. (C) A hydrophilic colloid, such as Irish moss jelly, is worked into rubber together with water, so as to effect a preliminary separation of the rubber globules; the mass is then kneaded in a dough mixer with the gradual addition of a solution of the reaction product obtained by heating glue or albumin with caustic alkali. If the amount of Irish moss used in the first operation is sufficient, the second colloid is not necessary and water alone may be added during the kneading operation. The product is a smooth paste coagulable by acids.

D. F. TWISS.

Concentrating [rubber] latex. H. W. BANKS, Assr. to UNITED STATES RUBBER Co. (U.S.P. 1,755,379, 22.4.30. Appl., 28.11.24).—Rubber latex mixed with colloidal materials, *e.g.*, gelatin or gum arabic, gradually separates into two layers, the lower containing little rubber, whilst the upper has a high concentration of rubber and a correspondingly smaller content of non-caoutchouc substances; the process may be repeated. Such concentrated latex yields highly transparent rubber films. D. F. TWISS.

Method of forming tacky rubber compound and products. M. C. TEAGUE, Assr. to AMER. RUBBER Co. (U.S.P. 1,746,875, 11.2.30. Appl., 9.6.24).—An acidic ingredient capable of imparting tackiness to rubber, *e.g.*, pine tar, rosin, or coal tar, is converted into an easily decomposable, water-soluble form, *e.g.*, by treatment with a volatile base such as aqueous ammonia. The solution is then added to rubber latex. During the eventual drying operation the volatile base is lost, leaving the ingredient uniformly distributed in the rubber from the latex. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER Co., LTD., D. F. TWISS, G. W. TROBRIDGE, and W. E. GORHAM (B.P. 335,271, 25.6.29).—Cellulosic or fibrous containers are provided with protective coatings by applying an aqueous dispersion of rubber or analogous material mixed with an aqueous dispersion of wax (such as paraffin wax) or of a mixture of waxes, the wax preferably amounting to 20—95% of the dry rubber-wax mixture. Vulcanisation is not necessary. A similarly treated lid becomes sealed in position on such a container when warmed. D. F. TWISS.

Manufacture of surface-finished rubber goods. H. W. EMERY, Assr. to ARCHER RUBBER Co. (U.S.P. 1,762,165, 10.6.30. Appl., 3.11.26).—A permanent, lustreless appearance is imparted to rubber-coated sheeting by applying to the surface of the compounded rubber a powder such as starch, asbestine, talc, or mica, with or without previous reduction of tackiness of the surface by treatment with soap solution. After removal of surplus powder by washing, *e.g.*, with a volatile solvent, such as carbon tetrachloride or benzene, the sheeting is dried and heat-vulcanised, the surface finally being hardened by halogenation, *e.g.*, by contact with an atmosphere or solution containing free halogen or a sulphur halide. D. F. TWISS.

Production of combinations of rubber and paper

and product obtained thereby. R. P. ROSE and H. E. CUDE, Assrs. to GEN. RUBBER CO. (U.S.P. 1,756,035, 29.4.30. Appl., 9.10.25).—A waterproof paper suitable for electric insulation is made by adding to paper pulp a protective colloid, such as alkaline casein, a dispersion of a paper waterproofing material, such as aluminium resinates or a synthetic resin, and latex. The addition of a coagulant then effects deposition of the dispersed material and rubber on the fibres, which are subsequently sheeted or shaped. D. F. TWISS.

[Nitrocellulose]-liquefied rubber composition and its manufacture. H. P. BUTLER (U.S.P. 1,746,142, 4.2.30. Appl., 10.5.28).—A solution of rubber in a suitable solvent, *e.g.*, toluene, with or without the addition of carbon tetrachloride, is heated under reflux for several hours at 100–120°. The solution, of which the viscosity has thus been decreased, is then mixed with a cellulose preparation in order, *e.g.*, to produce a homogeneous solution containing $\frac{1}{2}$ –1 pint of soluble pyroxylin with 2–12 oz. of liquefied rubber. D. F. TWISS.

Curing of moulded rubber articles. H. R. MINOR, Assr. to INDUSTRIAL PROCESS CORP. (U.S.P. 1,746,357, 11.2.30. Appl., 28.8.26).—In the rapid vulcanisation of moulded rubber articles such as motor tubes, a preheated inert gas, *e.g.*, carbon dioxide, is applied as the internal pressure medium; the gas is preferably heated by use of a portion of the steam withdrawn from the jacket of the mould. D. F. TWISS.

Rubber-vulcanisation process. W. P. TER HORST, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,756,315, 29.4.30. Appl., 28.11.27).—Unsymmetrical trisubstituted guanidines of the type $\text{NRR}'\cdot\text{C}(\text{NH})\cdot\text{NHR}$, where R and R' are aryl and alkyl radicals, respectively, *e.g.*, diphenylethylguanidine obtained by the interaction of phenylecyanamide and ethylaniline hydrochloride, are effective vulcanisation accelerators. D. F. TWISS.

(A) **Rubber-vulcanisation accelerator.** (B) **Vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,747,187–8, 18.2.30. Appl., [A] 6.10.25, [B] 11.10.28).—(A) Incorporation of basic organic accelerators, such as di-*o*-tolylguanidine, is facilitated by employing these in the form of their compounds with fatty acids, *e.g.*, stearic acid. (B) Vulcanisation accelerators are prepared by treating the reaction products of an aliphatic aldehyde-ammonia, *e.g.*, hexamethylenetetramine or crotonaldehyde-aminonia, and a mercaptobenzthiazole with an aliphatic aldehyde such as formaldehyde or aldol. D. F. TWISS.

Treatment of rubber. P. I. MURRILL and W. W. EVANS, Assrs. to R. T. VANDERBILT CO., INC. (U.S.P. 1,755,703, 22.4.30. Appl., 23.9.27).—The condensation product of an aldehyde, such as formaldehyde or aldol, with a diarylalkylenediamine, *e.g.*, diphenylethylenediamine, is used as an antioxidant. D. F. TWISS.

Manufacture of vulcanised rubber and of materials for use therein. C. J. T. CRONSHAW and W. J. S. NAUNTON, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,777,352, 7.10.30. Appl., 15.8.27. U.K., 3.9.26).—See B.P. 280,661; B., 1928, 101.

Coated fabrics (U.S.P. 1,746,162).—See VI. **Imitation leather** (B.P. 334,361).—See XV.

XV.—LEATHER; GLUE.

Analysis of leather. A. T. HOUGH (Cuir tech., 1930, 23, 47–48; Chem. Zentr., 1930, i, 2506).—The official method for the determination of ash is inexact. The insoluble constituents of the ash should be determined on fat-free leather which has been extracted with water. The extracted fat should also be ashed. The soluble ash should be heated to fuming with sulphuric acid in order to deduce the quantity of tans. A. A. ELDRIDGE.

Effect of temperature on single-bath chrome tanning. C. OTIN and G. ALEXA (Cuir tech., 1930, 23, 53–56; Chem. Zentr., 1930, i, 2504).—Quantities of hide powder (10 g.) were shaken for 48 hrs. with 200 c.c. of chromium sulphate solution of various concentrations at 25°, 32°, and 40°, the mixture being filtered with suction and the residue washed with distilled water (200 c.c.). The quantity of dissolved chromic oxide and sulphur trioxide increased regularly with the concentration of the tanning solution. Rise of temperature increased the quantities adsorbed, particularly from dilute solutions owing to hydrolysis. A. A. ELDRIDGE.

Diffusion of oak and chestnut extract into gelatin jellies. M. DE LA BRUÈRE (Cuir tech., 1930, 23, 73–75; Chem. Zentr., 1930, i, 2505).—Differences in appearance, due to differences in p_H value, are described. A. A. ELDRIDGE.

Photographic gelatin. LAWRIE.—See XXI.

See also A., Oct., 1295, **Tea tannin** (TSUJIMURA). 1303, **Combination between dyes and gelatin granules.** (RAWLINS and SCHMIDT).

PATENTS.

Tanning preparation. R. W. FREY, Assr. to U.S.A. (U.S.P. 1,757,040, 6.5.30. Appl., 31.7.28).—Ferrochrome or other iron-chromium alloy is dissolved in hydrochloric acid, the solution rendered basic by the addition of an alkali carbonate, and the product used in the tanning of leather. On completion of the tannage an oxidising agent is added to the tan liquor to convert Fe^{++} into Fe^{+++} . D. WOODROFFE.

Impregnation of leather or waste leather and manufacture of articles therefrom. J. TAYLOR and A. V. KELLER (B.P. 333,759, 29.8.29).—A condensation product of urea, thiourea, substituted ureas or their derivatives, or phenolic compounds or their derivatives with formaldehyde, paraformaldehyde, or other aldehydic compounds and derivatives is prepared, *e.g.*, by mixing the paraformaldehyde with water to a paste, adding the finely-powdered urea etc., and concentrating the product at not above 90° until a transparent syrup is produced. This is mixed with the leather or waste leather, allowed to dry, ground to powder, with or without the addition of pigment or colouring matter, and moulded. D. WOODROFFE.

Glueing of leather, and adhesive materials therefor. W. STELKENS (B.P. 314,867, 27.6.29. Ger., 3.7.28).—The surfaces to be glued together are first moistened with a solvent for the adhesive material or are softened by heat alone or in conjunction with the moistening process. The adhesive materials, which consist of mixtures of finely-powdered dry cellulose esters and ethers, *e.g.*, acetylcellulose, with solid softening

agents, *e.g.*, triphenyl phosphate, or which may be a film, *e.g.*, nitrocellulose, phenyl phosphate, and butyl phthalate, are then placed between them. Finally the surfaces are subjected to pressure. D. WOODROFFE.

Manufacture of imitation leather having a natural grain. O. G. BOHLIN (B.P. 334,361, 10.8.29).—The surface of imitation leather consisting of compounded caoutchouc and leather fibres is subjected to cold vulcanisation, the whole being subsequently subjected to hot vulcanisation. This procedure ensures a superficial layer which is more solid and more highly vulcanised than the underlying mass. D. F. TWISS.

Manufacture of glue. I. F. LAUCKS and C. N. CONE, Assrs. to I. F. LAUCKS, INC. (U.S.P. 1,757,805, 6.5.30. Appl., 13.9.24).—Vegetable protein-containing material, *e.g.*, ground soya-bean cake, is mixed with hydrated lime, powdered rosin or resins, caustic soda, water, sodium silicate, and potassium permanganate, the amount of the last-named compound being based on that of the protein matter present.

D. WOODROFFE.

Manufacture of adhesives. RÖHM & HAAS A.-G. (B.P. 311,339, 8.5.29. Ger., 9.5.28).—The use of one or more polymerised esters of acrylic acid, dissolved in acetone or formed into thin sheets (*e.g.*, a 10% solution of polymerised ethyl acrylate in acetone), is claimed. [Stat. ref.] D. WOODROFFE.

Treatment of hides, skins, etc. D. L. LEVY (B.P. 335,252, 21.6.29).—See U.S.P. 1,742,514; B., 1930, 434.

Tanning and tanning composition. F. S. LOW, Assr. to F. S. LOW and A. W. BERRESFORD (U.S.P. 1,764,516—7, 17.6.30. Appl., 9.8.28).—See B.P. 256,979; B., 1927, 497.

Polyazo dyes [for leather] (B.P. 313,562).—See IV. **Casein plastic** (U.S.P. 1,758,500).—See XIII.

XVI.—AGRICULTURE.

Microbiological analysis of soils. T. BAUMGÄRTEL and H. BUTENSCHÖN (Landw. Jahrb., 1930, 72, 256—268).—Previous work (Baumgärtel and Simon, B., 1930, 341) is extended, and results are confirmed by cylinder experiments. Cultures in Beijerinck's medium from soils treated with increasing amounts of potash fertilisers show increasing development of *Azotobacter chroococcum* and, where heavy dressings of potash have been used, larger numbers of protozoa. A. G. POLLARD.

Biochemical researches on fertile soil. II. **Adsorption capacity.** A. J. J. VANDE VELDE and A. VERBELEN (Natuurwetensch. Tijds., 1930, 12, 147—154; cf. B., 1930, 524).—The methods proposed for determination of adsorption capacity of soils by other authors are outlined, and results given of determinations carried out with four different soils with (1) aniline dyes, viz., methylene-blue, methyl-violet, and methyl-green; (2) peptone solution; and (3) centrifuged milk. Whilst the results were not concordant, the behaviour towards each of the reagents used was of the same order.

S. I. LEVY.

Chemical phases of submerged soil conditions. W. O. ROBINSON (Soil Sci., 1930, 30, 197—217).—

Examination of soil solutions of bog and similar soils is recorded. These differ from aerated soil solutions in containing relatively high proportions of ferrous and manganous carbonates, calcium, magnesium (brought into solution by microbiologically produced carbon dioxide), together with some hydrogen sulphide. Submergence of soils for short periods does not increase their acidity, excepting that due to carbon dioxide. Gas production is characteristic of all submerged soils and is largely influenced by algæ of the *Chlamydomonas* group. Hydrogen and methane are produced in the absence of algæ, whilst in their presence carbon dioxide, nitrogen, and variable amounts of methane are formed.

A. G. POLLARD.

Soils in the plastic state. G. B. BODMAN and M. TAMACHI (Soil Sci., 1930, 30, 175—195).—The Atterberg method of determining soil plasticity is modified and a method devised for determining the moisture content at which soils exhibit similar "stiffness." Relationships exist between these values and the air-dry moisture contents, the moisture equivalents, and the colloid contents for all degrees of stiffness. Organic and inorganic colloids produce similar effects on the moisture-stiffness relationships, but the p_H value of soil is without influence in this respect.

A. G. POLLARD.

Analytical methods in base-exchange investigations on soils. C. J. SCHOLLENBERGER and F. R. DREIBELBIS (Soil Sci., 1930, 30, 161—173).—Analytical details and technique are recorded for the leaching of exchangeable bases from soil with ammonium acetate solution and the determination in the extract of sodium, potassium, ammonium, magnesium, calcium, aluminium, and manganese.

A. G. POLLARD.

[Relation between] soil, fertilisers, and chemical composition of plants. D. J. HISSINK (Chem. Weekblad, 1930, 27, 529—533).—Examples are given to show that whilst the proportions of mineral constituents in the plant depend primarily on the plant itself, they are also dependent on the minerals present in the soil or added as fertilisers. Analysis of the plant and the soil cannot, however, suffice to determine the nature and amount of fertiliser which will produce the best crop.

S. I. LEVY.

Loss of residual chlorates from soil. C. R. MEGEE and R. W. LIPSCOMB (Mich. Quart. Bull., 1930, 12, 84—87).—The period necessary between application of sodium chlorate and sowing is considered.

CHEMICAL ABSTRACTS.

Ammonium sulphate and sodium nitrate in a sod orchard. R. D. ANTHONY (Penn. Agric. Exp. Sta. Bull., 1930, No. 249, 3—24).—Little difference between the effects of the two fertilisers was observed. When a heavy sod is present the tree suffers from lack of nitrates.

CHEMICAL ABSTRACTS.

Fertilisation of shade trees. II. **Conifers.** H. L. JACOBS (Davy Tree Expert Co. Res. Bull., 1930, 5, 15 pp.).—Advantageous chemical fertilisation is recorded.

CHEMICAL ABSTRACTS.

Comparison of some nodule-forming and non-nodule-forming legumes for green manuring. L. T. LEONARD and H. R. REED (Soil Sci., 1930, 30, 231—236).—No decided difference was observed in the

effects of nodule and non-nodule legumes used as green manures. A. G. POLLARD.

Legumes as a source of nitrogen in crop rotations. T. L. LYON (Cornell Univ. Agric. Exp. Sta. Bull., 1929, No. 500, 22 pp.).—Legume residues promote the availability of soil nitrogen more markedly than those of other mature crops. The nitrogen content of lucerne was unaffected by fertilisation with superphosphate and potassium chloride, but the crop was much heavier.

CHEMICAL ABSTRACTS.

Varying effect of lime on grassland with different schemes of manuring. W. E. BRENCHELEY (J. Min. Agric., 1930, 37, 663—673).—On grass land dressed repeatedly with dung only, liming decreased the hay yields. Where dung and artificial fertilisers had been used the ill-effects of lime were delayed for one or two seasons according to the amount of lime applied. On plots receiving only artificial fertilisers, liming tended to decrease the yield where sodium nitrate was the source of nitrogen, and to increase yields where ammonium sulphate was used. In the latter case there was a notable increase in the ratio of grass to weeds in the herbage. On unlimed soils treated with minerals and heavy dressings of ammonium sulphate, sodium silicate proved beneficial. In a season of severe frost and drought ammonium sulphate tended to kill much of the grass. Liming to a large extent prevented this injury. A. G. POLLARD.

Influence of manuring and spacing on the soundness of potatoes. T. REMY, F. VON MEER, and H. VÖLKER (Landw. Jahrb., 1930, 72, 209—246).—Heavy dressings of potash do not affect the quality of potatoes adversely, but extreme deficiency of potash tends to produce tubers of greater soundness. Increasing applications of nitrogenous fertilisers increase the contents of both total and amino-acid-nitrogen and of the nitrogen precipitable by copper hydroxide in the tubers. The ratio of the three quantities is not altered in a definite manner, but the proportions of amino-acid- and precipitable nitrogen tend to increase with larger dressings of nitrogen. No relationship exists between soundness and the quantity and nature of the nitrogenous substances in the tubers. A. G. POLLARD.

Effect of harvesting in different stages of ripeness on the valuable constituents of cereals. F. BERKNER and W. SCHLIMM (Landw. Jahrb., 1930, 72, 269—291).—Late-harvested grain has the greater dry weight per 1000 grains. There is a migration of starch to the grain in the later stages of ripening and a rapid increase in the protein content between the "milky" stage and full ripeness. A. G. POLLARD.

New sulphur-resin spray. W. NEWTON and R. J. HASTINGS (Sci. Agric., 1930, 11, 26—28).—Sulphur (4 pts.), resin (4 pts.), potassium hydroxide (7 pts.), and water (1 pt.) by wt. are mixed together. Sufficient heat is developed to fuse the mass, which must be vigorously stirred to prevent charring. The melt is poured into thin layers to cool, and is subsequently dissolved in water (16 lb. per 100 gals.). The spray has good spreading and adhesive properties and is an effective fungicide and contact insecticide. Satisfactory trials are recorded against *Botrytis tulipæ* and also aphids, mites, and red spider. A. G. POLLARD.

Properties and composition of the water-soluble fruit-tree carbolic sprays. J. WEICHERTZ (Chem.-Ztg., 1930, 54, 702—704).—Various commercial preparations (phenol content 5—13%) contained 5—9% of water; a further 4—5.5% could be added before the clear solutions showed turbidity. The addition of water produces an unstable water-in-oil emulsion; this gradually disappears to give a homogeneous solution (at, e.g., 54.2% water content) which on further addition of water leads to a very stable oil-in-water emulsion (cf. A., 1929, 1379). The view that the injurious action of the sprays is attributable to the presence of high-boiling tar-products, e.g., anthracene oil, must be abandoned (cf. Neodendrin preparation).

E. LEWKOWITSCH.

Organic nitrogen from agricultural waste. DIEGO. —See II. Nicotine insecticides and vine culture. FERRÉ.—See XVIII.

See also A., Oct., 1322, **Influence of temperature on composition and quality of peas** (BOSWELL). 1323, **Improvement of metabolism of plants by physiological culture** (BARBIERI). **Development of seedlings from seeds** (AXENTSEV). 1326, **Precipitation of silicic acid in plants** (FREY-WYSSLING).

PATENTS.

Treatment of alfalfa [lucerne], clover, and other hard-shelled seeds to promote prompt germination and remove weed seeds therefrom. H. R. WARREN, ASSR. to WARREN-TEED SEED CO. (U.S.P. 1,756,497, 29.4.30. Appl., 15.11.28).—Seeds of the same kind but of varying hardness are soaked in water or glucose solution (*d* 1.18) at 21—46°, and when the soft seeds have swollen, these are removed by flotation or screening and dried. The remaining seeds are scarified, soaked, and separated as before, and the balance of hard seeds is again scarified.

A. G. POLLARD.

Treatment of seeds to secure uniform germination. J. L. KELLOGG, ASSR. to WARREN-TEED SEED CO. (U.S.P. 1,756,488, 29.4.30. Appl., 15.11.28).—The process described in U.S.P. 1,756,497 (cf. preceding abstract) is improved by soaking the seed in a colour-fixing solution, e.g., alum, and subsequently scarifying in the presence of the solution. A. G. POLLARD.

Ripening of sugar cane. D. P. J. BURGIERES (U.S.P. 1,746,190, 4.2.30. Appl., 13.2.28).—To enhance the sucrose content of sugar canes in subtropical countries, their vegetative growth is checked shortly before harvesting by application of powdered lime or other suitable finely-divided substance to the tops of the plants, so that the "buds" are attacked without being destroyed. J. H. LANE.

Treatment of alfalfa [lucerne], clover, and other hard-shelled seeds to promote prompt germination and remove weed seeds therefrom. WARREN-TEED SEED CO. (B.P. 335,102, 1.11.29. U.S., 15.11.28).—See U.S.P. 1,756,488 and 1,756,497; preceding.

Fertiliser with a base of phosphate and sulphur. B. BODRERO (U.S.P. 1,777,908, 7.10.30. Appl., 15.7.26. Ger., 4.11.25).—See B.P. 262,017; B., 1927, 171.

Dicalcium phosphate (B.P. 335,007).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Drying of [sugar] beetroots by the Oxford process. P. ORTH (Bull. Assoc. Chim. Sucr., 1930, 47, 241—247).—The construction and operation of the dryer in use at Eynsham beet-sugar factory are described. The drying process is considered satisfactory. A control over the period Oct. 9—Dec. 25, 1929, showed an average increase of only 0.089% of invert sugar (calc. on fresh beets) as the result of the process, the weekly averages ranging from 0.037 to 0.101%. J. H. LANE.

Dextrorotatory matters other than sucrose in [French] beets harvested in 1929—1930. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 247—251).—Analysis of 32 samples of beets at various dates throughout this campaign showed an average direct polarisation (by hot aqueous digestion) of 15.03, compared with average sucrose contents of 14.19, 14.36, and 14.47%, as determined by the Herzfeld-Creydt, Clerget, and Sailard methods, respectively (cf. B., 1930, 343). Part of this abnormal difference is attributed to the presence of raffinose and part to the influence of nitrogenous substances. Sidersky, however, considers that it is due to pectic substances, which are dissolved to a greater extent in analysis by hot aqueous digestion than in the technical diffusion process. In the preceding three campaigns the author found that the total amount of sugar entering a factory, as calculated from the direct polarisation of the beets, agreed closely with that calculated from the polarisation of the diffusion juice, with due allowance for losses in diffusion, but last campaign the latter control indicated 1.24% less sugar (calc. on beets) than the former, and the actual yield of sugar confirmed the lower value rather than the higher. J. H. LANE.

Evolution of ammonia during evaporation [of beet-sugar juices]. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 252—254).—The amount of ammonia evolved in the working of beet juices is estimated at about 382 kg. per 1000 tons of beets, and some suggestions are made for its recovery. J. H. LANE.

Physico-chemical purification of sugar products by activated carbons in presence of sulphurous and phosphoric acids. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 259—263).—Beet syrup from the centrifugals, having purity 83% and alkalinity 0.03% CaO, was treated at 75—80° with 0.33 g. of Norit per 100 g. of sugar, sulphited to an alkalinity of 0.005% CaO, heated to 80—90°, and filtered. The colour was reduced in the ratio 180 : 100, the purity was raised by 2.02%, and there was no trace of inversion. When 0.05% of lime was used instead of Norit the decolorisation was only 180 : 130, and there was no increase of purity. Good results were obtained with 1.5 g. of carboraffin and 0.25—0.5 g. of commercial phosphoric acid per 100 g. of sugar. The rise in purity was over 1%, the decolorisation was 180 : 95 or 90, and filtration was greatly improved. The final alkalinities were 0.01—0.008% CaO, and no inversion occurred, although the syrup was heated at 90° for 25 min. J. H. LANE.

Ash determinations [of cane and beet products] by the conductivity method using the "Salometer." R. G. W. FARNELL (Internat. Sugar J.,

1930, 32, 347—349).—For such determinations the author recommends the "Salometer," which is accurate, easy to manipulate, portable, and independent of a lighting circuit. In a *résumé* of applications of the method in sugar work, reference is made to the control of clarification by comparison of the ash per 100° Brix of raw and clarified juices, to the control of continuous carbonatation processes, of remelts, and of fresh water for diffusion and boiler feed, to general refinery control, to the calculation of sugar yields from juices and massecuites based on the relative ash contents of the juices or syrups and corresponding centrifugal run-offs, to the influence of organic non-sugar on conductivity in relation to the various methods of cane juice clarification, and to use of ash determinations in controlling the sulphur dioxide content of raw cane sugars. J. H. LANE.

Semi-commercial production of xylose. W. T. SCHREIBER, N. V. GEIB, B. WINGFIELD, and S. F. ACREE (Ind. Eng. Chem., 1930, 22, 497—501).—Under the auspices of the U.S. Bureau of Standards, large-scale production of xylose was investigated in 1928 in an experimental factory plant. The raw material was cottonseed-hull bran, which consists of cottonseed hulls freed from "fuzz" and contains 30—35% of pentose. The material was heated with water under 1 atm. pressure for 2 hrs. to dissolve gums, then washed with cold water, agitated with cold 0.25*N*-sulphuric acid for 1 hr. to remove mineral matters, washed again, and digested with 0.2*N*-sulphuric acid under 10 atm. pressure for 2 hrs. to hydrolyse the xylan. After draining from the undissolved matters, the extract, containing xylose equal to about 26% of the weight of the original bran, was neutralised with milk of lime, filter-pressed, concentrated to *d* 1.28 in a vacuum evaporator, again filtered, concentrated to *d* 1.35 at 45—50°, and cooled gradually over a period of 30—48 hrs. in a rotating crystalliser. About half of the xylose in the extract was thus obtained as a brown crystalline product containing 90—94% of xylose and 1—2% of ash. One recrystallisation of this yielded about 65% of a white product of high purity. The process of manufacture is straightforward and does not require very elaborate plant. (Cf. Emley, B., 1930, 681.)

J. H. LANE.

Consumption of nitric acid in the oxidation of xylose. G. M. KLINE and S. F. ACREE (Ind. Eng. Chem., 1930, 22, 975—980).—The action of nitric acid on xylose with the production of dibasic sugar acids was investigated. The temperature used was 90—95° and oxides of nitrogen were recovered by scrubbing with caustic soda. The ratio of xylose to nitric acid was found to be about 1 : 2. Nitric acid may be used at 25—30% concentration. Too prolonged heating increases the loss of nitric acid, which under the best conditions amounts to about 15%. The sugar acids were approximately differentiated into mono- and di-basic acids by the use of bromophenol-blue and phenolphthalein as indicators. Some decomposition of dibasic acid into oxalic acid etc. seemed to occur.

C. IRWIN.

Conversion of sucrose into dextrose under the influence of moisture and heat, and the necessity of drying sugar after discharge from the centrifuges.

L. RAIMBERT (Bull. Assoc. Chim. Sucr., 1930, 47, 215—220).—A lecture. J. H. LANE.

Manufacture of rice starch. A. E. WILLIAMS (Ind. Chem., 1930, 6, 387—389).—Descriptive.

Use of "Lystonol" in beet-sugar manufacture. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 254—258).

Fermentation characteristics of *Rhizobium* species. WALKER and BROWN. **Cane molasses fermentation.** HILDEBRANDT and BOYCE.—See XVIII. **Boiler feed-water for sugar factories.** MOUSCADET.—See XXIII.

See also A., Oct., 1248, **Formation of starch paste** (GORBATSCHIEV). 1256, **Velocity of inversion of sucrose** (TAKETOMI). 1324, **Rye starch** (TAO).

PATENTS.

Treatment of raw sugar-beet shavings. ARCOS, LTD. From I. TISCHTSCHENKO and V. TOHEFRANOV (B.P. 329,112, 11.4.29).—Before desiccation, beet slices are treated with pulverised or dissolved alkali to prevent inversion of sucrose by acids in the juice or in the drying gases. J. H. LANE.

Treatment of beet juice for manufacture of sugar. J. FRIEDRICH (B.P. 331,308, 24.4.29).—Diffusion juice is heated under a pressure of 1—2.5 atm. and at temperatures up to 138° for 15 min. with about 0.15% of lime (calc. on beets). Impurities are thus precipitated, and the small quantity of lime remaining in solution may be removed by carbonating to the usual alkalinity or by treatment with oxalic acid at the boiling temperature. (Cf. B., 1930, 75.) J. H. LANE.

Manufacture of sugar. A. W. BULL, Assr. to DORR Co. (U.S.P. 1,755,165, 22.4.30. Appl., 4.12.28).—Beet juice is limed and carbonated practically simultaneously, so that it never becomes saturated with lime. Prior to liming and carbonating, raw juice is mixed with already carbonated juice, the precipitate in which adsorbs some of the impurities from the raw juice. The juice may, in part at least, be repeatedly circulated through a zone of liming and carbonation. These features are combined in a process of continuous carbonation in which beet juice, descending through a tall carbonation tank, is treated with a regulated supply of milk of lime in the upper part and carbonated by gas entering near the bottom. The juice leaves the tank at the bottom and a portion of it, e.g., one eighth, is conducted to filters or settling vessels while the remainder passes upwards through a second tank, where it is mixed with raw juice, and then flows again into the top of the carbonation tank. The volume of raw juice thus introduced into the circuit is equal to the volume of carbonated juice withdrawn as mentioned for filtration or sedimentation. J. H. LANE.

Manufacture or refining of sugar. J. B. TALBOT-CROSBIE and H. WISE (B.P. 331,314, 30.4.29).—Sugar solutions obtained by the extraction of desiccated beet or other sugar-containing plant, with or without previous purification, are used in place of water as solvent for raw sugar which is to be refined. J. H. LANE.

Syrup and its manufacture. J. K. DALE, Assr. to SUN-MAID RAISIN GROWERS OF CALIFORNIA (U.S.P.

1,746,993, 11.2.30. Appl., 11.3.27).—Purified syrups of neutral flavour are produced from raisins or other dried fruits by extracting with water, concentrating to about 55° Brix, cooling to allow crystallisation of tartaric acid compounds, filtering, diluting to 35—50° Brix, rendering slightly alkaline with sodium hydroxide, heating to 65—75°, filtering, acidifying to about pH 5.5, and concentrating to 55° Brix. Decolorising agents may be applied at a suitable stage, and sucrose or a material containing it incorporated to prevent crystallisation of dextrose. J. H. LANE.

Raisin syrup and its manufacture. H. W. DENNY, Assr. to SUN-MAID RAISIN GROWERS OF CALIFORNIA (U.S.P. 1,746,994, 11.2.30. Appl., 8.10.25).—Raisin syrup substantially free from raisin flavour and from tartaric acid and tannin is produced preferably by concentrating raisin juice to about 60° Brix to allow part of the tartaric acid compounds to crystallise, then filtering, diluting, liming to an alkalinity of 0.5% CaO, carbonating in one or two stages to a final alkalinity of 0.02—0.05% CaO, and eliminating remaining soluble calcium salts from the filtered juice by treatment with phosphoric acid followed by sodium or potassium hydroxide. Decolorising agents may be applied at any suitable stage. J. H. LANE.

Manufacture of dextrose. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,750,938, 18.3.30. Appl., 6.8.24. Renewed 20.6.29).—To produce pure dextrose hydrate from the impure hydrate, such as the third crop referred to in U.S.P. 1,471,347 (B., 1924, 27), the latter is partly remelted by heating with or without addition of water or light dextrose liquor, so that some of the crystals remain undissolved, and the magma is then crystallised in motion, without seeding, and at a temperature favourable to the crystallisation of the hydrate, preferably about 40° or below. J. H. LANE.

Production of dry lævulose or carbohydrate mixtures. W. C. ARSEM, Assr. to INDUSTRIAL TECHNICS CORP. (U.S.P. 1,763,080, 10.6.30. Appl., 31.1.27).—Dry lævulose is produced by heating inulin in a closed vessel with the calculated amount of water for hydrolysis and with a suitable hydrolytic agent; e.g., 1000 lb. of inulin mixed with 11.55 U.S. galls. of water are heated at about 100° with sulphur dioxide or carbon dioxide, under a pressure of 10—100 lb./in.² for 1 hr. or until hydrolysis is complete. J. H. LANE.

Continuous conversion of starch. INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 310,924, 11.5.29. U.S., 3.5.28).—A suitable mixture of starch, water, and acid is gelatinised by heat and forced through long tubes ($\frac{3}{4}$ in. bore, 15 ft. long) which are surrounded by steam under pressure. Four bundles of tubes in all are used, giving a total travel of the material of 60 ft. The pressure employed is usually higher than in batch-working, e.g., 50—80 lb./in.² in the production of liquid glucose or 50—100 lb./in.² for solid grape sugar. In the tubes the material attains approximately the same pressure and temperature as the surrounding steam, and when discharged it liberates steam, part of which may be used for gelatinising fresh starch material. Suitable apparatus is described and figured. The densities of

the starch suspensions and the amounts of acid used may be substantially the same as in batch-working. The material is forced through the apparatus in 4–8 min. according to the temperature of the steam and the degree of conversion required. The tubes may be of heat-resisting glass or metal with enamel linings to prevent any catalytic influence of bare metal on the conversion.

J. H. LANE.

Increasing the output in extracting crystallised sugar from molasses and syrups. E. RICARD, ASST. to Soc. ANON. DES DISTILLERIES DES DEUX-SÈVRES (U.S.P. 1,776,819. 30.9.30. Appl., 23.8.26. Belg., 5.9.25).—See F.P. 621,075; B., 1928, 137.

Ripening of sugar cane (U.S.P. 1,746,190).—See XVI. 2:3 [β]-Butylene glycol by fermentation (B.P. 315,263).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Colorimetric method of determining the soft resins of hops. J. M. GUTHRIE and G. G. PHILIP (J. Inst. Brew., 1930, 36, 455–459).—Minced hops are extracted with industrial methylated spirit and the α - and β -resins are extracted from the spirit solution by light petroleum. The last-named is evaporated and a solution in industrial methylated spirit of the residual resins is coloured by a methyl-alcoholic solution of uranyl acetate and compared in a Hellige universal colorimeter with a wedge of a standard colour prepared from a known solution of α -resin. The colorimetric reading permits the calculation of the sum of the α -resin and of the α -resin colorimetrically equivalent to the β -resin expressed as a percentage on the hops. C. RANKEN.

Fermentation cabinet. C. H. BAILEY (Cereal Chem., 1930, 7, 341–345).—A cabinet is described suitable for carrying out dough fermentation under carefully controlled conditions of temperature and humidity.

W. J. BOYD.

Some fermentation characteristics of various strains of *Rhizobium meliloti* and *R. japonicum*. R. H. WALKER and P. E. BROWN (Soil Sci., 1930, 30, 219–229).—The fermentation of dextrose and galactose by numerous strains of *R. meliloti* and *R. japonicum* is examined by means of changes in the p_H value of the media. Whilst individual strains of the organisms produced consistent results, different strains of the same species caused considerable variations in the degree of acidity or alkalinity produced. In general, *R. japonicum* produced a more alkaline reaction than did *R. meliloti*. Fermentation tests with the two sugars are insufficient to distinguish between the two species of organisms.

A. G. POLLARD.

Influence of the p_H value of the medium on alcoholic fermentation. I. Influence exerted by the products of fermentation on the velocity of multiplication of the yeast cell. II. Alcoholic fermentation in liquids of high hydrogen-ion concentration. III. L. CASALE (Annali Chim. Appl., 1930, 20, 336–353, 353–357, 357–361).—I. Sugar solutions containing inorganic yeast nutrients and of initial p_H values ranging from 2.8 to 8 have been fermented at 22°, the amount of fresh yeast added per litre corresponding with 0.0296 g. of yeast dried at 100°. The

maximum velocity of fermentation occurred at p_H 3.3–4, and the maximum spirit yield at p_H 2.8 and 5, whilst the greatest fermenting power corresponded with the initial p_H 6.93 and the final value 4. The sugar was completely utilised in all the solutions except that with initial and final values 2.8 and 2.6, respectively; in this solution the production of yeast was equal to that obtained in the other solutions, so that at p_H 2.6 inhibition of the action of the zymase, but not of cell multiplication, occurs. The amount of lactic acid formed increased with the initial p_H of the fermenting liquid, but about the same amount of volatile acid was produced in all the solutions, and the formation of glycerol followed the same course as the fermenting power. The activity of multiplication of the yeast was measured by two methods: (1) making use of the fermentative activity, and (2) using the increase in weight of the yeast. Since the first method measures the multiplication during a short period of the fermentation, whilst the second gives the total activity, different results were obtained by the two methods. By method (1) the activity of multiplication was greatest at p_H 6.9, whereas the weight of dry yeast formed was highest when the p_H fell during fermentation from 8 to 4, or from 5 to 2.8, or from 4 to 3. The effect of the p_H value of a fermenting solution consists in modifying the relation between the weight of dry yeast and that of the weight of mineral substances absorbed by the yeast, and also the relation between the cations and anions of the mineral substances.

II. Determination of the constants of yeast cell multiplication during alcoholic fermentation in solutions containing inorganic yeast nutrients and of various p_H values shows that cell multiplication gradually falls off as the fermentation proceeds, except in alkaline solutions (initial p_H 8), with which the rate of multiplication increases during the early stages of the fermentation.

III. A p_H value below 2.5 arrests both yeast cell multiplication and the action of the enzymes determining the formation of alcohol, but does not prevent the action of the other enzymes present, such as those giving rise to lactic and formic acids and acetaldehyde. The acetaldehyde present in highly acid fermented liquids appears to be, not an intermediate product of fermentation derived from pyruvic acid by the action of carboxylase, but a product of the action of another enzyme, probably on lactic acid (cf. Schade, B., 1906, 1112).

T. H. POPE.

Malting and brewing trials with a 6-rowed winter barley. H. L. HIND (J. Inst. Brew., 1930, 36, 435–439).—A British 6-rowed winter barley (F. 112) has been grown which gives a notable yield, and can be malted to produce a brewing material as satisfactory as that obtained from foreign barleys of the same type. Except for its high moisture content the variety closely resembles Californian Bay brewing barley. It requires a longer resting period before malting than that necessary for a 2-rowed English barley, and on that account is not fit to malt so early in the season as is imported foreign barley. It has also the defect that it is liable in wet weather after ripening and before cutting to develop *Cladosporium* on the pales at the germinal ends of the grains.

C. RANKEN.

Prediction of extract [yielded by a barley as malt]. L. R. BISHOP (J. Inst. Brew., 1930, 36, 421—434).—For any one variety there is an inverse relation between the nitrogen contents of barley and the extract yield of the resulting malt, whilst the extract increases with increase of grain size. Soil and season have no direct influence. An "extract equation," which permits the prediction of the amount of extract a barley should yield as malt, can be constructed for each separate variety of barley, and for a pure line of Plumage-Archer barley $E = 110.1 - 11.2N + 0.18G$, where E is the extract in lb. of dry malt made from the screened barley, N the nitrogen content on dry unscreened barley, and G the weight in g. of 1000 dry corns of unscreened barley. For Spratt-Archer barley 0.5 lb. must be added to the extract value. Variations in malting conditions are the chief source of differences between prediction and analysis. When adjustments are made for any given maltster, 68.5% of the predictions are within 0.8 lb. of the actual results obtained, whilst 95.5% of the predictions are within 1.6 lb. C. RANKEN.

Acid formation during the fermentation of beer wort. W. WINDISCH, P. KOLBACH, and R. ILLIES (Woch. Brau., 1930, 47, 417—421, 427—431, 437—441).—Worts prepared from several malts, and worts prepared from the same malt but varied as regards protein degradation, acidity, and buffering, were fermented under comparable conditions. Active, titratable, and volatile acidity, esters, and total, formol, and ammoniacal nitrogen were determined on worts and beers. For the esters an improved form of the method of Windisch, Reimers, and Hirschbruch (B., 1915, 812) was employed. The results obtained from different malts and from worts prepared under varying conditions were too complicated to lead to definite conclusions, but strong assimilation of ammonia was frequently associated with higher acid production. Addition of hydrochloric, sulphuric, phosphoric, or lactic acid to wort within practical limits of p_H , is without effect on nitrogen assimilation, but reduces the production of acid, particularly of volatile acid. The p_H of the beers varies with, but to a smaller degree than, that of the worts. Added acetic acid in part disappears during fermentation and the volatile acidity may be less in the beer than in the wort; nitrogen assimilation is also decreased. Addition of citrate buffer without alteration of the reaction of the wort leads to increased acid production, but acetate is partly destroyed. The latter case and that of one malt were the only two in which notable ester formation occurred. F. E. DAY.

Stimulation of cane molasses fermentation by certain metallic salts. F. M. HILDEBRANDT and F. F. BOYCE (Ind. Eng. Chem., 1930, 22, 1011—1014).—Yeast treated with suitable concentrations of copper sulphate (1 in 2000), manganous sulphate (1 in 5000), or sodium cyanide (1 in 12,000) in a solution of cane molasses, and then used as seed in a second solution to which no salts have been added shows a small but definite increase in the yield of alcohol, as compared with untreated yeast. The effect is not continued on seeding a third solution of molasses from the second.

W. J. BOYD.

[Contamination of wines by] nicotine [insecticides used] in vine culture. L. FERRÉ (Ann. Falsif., 1930, 23, 337—344).—The findings of Viala and Marsais (cf. Rev. Viticulture, 1928, 233; 1929, 70) that wines are highly contaminated by nicotine from insecticidal washes have been critically examined. From a consideration of the amount of nicotine deposited on the grapes it is shown that the amounts of nicotine reported to be present in the wines (231 mg. and 330 mg./litre) are impossible with the concentration normally used for the wash. The nicotine is deposited on the grapes in a soluble form and is readily removed by rain even when an adhesive is used. In hot, dry seasons the nicotine is rapidly lost by volatilisation, for after 8 days only traces could be detected. In wines made from grapes which had received one or two sprayings with nicotine washes not the slightest trace of nicotine could be found. The method of determination consisted in (i) steam-distillation after concentrating the wine, (ii) precipitation as silicotungstate, (iii) steam-distillation of the precipitate in the presence of magnesia, (iv) titration with 0.02*N*-sulphuric acid. It is concluded that the results obtained by Viala and Marsais are untenable as the presence of nicotine in wine would be discovered by taste alone. H. J. DOWDEN.

Buffering power of wines; keeping and plastering. V. MORANI and L. MARIMPIETRI (Annali Chim. Appl., 1930, 20, 313—335).—The potentiometric method for detecting addition of mineral acids to wine (cf. B., 1930, 390), with measurement of the specific buffering powers during neutralisation, may be applied to wines which, after addition of acid, have deposited solid substances as the result of keeping or plastering. Where extraneous causes do not intervene, the two processes, keeping and plastering, always determine a lowering of the p_H of the wine, this being caused by diminution of the extractive substances, especially of the proteins and of salts of organic acids. The method suggested should be applicable also to vinegar. T. H. POPE.

Oxidimetric determination of ethyl alcohol. E. KOSTUK (Bull. Assoc. Chim. Sucr., 1930, 47, 231—237).—For rapid routine determinations of alcohol in fermented liquids or vinasses, a quantity of the sample containing not more than 0.06—0.07 c.c. of absolute alcohol is distilled so that all the alcohol is obtained in 3—10 c.c. of distillate. Thus 100 c.c. of vinasses are distilled (apparatus described), and 10 c.c. of distillate collected, or 10 c.c. of 10- or 20-fold diluted wines or fermented worts are distilled and 3—4 c.c. collected in a test tube. Ten c.c. of a solution containing 33.832 g. of potassium dichromate, 250 c.c. of concentrated sulphuric acid, and 130 c.c. of 85% syrupy phosphoric acid (d 1.7) per litre are measured into another test tube and then heated nearly to boiling and poured into the tube containing the distillate previously warmed to 40—50°, the latter tube being rotated during the pouring to rinse down the walls. After 5 min. the mixture is transferred to a large vessel, into which both test tubes are thoroughly rinsed with water, and the liquid is finally diluted to about 300 c.c., treated with 6—8 drops of a solution of diphenylamine in concentrated sulphuric acid (1 g. per 100 c.c.), and titrated with a solution

containing 70 g. of ferrous ammonium sulphate and 20 c.c. of sulphuric acid per litre until the original brown colour, after passing through bluish-violet, changes sharply to green. The titre of the ferrous solution is ascertained by a blank titration of 10 c.c. of the dichromate solution under similar conditions, whence the volume of dichromate solution reduced by the distillate can be calculated (1 c.c. \equiv 0.01 c.c. of absolute alcohol).

J. H. LANE.

Boiler feed-water for distilleries. MOUSCADET.—See XXIII.

See also A., Oct., 1256, **Velocity of inversion of sucrose** (TAKETOMI). 1303, **Identification and determination of methyl alcohol in presence of ethyl alcohol** (IONESCO-MATIU and POPESCO). 1316, **Enzyme purification; pancreatic amylase** (SHERMAN and others). 1317, **Crystalline pepsin** (NORTHROP). 1318, **Fourth and fifth fermentation processes of sugar** (NEUBERG and KOBEL). **Co-enzyme** (VON EULER and MYRBÄCK). 1322, **Enzymic change in ripening barley** (LÜERS and others).

PATENTS.

Manufacture of yeast. A. W. HIXSON and N. E. A. HUGOSON, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,745,693, 4.2.30. Appl., 14.1.21).—Bakers' yeast is propagated with aeration in a solution containing molasses with an added oxidising agent such as calcium hypochlorite. An approximately constant p_H is ensured by the addition of phosphates or salts of a polybasic acid, whilst ammonium sulphate and urea are added as nutrients.

C. RANKEN.

Preparation of yeast. G. O. W. HEIJKENSJÖLD, Assr. to AKTIEB. BÄSTA (U.S.P. 1,757,568, 6.5.30. Appl., 7.3.29. Swed., 14.3.28).—Fermentation is started by seeding yeast into a sugar solution in a fermentation tank in greater amount than that required for the quantity of sulphite waste liquor which will fill the fermentation vessel. Sulphite waste liquor is added to the tank, and, periodically, solution containing yeast is withdrawn, from which the yeast is separated and returned to the tank, while the separated solution is run to waste. The withdrawn wort is replaced by fresh sulphite waste liquor, and the concentration of the sugar and nutrients regulated as desired.

C. RANKEN.

Manufacture of vitamin products. J. TAKAMINE, J. TAKAMINE, JUN., and N. FUJITA, Assrs. to TAKAMINE FERTILIZER CO. (U.S.P. 1,756,574, 29.4.30. Appl., 18.4.24).—Moistened protein-containing material, such as cereals, clover, starch residues, etc., is sterilised by steam and fermented by a fungus such as *Aspergillus oryzae*. The developed vitamin products and those already in the culture are adsorbed from an aqueous extract of the fermented mash by inert earths, from which they are subsequently dissolved by barium hydroxide. The barium is precipitated by sulphuric acid and protein matters are removed by lead acetate, the excess of which is removed by hydrogen sulphide. After purification by alcohol and acetone, the supernatant liquid is precipitated by ammoniacal silver nitrate, the metallic element of the resulting vitamin compound removed by hydrogen sulphide, and the vitamin solution concentrated under vacuum.

C. RANKEN.

[Microbiological] preparation of 2:3[β]-butylene glycol from carbohydrates. T. H. VERHAVE, SEN. (B.P. 315,263, 25.6.29. Holl., 10.7.28).—In the fermentation of carbohydrates (potato mash, cane molasses, etc.) with *Clostridium polymyxa*, *Aerobacter aerogenes*, or other bacteria producing β -butylene glycol, a sterilised mash is used containing the carbohydrate, a nitrogen compound (malt, ammonium sulphate), a phosphate (superphosphate, phosphorite), and a carbonate (limestone).

C. HOLLINS.

Centrifugal separator (U.S.P. 1,760,071).—See I.

XIX.—FOODS.

Baking characteristics of various types of wheat as reflected by different baking procedures. G. MOEN (Cereal Chem., 1930, 7, 351–357).—Using six flours of varying characteristics, the Standard Baking Procedure of the American Association of Cereal Chemists was compared with two other methods, one of which differed in having longer time and lower temperature of fermentation, with a modified formula, and the other in having shorter time and lower temperature of fermentation, with the standard formula. The standard procedure was the most efficient, giving the required information in the shortest time. In order to differentiate between flours of different strength, time and temperature of fermentation should be so adjusted that diastatic capacity is severely taxed. Addition of bromate to the standard formula emphasises desirable or undesirable characteristics in wheat which the ordinary standard procedure fails to reveal.

W. J. BOYD.

Effect of temperature on the dough and its influence on the standard baking test. V. E. FISHER (Cereal Chem., 1930, 7, 367).—In using the Standard Baking Test, the temperatures most suitable for creaming and doughing are respectively 23° and 21°.

W. J. BOYD.

Determination of carotene in flour with the Pulfrich photometer. ANON. (Mühle, 1930, 67, 209–210; Chem. Zentr., 1930, i, 2816).—The flour is compared photometrically with barium sulphate.

A. A. ELDRIDGE.

Analysis of egg yolk. M. MONHAUPT (Chem.-Ztg., 1930, 54, 697–698).—Moisture is determined by mixing the liquid yolk with a known weight of dry sand and heating at 100–105° to constant weight. Direct carbonisation of the yolk leads to loss of chlorine owing to the action of the metaphosphoric acid formed during combustion of the yolk on the sodium chloride added as a preservative; this is prevented by the addition of 6–10% of calcium hydroxide prior to heating. The ash is extracted with water, the filtered solution neutralised with sulphuric acid, and the chloride determined by titration with silver nitrate.

A. R. POWELL.

Keeping qualities of butter. VI. **Production of metallic flavour in butter and milk.** VII. **Microbic flora of off-flavoured butter.** G. L. A. RUEHLE (Mich. Agric. Exp. Sta. Tech. Bull., 1930, No. 102, 2–12, 13–46).—Metallic flavour may be imparted by iron or copper lactate and by decomposi-

tion of protein, *e.g.*, by bacterial or enzyme action. Numerous organisms were isolated.

CHEMICAL ABSTRACTS.

Decrease in interior quality of hens' eggs during storage as indicated by the yolk. P. F. SHARP and C. K. POWELL (Ind. Eng. Chem., 1930, 22, 908—910).—During storage the yolk membrane of an egg is progressively weakened owing to the stretching produced by the transference of water from the white to the yolk, and furthermore the viscosity is reduced, with the result that when the yolk is placed on a flat plate it assumes a flatter shape and, finally, a condition is reached when the egg cannot be opened without breaking the yolk. The "flatness" of the yolk as measured by the ratio of the height to diameter has been used as an index of the interior quality of eggs. The influence of temperature of storage was most pronounced. The average value for freshly laid eggs was 0.41, but after 3 days at 37° the value fell to 0.30, whereas it had only reached 0.34 after 100 days at 2°. The limit at which the egg could not be opened without rupturing the yolk was about 0.25. The total solids content of the yolk is influenced by the ingress of water and the effect of the storage temperature on this value resembles closely that on the "yolk index."

H. J. DOWDEN.

Copper content of certain foods. F. GRENDL (Pharm. Weekblad, 1930, 67, 913—921).—The average copper content of cows' milk, determined by the sodium diethyldithiocarbamate method, is 0.11 mg. per litre, and of goats' milk 0.15 mg. per litre. Pasteurised milk contains on an average 0.22 mg. per litre, which is practically the same as that found in human milk. The copper content of a variety of other common food-stuffs varies from 0.07 to 0.22 mg./100 g., calves' liver being exceptional in that it contains about 0.5 mg./100 g.

H. F. GILLBE.

Determination of husk in analysis of cocoa. M. WAGENAAR (Chem. Weekblad, 1930, 27, 533—539).—The various methods prepared for identifying and determining husk in cocoa are reviewed; the optical method based on examination in polarised light, worked out by the author (cf. B. 1929, 955), is declared to be the only satisfactory quantitative test.

S. I. LEVY.

Flavour of orange honey. E. K. NELSON (Ind. Eng. Chem., 1930, 22, 448).—The pleasant odour of orange honey suggested the presence of methylanthranilate and tests for this substance gave positive results.

J. H. LANE.

Vitamins in canned foods. IX. Tomato products. E. F. KOHMAN, W. H. EDDY, and C. ZALL (Ind. Eng. Chem., 1930, 22, 1015—1017; cf. B., 1930, 34).—Feeding experiments on rats and guinea pigs were used for testing the vitamin content of tomatoes. The vitamin-A content of green tomatoes almost doubles on ripening. Canning of either green or ripe tomatoes has little effect on their vitamin-A content, and there is little loss in concentrating the juice *in vacuo* or in an open pan. The vitamin-A is largely removed with the pulp on filtering. There is little difference in the vitamin-B content of green and ripe tomatoes, raw or canned. Filtration of the juice through celite results in much loss of vitamin-B, but vacuum concentration causes

very little loss. 7 G. of green tomatoes, raw or canned, are about the equivalent in vitamin-C of 5 g. of ripened, green-picked tomatoes, raw or canned. Considerable loss of vitamin-C results from the various commercial processes of canning, except in the case of tomatoes canned whole. When the process involves the mixing of tiny air bubbles with the product, as in cycloning, this loss is greater than in processes in which intimate contact with air is avoided. The vitamin-B content is affected to a smaller degree in the same way. When the cycloning is carried out on the hot juice the atmosphere of steam appears to exert a protective action.

W. J.-BOYD.

Drapes of *Schinus molle* as adulterant of *Piper nigrum*. A. CREMONINI (Annali Chim. Appl., 1930, 20, 309—312).—This adulteration of ordinary pepper by false (red or Peruvian) pepper may be detected by immersing a small portion, grain by grain, in Fehling solution kept boiling for a time. Reduction of the solution is effected by the sugar present in *Schinus molle* (cf. B., 1928, 873). If the grains on which the cuprous oxide has deposited are removed from the Fehling solution, washed in water, and cut through with a sharp knife, the internal, woody involucre of irregular stellate form, surrounded by vacuoles, which in the just ripe fruit are full of essential oil, is visible to the naked eye. If the drupes are freshly and not too highly torrefied, the small amount of essential oil remaining in the vacuole may be detected by heating with nitric acid (*d* 1.40), copious evolution of red vapours occurring; if the drupes are then removed, crushed, and heated with water, vapour with a strong odour of terpenes is emitted. Ground material may be tested by the above reactions and may be examined microscopically for the very hard, woody fragments furnished by the interior of the drupe. The extent of the adulteration may be judged approximately from the reduction of Fehling solution, 1 g. of the lightly torrefied, air-dried drupes (26—28) containing about 0.1 g. of reducing sugar (dextrose).

T. H. POPE.

Fermentation cabinet. BAILEY.—See XVIII.

See also A., Oct., 1295, **Tea tannin from green tea** (TSUJIMURA). 1321, **Vitamin-A of butter** (MORTON and HEILBRON).

PATENTS.

Preservation of eggs and the yolks and whites thereof. J. FOUSEK (U.S.P. 1,756,188—9, 29.4.30. Appl., 11.5.27).—Equal weights of sugar and (A) egg yolks or (B) egg whites are mixed at about 60° until the mass is smooth and of a honey-like consistency.

E. B. HUGHES.

Improvement of tea. J. P. CROFT (B.P. 334,224, 1.3.29).—The caffeine content of tea is increased by mixing with it a hot, concentrated, aqueous solution of caffeine, kneading till dry, and blending the product with more tea in suitable proportion. The preparation of a malted tea is described.

E. B. HUGHES.

Preservation of grain and the like. R. A. LEGENDRE (U.S.P. 1,777,044, 30.9.30. Appl., 21.1.29. Fr., 2.4.27).—See B.P. 310,980; B., 1929, 535.

Manufacture of aerated waters, sparkling drinks, and the like. C. HOLLINS and E. CHAPMAN, Assrs. to

BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,777,365—7, 7.10.30. Appl., [A] 5.8.27, [B, C] 9.7.28. U.K., [A—C] 19.8.26).—See B.P. 280,302; B., 1928, 67.

Purification of phosphatides [from soya beans etc.]. H. BOLLMANN (U.S.P. 1,776,720, 23.9.30. Appl., 4.9.26. Ger., 6.10.25).—See B.P. 259,166; B., 1926, 993.

Reconditioning of musty grain and the like [by ultra-violet rays]. E. VON HORVÁTH (B.P. 335,445, 30.11.29).

[Apparatus for] preservation of fruits, vegetables, and other foods. J. MAYOR (B.P. 335,013, 7.8.29).

Vitamin products (U.S.P. 1,756,574).—See XVIII. **Emulsions (U.S.P. 1,758,651).**—See XX. **Liquid treatment (U.S.P. 1,756,275).**—See XXIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Evaluation of tincture of iodine. F. HALLERMANN (Apoth.-Ztg., 1930, 45, 252—253; Chem. Zentr., 1930, i, 2459).—For the determination of total iodine 2 g. of the tincture are treated with 10 c.c. of chloroform and with a solution of ferric chloride, the solution being gently mixed at intervals for 1.5 hrs. Phosphoric acid (25%, 25 c.c.) is then added, and the solution titrated with 0.01N-thiosulphate. A. A. ELDRIDGE.

Solution of magnesium citrate. E. W. REES (Amer. J. Pharm., 1930, 102, 496—505).—Of 69 samples of solution of magnesium citrate, U.S.P. X., having ages varying from 1 to 3 years, 64% contained a deposit which consisted chiefly of the tribasic citrate, $Mg_3(C_6H_5O_7)_2 \cdot 14H_2O$. This compound is formed in the solution made according to U.S.P. X., and, if these directions are adhered to, the product remains clear for 3 months. Attempts to prepare a permanently clear solution were not successful. Samples containing the crystalline deposit can be restored by warming and agitating. Freedom from mould growth and the stability of the solution are dependent on the presence of carbon dioxide. The U.S.P. X. samples contain free citric acid and not magnesium acid citrate, as is evidenced by the fact that the sugar present is inverted under the same conditions as a plain sugar solution containing the same amount of free acid.

E. H. SHARPLES.

Citric acid assay of solution of magnesium citrate, U.S.P. H. V. ARNY and H. H. SCHAEFER (J. Amer. Pharm. Assoc., 1930, 19, 941—945).—Results of the U.S.P. X. tests for free and total acid in commercial and experimental samples of this solution are given. The tests are satisfactory, but the limits imposed for the former value are unnecessarily severe. Statements that citrate is rapidly lost from the solution by decomposition in presence of air are not confirmed. The U.S.P. IX. formula is preferred to that at present official for this preparation. H. E. F. NOTTON.

Methods of assay of chloral hydrate. H. A. WATSON (Amer. J. Pharm., 1930, 102, 506—525).—Seven classes of methods of assay of the compound have been compared. Determination of the formic acid produced on treatment with magnesium oxide is unreliable owing to the instability of magnesium formate, which substance is a probable cause of error in Kippenberger's method

in which the carbon monoxide produced on decomposition of the chloroform formed by treatment with magnesium oxide is measured. Conversion of the chloral hydrate into chloride either with zinc dust (Self's method) or with aqueous or alcoholic alkali gives good results, using the author's modifications. Ruff's iodometric method is satisfactory when the interfering secondary reactions are reduced to a minimum by diluting the alkali to ten times the volume and then adding it drop by drop with constant shaking. A modification of the method of Archangelsky, in which the formic acid produced by the action of alkali is determined by the reduction of mercuric to mercurous chloride, gives uniform results about 0.5—1% low. E. H. SHARPLES.

Quinizarinsulphonic acid (rufianic acid) as a precipitant for alkaloids. L. ROSENTHALER (Pharm. Zentr., 1930, 71, 561—562).—Whilst precipitates are obtained with most alkaloids, often at very great dilutions, they are very similar in appearance, and the reagent is not likely to be important for microchemical differentiation. S. I. LEVY.

Sterilisation of hexamethylenetetramine solutions. G. H. NIJHOFF and H. D. VAN OORT (Pharm. Weekblad, 1930, 67, 884—888).—Sterilisation by boiling causes decomposition with formation of ammonia and formaldehyde. It is recommended that 2% of sodium bicarbonate should be added to the solution and sterilisation effected by Tyndallisation. The extent of the decomposition under various conditions has been determined. S. I. LEVY.

Removal of the bitter principle of lupin seed by Thom's process. M. GERLACH (Landw. Jahrb., 1930, 72, 247—253).—The bitter principle of yellow lupin seeds cannot be removed from whole seed by 50% methyl or ethyl alcohol, nor by denatured spirit. Spirit (50% denatured) containing a small amount of hydrochloric acid removed 75% of the alkaloid from crushed seeds, but with considerable losses of total dry matter, fat, and protein. Satisfactory extraction without attendant losses was obtained by heating the whole seeds in water containing a little hydrochloric acid and subsequently treating with 50% denatured spirit. A. G. POLLARD.

Preparation of extractum hydrastis fluidum and methods of analysis. Z. CSIPKE (Ber. Ungar., pharm. Ges., 1929, 5, 73—85; Chem. Zentr., 1929, i, 1583).—A report on the known methods of preparation and on the official determination of the alkaloid content in the drug and the extract. L. S. THEOBALD.

Determination of theophylline and ethylenediamine-theophylline. P. W. SCHMITT (J. Amer. Pharm. Assoc., 1930, 19, 821—825).—A method for the analysis of ethylenediamine-theophylline ("Euphyllin") consists in the determination of theophylline by precipitation with a standard ammonio-silver chloride reagent followed by determination of the excess of silver in the filtrate and a determination of the ethylenediamine by calculation from the total nitrogen content.

E. H. SHARPLES.

Comparative methods of assay of Chinese ephedras. Y. D. Hsu (J. Amer. Pharm. Assoc., 1930, 19, 817—820).—From an examination of various methods, the following is recommended as being con-

venient and satisfactory for the determination of total alkaloids in ephedras. A mixture of the dried, powdered drug (20 g.), distilled water (40 c.c.), and freshly slaked lime (5 g.) is macerated for 10 min., 100 c.c. of 2% ammonium chloride solution in 70% alcohol are added, and the mixture is shaken. Five further quantities of 100 c.c. are periodically added with shaking and the mixture is allowed to macerate for 12 hrs. After filtration, the alcohol is evaporated and the filtered concentrate after the addition of 10 c.c. of ammonia solution is extracted three times for 1 hr. each with chloroform, 50, 40, and 40 c.c., respectively. The combined extracts are evaporated *in vacuo*, the residue being dissolved in neutral alcohol and titrated with 0.1*N*-hydrochloric acid. A sample consisting of a mixture of *E. equisitina* and *E. sinica* yielded 1.999% of total alkaloid. Further extraction with 200 c.c. of alcoholic ammonium chloride gave 0.056% of alkaloid.

E. H. SHARPLES.

Jasmine. ANON. (Parf. mod., 1929, 22, 691—705; Chem. Zentr., 1930, i, 2177).—A description of manufacture and use.

L. S. THEOBALD.

Zdravet in modern perfumes. ANON. (Parfums de France, 1929, 7, 315—316; Chem. Zentr., 1930, i, 2177).

Balsamic mass from acetic acid tar. PALLAS.—See II. Tobacco-seed oil. KANDILIS and KARNIS.—See XII. Adulterant of *Piper nigrum*. CREMONINI.—See XIX.

See also A., Oct., 1286, Homologues and analogues of phenylpropanolamine (HARTUNG and others). 1294, Preparation of thymol from piperitone (KIMURA). Sagittol (YANOVSKY). 1295, α -Tetrahydrofurfuryl chloride and ethers (KIRNER). 1300, Catalytic reduction of nicotine and metan nicotine (HARLAN). 1303, Identification and determination of methyl alcohol in presence of ethyl alcohol (IONESCO-MATTU and POPESCO). 1304, Micro-detection of alkaloids (LANDER). 1315, Pharmacology of gold compounds (VON ISSEKUTZ and others). Local anæsthetics derived from dialkylaminopropanediols (RIDER). 1320, Vagotonin (PÉNAU and SANTENOISE). Crystalline insulin (GEILING and DE LAWDER; JENSEN and DE LAWDER). Oestrin (MARRIAN). Crystalline menoformone (DINGEMANSE and others). Relaxative hormone of the corpus luteum (FEVOLD and others). 1323, Leaves of *Solanum pseudocapsicum* (BREYER-BRANDWIJK).

PATENTS.

Manufacture of [pyro]catechol [4-(3:4-dihydroxyphenyl)thiazoles. T. B. JOHNSON, ASSR. to WINTHROP CHEM. CO., INC. (U.S.P. 1,743,083, 7.1.30. Appl., 3.2.28).—Chloroacetylpyrocatechol [3:4-dihydroxyphenyl chloromethyl ketone] is condensed with thioamides, thiocarbamides, or thiocarbamates to give thiazoles which have therapeutic value. 4-*mp*-Dihydroxyphenylthiazoles having various substituents in the 2-position already described (cf. Johnson and Gatewood, A., 1929, 943) are claimed.

C. HOLLINS.

Manufacture of basic bismuth salts or organic arsinic acids. I. G. FARBERIND. A.-G. (B.P. 334,449, 14.11.29. Ger., 22.11.28).—Non-mercurated alkali arylarsinates react with solutions of bismuth salts (excluding

bismuthyl potassium sodium tartrate) to give alkali-insoluble basic bismuth arylarsinates. Examples are basic bismuth salts of 3-acetamido-4-hydroxybenzene-arsinic acid, methylbenzimidazole-arsinic acid, and 3-amino-4-hydroxybenzene-arsinic acid. C. HOLLINS.

Manufacture of pharmaceutical compounds. W. T. WINCKLER, ASSR. to VON WINKLER LABS., INC. (U.S.P. 1,751,964, 25.3.30. Appl., 7.11.28).—Sulphoricinoleic acid, *i.e.*, sulphonated castor oil, is converted into a silver salt by the addition of silver ammonium formate. The product is specific for gonorrhœa.

C. HOLLINS.

Ozonising tobacco. S. ROSENHOCH (U.S.P. 1,757,477, 6.5.30. Appl., 9.7.28. Ger., 11.7.27).—The tobacco is brushed clear of foreign matter, slightly moistened with sterilised water, and, after 5—10 hrs., the leaves are separated, foreign plant matter is removed, and the leaves are again moistened. After 10—16 hrs. they are spread thinly on an electrically charged conveying device and treated with a countercurrent of ozone in an amount varying with the nicotine content of the tobacco. The electrical treatment breaks up the cells without destruction of aromatic resins or oils, so that the ozone can enter the leaf and effect its refinement. Suitable apparatus is described.

E. H. SHARPLES.

Chemical emulsion. J. R. CONOVER, ASSR. to LAB. OF RES. CHEMOTHERAPY (U.S.P. 1,758,651, 13.5.30. Appl., 29.5.24).—Emulsions are made from substances which are identical with those found in animal tissue or tissue-producing substances, such as lanolin, other animal fats and waxes, casein, egg and serum albumins, etc. The emulsions closely resemble living tissue and provide efficient carriers for germicidal and chemotherapeutic agents. *E.g.*, the colloidal potassium silver salt of polypeptides derived from casein, which has a germicidal activity equal to that of a 1.33% phenol solution and which retains its activity indefinitely, is prepared by mixing an aqueous silver nitrate solution with a solution of casein in dilute sodium or potassium hydroxide under suitable conditions, and adding the mixture to an emulsion made by stirring lanolin with a solution of casein in alkali or alkaline-earth hydroxide or carbonate solution.

E. H. SHARPLES.

After-treatment of sterilised catgut for surgical use. R. GRAF & Co., A.-G. (B.P. 333,847, 10.12.29. Ger., 14.12.28).—Catgut, sterilised in the usual manner, particularly with iodine, is treated with solutions which contain protein, such as bacteriological meat-broth solution, for 12—24 hrs., and then immediately with 96% alcohol for a similar period. The catgut is thereby freed from injurious chemicals, particularly free iodine, and is also rendered non-irritant.

E. H. SHARPLES.

Production of medicaments containing bismuth. A. LIEBRECHT, ASSR. to CHEM.-PHARM. A.-G. BAD HOMBURG (U.S.P. 1,777,173, 30.9.30. Appl., 24.7.25. Ger., 29.7.24).—See B.P. 237,912; B., 1927, 60.

Manufacture of inactive menthol. K. SCHÖLLKOPF and A. SERINI, ASSIS. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,776,667—1,776,671, 23.9.30. Appl., 20.9.28. Ger., [A—D] 15.2.27, [E] 15.12.26).—See B.P. 285,403; B., 1928, 873.

Phenanthridone etc. (B.P. 333,173).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Jelly strength of photographic gelatin. J. P. LAWRIE (J.S.C.I., 1930, 49, 403—404 T).—In testing jelly strengths of gelatins by Tracey's apparatus (B., 1928, 310), considerably divergent results were obtained. Strictly controlled experiments were carried out in an attempt to ascertain the cause of the irregularities, but it was found to be impossible to repeat or duplicate results. The irregularities are attributed to the important influence of time and temperature during the making of the gel, and it is suggested that it is essential to use a proper room, thermostatically controlled. Apparently no consideration has been given to the possibility of the irregularities being produced by mechanical defect or faulty design of the apparatus used.

Antifogging and antisensitising compounds. S. E. SHEPPARD (Phot. J., 1930, 70, 439).—The effect of the presence of such substances on the *E.M.F.* of silver-ion concentration cells, using concentrations of the order of those afforded by saturated solutions of the silver halides, has been investigated at various p_H values. A removal of silver ions, presumably by complex formation, was shown by the antifogging agents thioacetanilide and nitrobenziminazole, the desensitising dyes phenosafranine and chrysoidine, and, in an extension of the work to sensitisers, by orthochrome-T, kryptocyanine, erythrosin, and the general sensitiser allylthiourea. This supports the suggested common initial behaviour of such compounds in forming complexes with the silver halide. A tentative explanation of the inability of chrysoidine bases to desensitise is given.

J. W. GLASSETT.

Desensitising properties of Basic Scarlet N. (Miss) F. M. HAMER (Phot. J., 1930, 70, 436—437).—Basic Scarlet N may be separated by extraction with ether into two constituents; the soluble and insoluble components react similarly to chrysoidine and safranine, respectively. The fact that the desensitising properties of a 1:5000 bath are unaffected by sodium carbonate and sulphite present in normal developer concentrations is not in accordance with the presence of chrysoidine, but the striking increase in loss of image, or inhibition of development, obtained by increasing the bath from 1:5000 to 1:500 is strong confirmation of its presence. Comparative tests indicate that Basic Scarlet N is not superior to safranine as a practical desensitiser.

J. W. GLASSETT.

See also A., Oct., 1260, **Effect of light on dyes** (KRAIS). 1266, **Emulsions on glass for recording distances** (COOKSEY and COOKSEY).

PATENTS.

Manufacture of light-sensitive layers. KALLE & Co. A.-G. (B.P. 333,820, 2.11.29. Ger., 3.11.28).—Aromatic azides (e.g., azidosalicylic acid) which are decomposed by light action to form a dye are dissolved in water or an organic solvent, or incorporated in gelatin or collodion solutions, and applied to a paper or film support and dried. After exposure the unchanged azide is removed by application of a solvent. The colour of the image may be modified by the inclusion of acids, bases, or metallic salts in the sensitive layer.

J. W. GLASSETT.

Light-sensitive plates or the like. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. to ALLGEM. ELEKTRICITÄTS-GES. (B.P. 315,355, 27.6.29. Ger., 12.7.28).—The support for the light-sensitive layer is prepared from artificial resins produced from the condensation products of polyhydric alcohols and polybasic acids or their anhydrides, e.g., glycerol-phthalic anhydride resins. The transparency, flexibility, and colour of the support can be controlled during manufacture by the addition of fillers, softening agents, and dyes. J. W. GLASSETT.

Photographic reducers. I. G. FARBENIND. A.-G. (B.P. 334,144, 29.11.29. Ger., 29.11.28).—The retardation of the reducing action in the higher densities, caused by either the localised concentration of the reaction products or the more rapid change in p_H at those parts, is minimised, in the first case, by the incorporation of a certain amount of the reaction products in the reducer, and, in the second, by the addition of a buffer salt. The following reducers are claimed: (A) 10% sodium thiosulphate solution 100 c.c., ferric oxalate 4 g., ferrous oxalate 4 g.; (B) potassium dichromate 2 g., potassium bisulphate 0.6 g., potassium sulphate 9 g., water 100 c.c. J. W. GLASSETT.

Chemically transforming photographic silver images. F. LIERG (B.P. 333,361, 8.7.29).—The process depends on the use of such compounds as thiocarbamide, thiosinamine, etc. which readily form water-soluble complexes with the silver halides, but which form insoluble or only slightly soluble complexes with other silver salts. The silver image is converted first into a silver halide, and is then precipitated as the new salt by treatment in a bath containing thiosinamine and the new salt-forming agent such as potassium ferrocyanide, tartaric acid, sodium nitrite, sodium phosphotungstate. The process may be adapted to the formation of azo-dye images and to the preparation of dye-repellant images suitable for dye-printing processes.

J. W. GLASSETT.

Imbibition processes for producing pictures in colour. UVACHROM A.-G. F. FARBENPHOT. (B.P. 383,392, 8.8.29. Ger., 16.2.29).—Improved reproduction is obtained by (a) using only the more highly-dispersed fractions of the dyes obtained by known methods by ultrafiltration, fractional precipitation, etc.; (b) treatment of the relief with aqueous glycollic acid; (c) exclusion of the yellow rays from the printing light used for the preparation of the relief; (d) addition of casein to the gelatin layer for the reception of the dye; and (e) regeneration of the relief during printing by treatment with an oxidising agent which destroys the strongly absorbed dye.

J. W. GLASSETT.

Manufacture of monochromatic photographic pictures. C. ROEHRICH (B.P. 308,361, 21.3.29. Ger., 22.3.28).—A dichromated gelatin plate is exposed behind a negative of the subject and treated with a suitable red dye which is absorbed most strongly in the unhardened parts. This dyed image is used in the subsequent copying process and exerts a non-uniform filtering action which varies in accordance with the light and shade of the original picture. Control of contrast may be obtained by suitably dyeing the unhardened and hardened parts. [Stat. ref.] J. W. GLASSETT.

Colour photography. W. V. D. KELLEY (B.P.

333,931 and 333,933, 23.5.29).—(A) The blue- and green-sensitive layer is separated from the red-sensitive emulsion by a thin layer of gelatin dyed with congo-red, which may be readily bleached subsequent to exposure by treatment with a dilute solution of sodium hyposulphite. (B) If the different colour-sensitive emulsions are coated on separate supports and placed together face to face for the exposure, the red-sensitive layer is bathed in a solution of congo-red, which does not penetrate, but forms an effective filter in the surface layers. J. W. GLASSETT.

Colour photography. SPICERS, LTD., J. W. GOLD-SMITH, T. T. BAKER, and C. BONAMICO (B.P. 333,865, 1.6.29).—In order to apply a multicolour screen to a film of cellulose acetate, nitrocellulose, or other cellulose esters, the surface of the film is treated in an alkaline bath composed of a 0.5–5% solution of caustic potash in methylated spirit, which renders the film capable of absorbing dye solutions very readily and facilitates their removal or bleaching. J. W. GLASSETT.

Production of films in natural colours by a single exposure. F. LIERG (B.P. 333,697, 2.7.29).—A film support is coated on one side with a blue-sensitive emulsion and on the other with superimposed green- and red-sensitive emulsions with the latter uppermost. By a suitable optical device both sides of the film are exposed simultaneously, and, after development and fixation, the blue screen image is mordanted, dyed yellow, and protected by a coat of lacquer. The green and red screen images are then mordanted, dyed with a bluish-red dye (rhodamine), treated in a hardening bath, and dried. The film is finally immersed in a bath of a bluish-green dye, which combines with the bluish-red dye to give a pure blue; owing to the hardness of the gelatin layer it does not penetrate to the red image underneath. J. W. GLASSETT.

Multicolour photographs. H. and C. HERZOG (J. HERZOG & Co.) (B.P. 333,668, 4.6.29).—The positive material consists of silver halide emulsions dyed with non-diffusing, highly transparent, colloidal dyes coated upon a transparent support. Printing is made from the separate colour-record negatives through the support side of the appropriately coloured positive material, and, after development in a tanning developer (sulphite-pyrocatechol), the unhardened parts are removed in hot water. A series of these monochrome images are then transferred in register to a common support. The colour may be controlled by treating the finished monochrome images with solvents for or reducing agents of the dye. J. W. GLASSETT.

[Lens unit for] production of photographic images. H. D. BEACH (B.P. 335,696, 27.8.29. U.S., 5.3.29).

Method of enabling pictures or the like to be exhibited in substantially natural colours. A. BERNARDI, and RAYCOL, LTD. (B.P. 335,310, 18.7.29).

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Charge for blasting caps. W. DE C. CRATER, ASSR. to HERCULES POWDER Co. (U.S.P. 1,759,565, 20.5.30. Appl., 13.8.29).—Nitrolactose is employed as the main charge for blasting caps, in conjunction with an initiating

charge of fulminate and chlorate, lead azide, diazodinitrophenol, etc. If desired, the nitrolactose may be mixed with the initiating charge. W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Vapour pressures of fumigants. IV. $\alpha\alpha\beta\beta$ -Tetra-, penta-, and hexa-chloroethanes. O. A. NELSON (Ind. Eng. Chem., 1930, 22, 971–972; cf. Young and Nelson, B., 1929, 456).—The vapour pressures of tetra- and penta-chloroethanes were determined by the static method at temperatures ranging from 25° to 146° and 160°, i.e., the b.p. of these compounds. Hexachloroethane being a solid which sublimes rapidly at its m.p. (185°), its vapour pressure over the range 15–60° was determined by saturating air with the vapour and cooling to –78°. C. IRWIN.

Water softening: certain properties of some base-exchange materials. A. R. MARTIN (J.S.C.I., 1930, 49, 389–394 r).—There are two main types of base-exchange material, the synthetic products and certain minerals. The minerals may be subdivided into non-porous glauconites and porous minerals. The bulk densities, volumes of interstitial spaces, and exchange values at varying rates of flow of water have been determined. The regeneration of the base-exchange materials and their salt consumption have been studied. The facts can be interpreted by supposing that with a glauconite the exchange of bases is wholly confined to the outer surface of the material, where it is very rapid. In the case of the synthetic products there is, in addition to the action at the outer surface, an action in the interior of the particle, diffusion to and from the interior being a relatively slow process. The interior action may be either in the mass of the material or at capillary surfaces. The mechanical disintegration and solubility of the base-exchange materials have been investigated, and in particular the effect of the original silica content of the water on their solubility.

Purification of [boiler feed]-waters in sugar factories and distilleries. G. MOUSCADET (Bull. Assoc. Chim. Sucr., 1930, 47, 207–215).—One or two coats of an aqueous suspension of finely-divided graphite, preferably colloidal, applied to the interior of sugar-factory boilers, after they have been cleaned and dried at the end of a campaign, will reduce incrustation during the following campaign, the salts precipitated from the water tending to form a sludge instead of a hard deposit. "Magnetine," a mixture of a special graphite with finely-powdered metals such as aluminium and copper is recommended for preventing corrosion as well as incrustation of boilers. In the discussion on the paper the use of phosphates for the treatment of boiler feed-water was advocated by Zamaron. J. H. LANE.

Water treatment for raw water ice-making. L. M. CULTER (J. Amer. Water Works' Assoc., 1930, 22, 1215–1219).—During the freezing of raw water the carbonates of magnesium and calcium and oxides of iron and aluminium as well as silica and suspended matter gradually accumulate in the core of the block and can be readily removed. The more soluble carbonate of sodium which replaces those of magnesium and calcium when the raw water is previously softened by base exchange or by soda ash treatment remains trapped in the body of the

ice block. Removal of colour is of great importance, and with some waters may be effected by the use of lime. Simultaneous removal of bacteria takes place. When the water has a low p_H and free carbon dioxide the colour is removed by treatment with sodium aluminate and aluminium sulphate to bring the final alkalinity to 10 p.p.m. H. INGLESON.

Protozoological examination of water. (MME.) N. L. WIBAUT (Chem. Weekblad, 1930, 27, 526—529).—Tabulations of the numbers of bacteria (*B. coli*) and protozoa present in previously sterilised waters at successive intervals after seeding show that in most cases both increase at first, the latter much faster than the former; after the period of increase both decrease. The conclusion is drawn that the protozoa can entirely eliminate the bacteria in most cases. Comparative tests of the number of protozoa and bacteria present in a water may be used to determine the date of infection of the water with bacteria. S. I. LEVY.

Effect of iron in the accuracy of the determination of dissolved oxygen [in water] by the Winkler method O. M. URBAIN and J. N. MILLER (J. Amer. Water Works' Assoc., 1930, 22, 1261—1265).—In such determinations the error due to the presence of iron increases with the time which elapses between the addition of sulphuric acid and the titration of the iodine liberated and with the amount of iron present in the water. A graph of corrections for iron contents up to 100 p.p.m. and for times up to 24 hrs. is given. Waters containing more than 50 p.p.m. of iron give wholly unreliable results, whilst those containing less than 50 p.p.m. give inaccurate results unless the titrations are made immediately. H. INGLESON.

Hydrogen sulphide in industry: occurrence, effects, and treatment. W. P. YANT (Amer. J. Pub. Health, 1930, 20, 598—608).

Acute response of guinea-pigs to vapours of some new commercial organic compounds. II. Ethylbenzene. W. P. YANT, H. H. SCHRENK, C. P. WAITE, and F. A. PATTY (U.S. Pub. Health Rep., 1930, 45, 1241—1250).

PATENTS.

Sewage disposal plant. T. B. SIMS, ASSR. to L. J. WARDLAW (U.S.P. 1,757,262, 6.5.30. Appl., 23.4.28).—A central scum chamber or gas vent in a double-storey settlement tank is fitted with a gastight cover and is maintained under reduced pressure by means of a suitable pump. The increased rate of removal of dissolved and fermentation gases thus induced causes a separation of the lighter particles of sludge and forms a scum which after mechanical agitation is withdrawn intermittently for further treatment. The degassing of the sewage also facilitates the dissolution of oxygen at a later stage with consequently increased stability. C. JEPSON.

Treatment of [sewage] sludge. Sewage disposal system. T. B. SIMS (U.S.P. 1,757,263—4, 6.5.30. Appl., [A] 10.12.28, [B] 29.4.29).—(A) The digestion of sludge is facilitated by mixing, preferably with vigorous agitation, the heavier and more decomposed sludge from the bottom of the tank with the less decomposed scum. The use of an intermittent vacuum is suggested to carry out this purpose and also to skim floating matter from the surface of the sewage and introduce the same

into a centrally situated sludge-digestion chamber normally maintained under partial vacuum to remove the gases generated. (B) In a system which includes the treatment of settled sewage on a percolating filter intermittently operated by means of a dosing chamber, the latter is so arranged that the rise and fall of the water therein may be utilised to supply compressed air for the aeration of the sewage prior to its entrance into the closing chamber, and also to create a partial vacuum over a portion of the sedimentation tank (cf. U.S.P. 1,757,262; preceding), by means of which hydrogen sulphide is removed from solution and the liquor rendered more amenable to stabilisation by aeration at a later stage. C. JEPSON.

Recovery of grease from garbage. W. D. CLEARY, ASSR. to DE LAVAL SEPARATOR Co. (U.S.P. 1,764,390, 17.6.30. Appl., 19.11.26).—The mixture of water, grease, and insoluble matter expressed from digested garbage is heated to 82° and centrifuged to remove the suspended solids. It is then allowed to separate and the floating grease is passed to a centrifugal separator, whence it is obtained in a fairly pure condition but may be washed and re-separated if desired. C. JEPSON.

Water-purifying material and its manufacture. H. KRIEGSHEIM and W. VAUGHAN, ASSRS. to PERMUTIT Co. (U.S.P. 1,757,372, 6.5.30. Appl., 19.9.22).—The base-exchange, water-softening properties of glauconite are improved by successive treatment with a weak solution of sodium silicate and an acid-reacting solution, e.g., aluminium sulphate. An improved product, stated to contain a higher proportion of combined silica and to be harder and less affected by heat than the natural product, is made by treating the mineral with a strong solution of sodium silicate, drying for about 1 hr. at 200°, and subsequently washing the crushed mass with calcium chloride solution (5%) before treatment with aluminium sulphate. C. JEPSON.

Purifying water. L. L. HEDGEPEETH, ASSR. to W. C. OLSEN (U.S.P. 1,757,526, 6.5.30. Appl., 17.4.28).—In the treatment of coloured waters etc., considerable improvement over the usual procedure is claimed if, after the water has been coagulated, e.g., with alum, and the p_H adjusted to the isoelectric point of the precipitated lakes to prevent peptisation, a further addition of a caustic coagulant, e.g., an alkaline aluminate, is made. This reacts with any unaffected alum and produces a secondary precipitation which is also brought to a maximum by adjustment of the p_H value. The dispersal of the matter carried down by the first precipitation is prevented by the coating of aluminium hydroxide produced by the secondary reaction. C. JEPSON.

Liquid treatment [with zeolites]. A. S. BEHRMAN, ASSR. to GEN. ZEOLITE Co. (U.S.P. 1,756,275, 29.4.30. Appl., 23.5.29).—Soluble impurities, e.g., alkali carbonates and bicarbonates, may be removed from water by conversion into calcium or magnesium bicarbonates by treatment with the zeolites of these metals and subsequent precipitation as carbonates by any known method, e.g., addition of calcium hydroxide. Deposition of the carbonates on the grains of zeolite is prevented by pre-carbonation if required. The removal of sodium bicarbonate is of importance in ice manufacture, as it renders the product brittle and opaque. C. JEPSON.

B.—APPLIED CHEMISTRY

DEC. 5 and 12, 1930.*

L—GENERAL; PLANT; MACHINERY.

Chemical engineering memoranda. XII. Exchange of heat: heaters and coolers. C. H. BUTCHER (Ind. Chem., 1930, 6, 404—407).—The total cost of pumping fluids or gases can be shown by a graph as a function of the velocity. The design of a heat exchanger involves this consideration, together with the provision of a sufficient velocity for efficient heat transfer. Types of tubular heaters and double-pipe heaters are discussed, and an example is given of the calculation of the design of a heat exchanger between two liquids. McAdams and Frost's formula (B., 1923, 587 A) for film coefficients is used with a 50% safety factor. C. IRWIN.

The packing of particles. A. E. R. WESTMAN and H. R. HUGILL (J. Amer. Ceram. Soc., 1930, 13, 767—779).—Apparatus is described by means of which equal true volumes of approximately spherical particles can be packed to the same apparent volume. The apparent volume per unit true volume, V_a , was determined for systems of one, two, and three sizes of roughly spherical particles. For one-size systems, V_a was approx. 1.60. With two- and three-size systems, the shape of the V_a curve or surface depends on the diameter ratios. It can be calculated either directly or by using an empirical relation between the minimum V_a and the diameter ratio of two-size systems. The method of calculation is capable of extension to systems of four or more sizes. In applying the packing theory to ceramic problems the relative strength, colour, shrinkages, etc. of the aggregate and bond must be considered, as well as the solubility of the aggregate in the bond and the importance of the surface area of the aggregate. F. SALT.

Removal of water during concentration. A. ALISON (Ind. Chem., 1930, 6, 402—403).—A nomograph is given which enables the weight of water removed from a solution during concentration to be read, given the weight of liquor and the initial and final percentages of solids. The chart is equally applicable to the drying of solids. C. IRWIN.

Oil viscosimeter. H. SCHAFFER (Petroleum, 1930, 26, Motorenbetrieb, 3, 4—8).—A viscosimeter is described which permits the viscosity of an oil to be determined directly in c.g.s. units. It is similar in design to the Engler viscosimeter except that the oil flows through a capillary tube of suitable dimensions, e.g., 4.5 cm. in length and 1.025 mm. in internal diam. The oil container and the capillary tube are surrounded by a water-bath which can be heated either electrically or by gas. The instrument has a viscosity range from 0.01 to 10 c.g.s. units with the same capillary tube.

Provision is made for increasing the air pressure in the oil container, and thereby shortening the time required for determining the viscosity of very viscous oils. The construction and method of using the instrument are described in detail. A. B. MANNING.

Immersion refractometer. A. DOLINEK (Z. Zuckerind. Czechoslov., 1930, 54, 627—630).—The "Askania" immersion refractometer gives, without change of prism, a refractive-index range of from 1.333 to 1.378, expressed as $d_{1.0000}$ —1.200 (for sucrose solutions), and reads to $d_{0.00001}$. It is mounted on a stand fitted with a mirror and a revolving tray holding ten 40-c.c. capsules. It can be used for determining moisture in fairly light-coloured products such as raw sugars and refinery massecuites and syrups, the readings being taken on half-normal solutions. Tables for these determinations are given, together with some examples showing a maximum error of 0.09% for the moisture content of raw beet sugars. J. H. LANE.

Tunnel kiln. TARRANT.—See VIII. Gas-purification plant. MICHEL.—See X.

PATENTS.

Furnaces and methods of operating the same. BABCOCK & WILCOX, LTD. From FULLER LEHIGH CO. (B.P. 334,757, 15.10.29).—The air supply to the furnace is passed through a grate at a velocity sufficient to keep the fuel substantially in suspension. The walls of the furnace are preferably inclined, and the grate bars are intermittently oscillated or tripped to discharge ash by mechanical means. B. M. VENABLES.

Preventing the formation of deposits or incrustations in rotary tubular furnaces. F. KRUPP GRUSONWERK A.-G. (B.P. 334,868, 10.6.29. Ger., 20.4.29).—Incrustations that may be caused, e.g., by liquefaction of a constituent of the charge may be prevented by baffling the flow of gases at the higher end of the kiln and by permitting a portion of the gases to escape from the interior of the kiln at an intermediate point, such portion being preferably passed through flues formed in the thickness of the lining towards the lower end and serving to heat the lining. B. M. VENABLES.

Charging of powders into furnaces and other reaction chambers. F. B. GRANT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,163, 14.6.29).—The outlet of a supply hopper is covered by a horizontal screen which is reciprocated vertically to discharge the material. The material should be subjected to a preliminary screening through apertures rather smaller than those of the feeder. B. M. VENABLES.

* The remainder of this set of Abstracts will appear in next week's issue.

Heat exchanger. BERGEDORFER EISENWERK A.-G. (B.P. 335,395, 8.10.29. Ger., 19.10.28).—A form of tubular exchanger of which the tubes can be easily removed for cleaning is described. B. M. VENABLES.

Heat-exchanging appliances. A. MORTREUX (B.P. 334,795, 23.11.29).—An apparatus is described in which air to be heated is passed across a bank of tubes through which products of combustion from an adjacent oil burner or other heating means are passed in a zig-zag manner. B. M. VENABLES.

[Heat]-insulating material for covering chill rooms, pipes, etc., and its method of application. J. GARLAND (B.P. 334,884, 10.6.29).—A mixture of 90 pts. of granulated cork, 8 pts. of calcined magnesite, and 2 pts. of crystallised magnesium chloride in water is applied to the surface, allowed to set, and then faced with a mixture of calcined magnesite, magnesium chloride solution, and wood flour. C. A. KING.

Treatment of steam-boiler water. R. E. HALL, Assr. to J. M. HOPWOOD (U.S.P. 1,759,615; 20.5.30. Appl., 4.12.26).—Foaming in steam boilers or evaporators may be reduced by the continuous admission of a small amount of insoluble gas in the form of fine streams distributed over and slightly below the steam-water interface. This method may be used either alone or in conjunction with certain anti-foaming compounds, e.g., those of the tannin type or higher alcohols derived from waxes, all of which accelerate the coalescence and disruption of the liquid bubble films with certain classes of water. C. JEPSON.

Drying apparatus. B. J. OWEN and R. O. DAVIES (B.P. 334,940, 13.6.29).—In an apparatus of the type in which currents of air or other drying gas pass upwards through a pervious conveyor, the air currents are divided and subdivided so that they may be regulated according to the decreasing moisture and increasing permeability of the material under treatment on the conveyor. B. M. VENABLES.

Drum dryer. MASCHINENFABR. IMPERIAL GES.M.B.H., and A. SCHWIETER (B.P. 334,515, 1.6.29).—In a drum dryer of the type where the material is contained in the annular space between an outer imperforate drum and an inner perforated one, and in which the drying gas is admitted at points distributed over the length of the kiln by means of an axial perforated conduit, part of the drying gas is caused to move concurrent with the fresh material and another part countercurrent to the partly dried material by drawing off the gas at some intermediate point in the length of the kiln through a stationary hood which surrounds it at that point. The material may be conveyed across the necessary openings in the shell either by covering them with gauze or by permitting the material to accumulate, at first, in the bottom of the hood. B. M. VENABLES.

Centrifugal dryers. G. H. ELMORE (B.P. 335,469, 16.12.29).—The straining surface is conical and the slope rather greater than the angle of repose of the retained solids, viz., 38° or more with respect to the axis. An internal distributor with spiral flights rotating at a different speed restrains the solid from passing too quickly down the cone. Fan blades are provided

outside the screen to create draughts in such a direction as to prevent re-mixing of solid and liquid.

B. M. VENABLES.

Gyratory crushers. E. B. SYMONS (B.P. 335,011, 6.8.29. U.S., 10.5.29).—A method of excluding dust from the main spherical bearing of a gyratory cone crusher is described. B. M. VENABLES.

Tumbling mills. AMER. FOUNDRY EQUIPMENT CO., Assees. of L. D. PEIK (B.P. 317,002, 9.7.29. U.S., 8.8.28).—The casing of the apparatus is stationary, and within it is a slat conveyor which follows a course forming, in side view, a semi-circle within a V, the former being the active part and having one arm higher than the other, so that the material may be discharged by reversing the conveyor. Sand-blast jets and means for continuously removing the used sand may be provided. B. M. VENABLES.

Tube mills. J. S. FASTING (B.P. 335,062, 24.9.29).—Preliminary to the long compartment of a tube mill is a coarse-grinding section having apertured grinding plates and sectionalised screens. Each screen is in the form of a half-round trough, and is provided with an internal hook-shaped guide for the material, thus rendering the whole screening surface effective. The bolts for securing the grinding plates are accessible from outside. B. M. VENABLES.

Filters. J. A. PICKARD and F. ROGERS (B.P. 334,569, 7.5.29).—A constructional form of filter having passages tapering in the direction of flow for retention of filter aid is described. One form comprises washers of sheet material, alternately large and small, the former having projections of definite height raised up out of the sheet on both sides, and the latter being plain. B. M. VENABLES.

Filter presses. ALLIANCE ARTIFICIAL SILK, LTD., W. H. YATES, and J. A. BLACK (B.P. 335,191, 13.6.29).—In a filter press of the closed-outlet type having an inspection chamber to each section to locate a broken cloth, a cock is provided on each side of each inspection chamber, and one of each pair is of the three-way type, so that a sample of liquor can be taken. B. M. VENABLES.

[Pressure] filtration of liquids and like operations involving the contact of liquid and solid material. A. R. JAHN (B.P. 334,663, 17.7.29).—The liquid is passed in a serpentine course through charcoal or similar material and then strained. Such apparatus is suitable for the decolorisation of sugar solutions, for recovery of precious metals from ore-bearing sands by cyanide solutions, etc. B. M. VENABLES.

Centrifuges. RAMESOHL & SCHMIDT A.-G. (B.P. 335,466, 10.12.29. Ger., 10.12.28).—The bulk of the solid matter is separated on the wall of the bowl, but a number of clarifying filters are situated within the bowl and rotate with it; these are in the form of vertical cylinders with axial outlets, and are surrounded by adjustable casings which are slit down the circumference at one point only, the slit forming the inlet to the filter. By fixing the casings in different angular positions the partially clarified liquor may be admitted to the filters under variable centrifugal pressure. B. M. VENABLES.

Centrifugal machines. A. F. DUNSMORE (B.P. 335,055, 18.9.29).—A centrifuge is provided with a detachable inner basket which, together with the permanent outer basket, forms a cone clutch; the use of positive driving lugs, and of drop doors in the bottom of the inner basket only, is also claimed.

B. M. VENABLES.

Separation of solids from liquids. BRIT. GLUES & CHEMICALS, LTD., A.-G. F. CHEM. PROD., and W. WACHTEL (B.P. 334,585, 6.6.29).—A device is described by which the bulk of the liquor is drawn away downwards (through apertures which are too large to choke) in a substantially clarified state, while the solid matter passes straight on horizontally in the remainder of the liquor and is afterwards screened out.

B. M. VENABLES.

Apparatus for separating liquids from fluids containing same. H. HOCKING (B.P. 334,200, 24.5.29).—The apparatus is of the deflecting type, the deflecting surfaces being concentric cylinders with outlets on opposite sides alternately. The interior parts may be withdrawn without disturbing the pipe connexions.

B. M. VENABLES.

Extraction of emulsified, suspended, or dissolved substances from liquids by means of volatile solvents. E. LANGFELDT and R. HELLERUD (B.P. 334,950, 14.6.29).—The solvent is introduced in a state of vapour into the liquid from the top or bottom, according to whether the condensed vapour is heavier or lighter than the liquid under treatment. The incoming vapour may be finely distributed by being passed through a porous wall.

B. M. VENABLES.

Evaporating plant. J. A. REAVELL (B.P. 334,623, 11.6.29).—A solution containing a volatile constituent, a salt, and impurities that may be precipitated (*e.g.*, ammonia, calcium acetate, and cellulosic matter, such as may result from the production of acetic acid by fermentation of cellulose and neutralisation of the acid with ammonia) is passed through an evaporator, a filter, and second evaporator in series, and then through a set of multiple-effect evaporators again in series. The first two evaporators are heated in parallel by live steam, and are at approximately the same temperature, the combined vapour from them serving as the heat supply to the first of the multiple-effect evaporators. Heat exchangers are used at suitable points.

B. M. VENABLES.

Catalytic gas reactions. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 335,411, 23.10.29. Ger., 28.11.28).—In processes in which the catalyst has to be heated or cooled, and as a substitute for a large number of small tubes, vessels of which the width is small compared with their length and breadth are used to contain the catalyst, any part of which is only a short distance from the heat-transmitting wall. A number of vessels may be placed abreast, and each may have external projections or shelves which intercalate with those on its neighbours, forming zig-zag passages for the external fluid.

B. M. VENABLES.

Deodorisation, pasteurisation, and concentration of fluids. TE AROHA DAIRY CO., LTD. (B.P. 314,022, 14.6.29. N.Z., 19.4.29).—The liquid is sprayed,

preferably by saturated steam, into a horizontally disposed cylindrical chamber heated by a steam-jacket and maintained under vacuum. Within the chamber are longitudinal paddles rotating at centrifugal speed, which spread the liquid in a layer round the wall to a depth which is determined by the position of the outlet to the vacuum-producing and condensing apparatus. Owing to the rapid treatment and low pressure, the temperature of the liquid does not substantially rise, and the process is suitable for delicate fluids such as milk; if operation at an elevated temperature is desired the liquid should be preheated.

B. M. VENABLES.

Eliminating or recovering substances from gaseous bodies. CARRIER ENG. CO., LTD., and C. L. SAINTY (B.P. 335,241, 18.6.29).—The gas is scrubbed by sprays of a liquid which is used repeatedly in counter-current stages. The liquor from the most concentrated stage only is run to waste, or may be treated for recovery of dissolved matter and re-supplied as fresh liquor to the dilute or gas-outlet end. Preferably the gas flows in a horizontal flue and the stages are divided from each other by moisture-eliminating baffles which drain into a launder which is also sectionalised by weirs and from which the spray pumps draw.

B. M. VENABLES.

Production of cleansing and polishing compositions. W. J. A. HUYZER (B.P. 335,005, 26.7.29).—The compositions comprise natural magnesite ground to 250-mesh and mixed with the usual ingredients to form a cream or paste.

L. A. COLES.

Apparatus for quantitative spectrum analysis. ADAM HILGER, LTD., and F. TWYMAN (B.P. 334,618, 11.6.29).—One part of a spectrum is brought alongside another part so that a line produced by a constituent which is to be determined is adjusted to be a continuation of a line belonging to a constituent present in known quantity. The adjustment of position is effected by blocks of glass with parallel faces, one or both of which may be pivoted; a tint wedge may be provided to match the brilliancy.

B. M. VENABLES.

Detecting and determining the quantity of moisture in a substance. H. C. FREEMAN (B.P. 335,308, 17.7.29).—The substance, *e.g.*, flour, is mixed with another substance, *e.g.*, calcium carbide, that will generate gas by reaction with water. The mixing is effected, if desired with the aid of coarse sand, in a closed vessel provided with a pressure gauge. A correction for undecomposed water is necessary, and is of the order of 20% of the moisture.

B. M. VENABLES.

Cooling tower. R. A. LEWIS (U.S.P. 1,778,364, 14.10.30. Appl., 29.4.27. U.K., 29.4.26).—See B.P. 272,325; B., 1927, 639.

Apparatus for carrying out exothermic catalytic gas reactions. H. HARTER (U.S.P. 1,779,092, 21.10.30. Appl., 9.7.28. Ger., 10.8.26).—See B.P. 275,983; B., 1928, 391.

[Controlling the combustion of fuel in boiler] furnaces. J. GORDON & Co., LTD. (B.P. 315,000, 5.7.29. U.S., 7.7.28).

[Continuous] linings for furnace arches, walls, etc. H. W. SPENCER (B.P. 336,520, 28.1.30).

[Cooling the walls of] furnaces. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 336,263, 10.7.29).

[Boiler-steam] dryers. T. SUGDEN (B.P. 335,186, 16.3.29).

Cooling or drying machinery [for cattle cakes etc.]. A. W. SIZER (B.P. 334,875, 9.3.29).

Cooling systems. ELECTROLUX, LTD., ASSES. OF PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 334,696, 27.8.29. Swed., 2.3.29).

[Absorption] refrigeration [apparatus]. ELECTROLUX, LTD., ASSES. OF A. LENNING and R. S. TAYLOR (B.P. 336,506, 31.12.29. U.S., 4.1.29).

Absorption refrigerating apparatus. H. D. FITZPATRICK. From N.V. "KODOWA" REFRIGERATOR Co. (B.P. 336,275, 12.6.29).

Refrigerators for cooling liquids. ELECTROLUX, LTD., ASSES. OF R. S. TAYLOR (B.P. 316,139, 16.7.29. U.S., 23.7.28).

Apparatus [gun] for spraying liquids. R. E. SERPOLIER and M. A. DROUOT (SERPOLIER & DROUOT) (B.P. 336,173, 27.12.29. Fr., 13.8.29).

Fire-extinguishing systems. Fyre-Freeze Corp. (B.P. 336,330, 29.7.29. U.S., 23.11.28).

Gastight unions or joints. C. R. BURCH, F. E. BANCROFT, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 336,015, 13.7.29).

Gas detector (B.P. 334,640). Distillation apparatus (U.S.P. 1,755,778).—See II. Friction- and heat-resisting articles (U.S.P. 1,758,055).—See V. Compressed mixtures (B.P. 334,448).—See VII. Tunnel kilns (B.P. 334,951). Refractory cement (U.S.P. 1,763,882).—See VIII. Heat-conducting cement (B.P. 335,086).—See IX.

II.—FUEL; GAS; TAR; MINERAL OILS.

Economic value of coal cleaning in relation to the problem of dust and sulphur emission from chimneys. R. LESSING (Fuel, 1930, 9, 348—358).—The ash collected from various points in a pulverised-fuel-fired boiler installation has been compared physically, chemically, and microscopically with that prepared by incinerating the coal fractions (dust through 50 I.M.M. sieve, clean coal $d < 1.35$, middle fraction $d > 1.35$ and < 1.5 , refuse $d > 1.5$) in the laboratory. The results indicate that the portion of the coal ash which reaches farthest in the boiler system is that derived from the refuse. The ash from the fines and from the clean coal is largely retained at an earlier stage in the combustion chamber and economiser, probably because its lower fusing point causes the particles to sinter together to form larger aggregates. The abolition of the flue dust nuisance can therefore be brought about by removing the refuse from the coal, a process now commercially possible by gravity separation. Coal cleaning also diminishes the trouble due to clinker formation in plants using mechanical stoking, and offers a more rational and more economical means of removing sulphur from the flue gases than by washing the latter with water. It is calculated that the use of cleaned instead of uncleaned

coal effects a net saving of about 1s. per ton in the case of mechanical stoking, and 1s. 3d. in the case of powdered fuel.

A. B. MANNING.

Carburetted water-gas and influence of temperature on the composition of the tar formed. T. B. GLOVER (Gas J., 1930, 191, 702—704; 192, 38—42).—Tar samples were collected from a carburetted water-gas plant during periods in which different cracking temperatures and rates of oil injection were maintained. These tars were analysed by sulphonation and nitration, and the formation of naphthalene was particularly noted. As the temperature was increased, with approximately constant rates of oil feed, from 720° to 740°, 770°, 800°, and 820°, the paraffin content of the tar decreased, but the percentage of hydrocarbons attacked by concentrated acids and of those forming crystalline picrates increased. Tests at 770° with different oil rates showed only small changes in the composition of the tar; in these tests another plant and a different oil were used, so that the results are not directly comparable with the first series. Many incidental measurements are recorded for changes in gas composition, rates of gas production, efficiency of oil cracking, and time of contact in the cracking chambers. R. H. GRIFFITH.

Thermal decomposition of low-temperature tar of Fushun coal. I. T. MIZOSHITA (Rep. Lab. S. Manchuria Rly., 1929, 33—35).—The thermal decomposition of the tar under various conditions of temperature and pressure has been studied. The yield of cracked distillate, and the distillation ranges, compositions of gas and of cracked gasoline, together with the characteristics of refined gasoline, are tabulated. The yield of 13.5% of gasoline which is obtained at 430° and under 25—30 atm. is somewhat lower than that from neutral oil, owing to the presence of acidic matter. The cracked gasoline affords a good fuel having antiknock properties.

H. J. DOWDEN.

Utilisation of high-temperature coal-tar pitch.

I. Preparation of creosote oil substitute from pitch. M. TANAKA, K. MORIKAWA, and I. MORIKAWA.

II. Preparation of active carbon from the extraction residue. S. WATANABE (Rep. Lab. S. Manchuria Rly., 1929, 29—32, 32—33).—I. By extracting pitch at different temperatures and pressures with various solvents a mixture of hydrocarbons and resinous matter with high b.p. and viscosity was obtained. Benzol and naphtha give yields of 65—70% of highly viscous oil containing much resin, which can be removed by dilute chromic acid solution. The presence of aluminium chloride before extraction destroys resins, but reduces the yield of oil to 30—40%. Extraction for 3 hrs. with gasoline-naphtha (equal vols.) at 100—110° and 1 atm. gave a yield of 46% of good quality oil needing no further refining. The solvent may be completely recovered, but it is desirable to leave 15—20% of solvent to lessen the viscosity. When mixed with an equal volume of creosote, the mixture is a suitable substitute for creosote oil.

II. The residue from the extraction of pitch with organic solvents consists of about 30% of "free carbon" which by calcining with potassium sulphate in equal proportions yields a highly activated carbon, the

decolorising power of which is superior to that of either animal charcoal or commercial active carbon.

H. J. DOWDEN.

Tar-forming value and oxidisability of hydrocarbon oils and fats. R. BÜRSTENBINDER (Chem.-Ztg., 1930, 54, 782—783).—The determination of the tar-forming value (cf. Kissling, B., 1908, 1053) by the usual method leads to uncertain results: with many oils relatively greater changes occur if oxidation is prolonged beyond 70 hrs., and oxidation is also markedly affected by the use of metallic catalysts. A more useful value is obtained by heating the oil (100 g.) at 120° with 1 g. of pure finely-divided copper oxide for 9, 70, or 360 hrs. The acid and saponification values of the oil are determined before and after the oxidation, and the differences between the corresponding values are returned as the "acid-" and "saponification-oxidation values" after the stated times of treatment. A blank test should be made.

E. LEWKOWITSCH.

Separation [from high-temperature tar] and synthetic preparation of phenol. T. MIZOSHITA (Rep. Lab. S. Manchuria Rly., 1929, 36).—High-temperature tar from the Anshan Iron Works was found to contain phenol 0.1897% and cresols 0.5691%, the values being relatively low owing to the high temperature of coking. In the synthesis of phenol a yield of 84% is obtained by the interaction of 2 mols. of sodium chloride and 1 mol. of chlorobenzene for 2 hrs. at 320°, using cupric or cuprous chloride or metallic copper as catalyst.

H. J. DOWDEN.

Determination of naphthalene in mixtures with phenols and with anthracene. M. M. CASTILLA (Anal. Fis. Quím., 1930, 28, 1084—1088).—Phenols do not interfere with the formation of hydrocarbon picrates if the proportion of phenol in the original mixture does not exceed 50%. Knublauch's method (B., 1917, 702) for the determination of naphthalene yields accurate results if not more than the theoretically necessary quantity of picric acid be added and if the precipitate be washed with sufficient 0.2% picric acid solution; with anthracene the results are unsatisfactory.

H. F. GILLBE.

Determination of the acid constituents of shale oil and its residues. E. VON PEZOLD (Chem.-Ztg., 1930, 54, 678—679).—Of several methods used to determine the acid constituents of a "topped" Estonian shale oil the following was the most accurate, and is recommended for the purpose. A solution of the oil (5 g.) in 40 c.c. of benzene was extracted with dilute (2%) sodium-hydroxide. The extract was washed with benzene to remove dissolved neutral oils. The alkaline solution was then freed from colloidal suspended matter by being boiled, set aside, and filtered. The filtrate was acidified and extracted with ether. The ethereal solution was washed with water, dried over anhydrous sodium sulphate, filtered, the ether removed by evaporation on the water-bath, and the residue dried at 50° to constant weight. The result is probably slightly low owing to polymerisation of part of the acid constituents by the alkali.

A. B. MANNING.

Ichthyol oil from Kashpir shale. E. V. RAKOVSKI and S. I. SOKOLOV (J. Appl. Chem., Russia, 1930, 3,

81—89).—The characteristics of the oil and its fractions are described. The oil differs from the Scottish shale oils.

CHEMICAL ABSTRACTS.

Vanadium in petroleum ash. R. K. BROZ (Arh. Hemiju, 1930, 4, 86—91).—Maracaiba (Venezuela) petroleum ash contains 43.58% V. R. TRUSZKOWSKI.

Viscosimeter. SCHAFFER.—See I. **Consistent fats.** PYHÄLÄ.—See XII. **Petroleum oil for sprays.** RICHARDSON.—See XVI. **Activity of decolorising carbons.** VAŠÁTKO.—See XVII.

PATENTS.

Coal and refuse tester. H. MORGAN (U.S.P. 1,758,756, 13.5.30. Appl., 5.9.29).—An apparatus for determining the proportion of coal and slate in a mixture comprises a tank containing a liquid, e.g., zinc chloride solution, in which the coal will float and the slate sink, a container for the sample to be tested, which fits within the tank and has a perforated wall, and a trap, which fits closely within the container and has also a perforated wall. A hinged rod extends across the lower end of the trap, the bottom of which is composed of two parts hinged to the rod and capable of being closed after the trap has been inserted into the container. Thus after the separation of the coal and slate is complete the bottom of the trap may be closed and the separated coal removed by lifting the trap from the container.

A. B. MANNING.

Production of comparatively smokeless fuels from lignites, bituminous coals, and the like. METALGES, A.-G., and O. HUBMANN (B.P. 333,811, 29.10.29).—The raw fuel is separated into dust (0— $\frac{1}{4}$ in.) and coarser material. The latter is subjected to low-temperature carbonisation and the product, either as a whole or after screening off the large lumps, is mixed with the dust and briquetted. If necessary, the carbonisation product is treated with air before briquetting in order to diminish its tendency to spontaneous ignition.

A. B. MANNING.

Carbonisation of coal and the like. S. R. ILLINGWORTH, and ILLINGWORTH CARBONIZATION CO., LTD. (B.P. 333,597, 18.4.29).—The charge of coal is tamped as it is fed into the retort, suitable apparatus for which is described. A quantity of fines is charged with the coal. The process yields a coke of increased density and hardness, and permits the use of coals of lower caking quality.

A. B. MANNING.

[Carbonisation] retorts. F. E. HOBSON (B.P. 333,563, 13.5.29).—A vertical retort has a central heating tube and a number of radially disposed, vertical heating flues arranged between the central tube and the outer casing. The central heating tube is provided with angular vanes on its outer wall, and can be given a combined reciprocating and rotary motion. The other heating flues are separated by louvres, which may be either fixed or interchangeable. The material to be carbonised is fed from a hopper into the space between the central tube and the surrounding heating flues, and is agitated and at the same time conveyed through the retort by the motion of the former. The volatile products of carbonisation pass between the louvres into the outer annular space and thence to a gas chamber and discharge pipe.

A. B. MANNING.

Coke ovens, carbonising chambers, and the like. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 334,050, 16.8.29).—The individual offtakes from the ovens, which are connected to the common collecting main during carbonisation, are connected to the suction or foul main during the charging period in order to avoid abnormal pressure fluctuations in the collecting main. For this purpose a valve-controlled by-pass is provided between the gas offtake of each oven and the suction main; the valve can be automatically and gradually closed when charging is complete. The ducts connecting the offtakes with the collecting main may also be provided with valves. The fresh charge is preferably introduced into the chamber through charging holes near the ends thereof, the gas offtake being arranged centrally. A. B. MANNING.

Coking retort. H. TEATER (U.S.P. 1,758,183, 13.5.30. Appl., 19.11.27).—A vertical retort of sheet metal has a narrow, annular, carbonising chamber, surrounded on the outside and inside, and also on top, by heating chambers through which superheated steam can be circulated. The retort is provided with charging doors at the upper end and with a hinged drop-bottom through which the treated material is discharged. The process is carried out in two stages: in the first the coal is externally heated to drive off the volatile products, and in the second superheated steam is passed through the charge. A. B. MANNING.

Coking retort oven. J. BECKER, ASSR. to KOPPERS Co. (U.S.P. 1,755,381, 22.4.30. Appl., 16.5.21. Renewed 24.1.30).—Each of the heating walls of a coking retort oven consists of two series of horizontal combustion flues. The regenerators below the coking chambers and parallel thereto are arranged in two series, one at each side of the battery, one series operating for inflow while the other operates for outflow. The regenerators of each series are disposed in groups of three, the central inflow operating generator supplying gas and the other two supplying air. The groups of generators of each series respectively are connected by ducts with the opposite ends of both series of combustion flues. The ovens may be heated by a gas, *e.g.*, producer gas, obtained from a separate generator. A. B. MANNING.

Recuperative coking retort oven. J. VAN ACKEREN, ASSR. to KOPPERS Co. (U.S.P. 1,758,544, 13.5.30. Appl., 29.9.21).—The heating walls of a coking retort oven comprise two series of vertical combustion flues. Below the coking chambers are recuperator units disposed into two series operating alternately with one another. The inflow and outflow passages of the recuperator units of both series are connected by ducts with the tops and bottoms respectively of both series of combustion flues of each heating wall. Separate fuel gas connexions communicating with the tops of both series of combustion flues are provided. The setting may be operated with an extraneously derived generator gas or with coke-oven gas burned in an atmosphere of ordinary preheated air or in a mixture of air and a neutral gaseous diluent. A. B. MANNING.

Production of coke and apparatus therefor. H. KOPPERS (U.S.P. 1,758,524, 13.5.30. Appl., 5.6.22. Renewed 7.7.27. Ger., 7.10.21).—Coal which has been

ground to uniform size is distilled in continuously operated vertical retorts or oven chambers. Each chamber is provided with a superstructure which is open at the top and along the walls of which are rods which are intermittently moved up and down. In this manner passages are formed and maintained open in the upper portion of the heated distillation chambers so that the gases can escape readily from the coking material to suitably placed offtakes. The fine coal dust forms a gastight closure of the chamber so that special closing devices may be dispensed with.

A. B. MANNING.

Furnaces for briquetting small coal and the like. H. ALEXANDER (B.P. 333,713, 19.7.29).—Powdered bituminous coal, either alone or blended with another coal, is charged into metal containers having removable perforated caps. The charged containers are carried on trucks into an externally heated furnace, wherein the material is heated at 315–425°. When the heat treatment is completed the trucks are withdrawn from the furnace and the briquetted material is discharged from the containers. The volatile products evolved during the treatment are collected, and the non-condensable gases utilised for heating the furnace. The furnaces are arranged in batteries, and the spent gases from each heating process utilised for preheating the charge in the next furnace. A. B. MANNING.

Production of finely-divided carbon. N. GOODWIN, ASSR. to DELANO LAND Co. (U.S.P. 1,758,152, 13.5.30. Appl., 10.3.24. Renewed 23.10.29).—A mixture of a hydrocarbon gas or vapour and air, in proportions insufficient for complete combustion, is burned in an externally heated chamber, the temperature during combustion being maintained at above 650°. The air for combustion and the hydrocarbon gas are preheated, the former in a suitably arranged regenerator, and the latter by the waste gases from the external heating of the combustion chamber. The yield and quality of the carbon produced may be varied by varying the degree of preheating of the air and gas.

A. B. MANNING.

Reactivation of charcoal. E. W. RICE (U.S.P. 1,758,202, 13.5.30. Appl., 31.12.25).—Charcoal laden with adsorbed organic matter is passed through an inclined rotary kiln wherein it is subjected to the action of hot gases containing sufficient oxygen to burn the adsorbed impurities. The temperature in the kiln is maintained at 200–400° and the hot gases are preferably continuously recirculated through the apparatus.

A. B. MANNING.

Apparatus for reactivating charcoal. C. N. WHITAKER, ASSR. to SHELL OIL Co. (U.S.P. 1,744,429, 21.1.30. Appl., 7.12.26).—The charcoal is placed in a cylindrical basket which is supported within a vertical cylindrical chamber in such a manner that superheated steam entering the basket through a central nozzle at the bottom thereof passes up through the material, down through the annular space between the basket and the outer vessel, and thence to a discharge pipe. Treatment of the charcoal with superheated steam at 600–650° for 1 hr. restores its efficiency to at least 92% of the initial efficiency. A. B. MANNING.

Water-gas generators. A. BREISIG (B.P. 333,842, 2.12.29. Austr., 3.12.28).—In a water-gas generator working with alternate "blow" and "run" periods, a part of the waste gases produced during the "blow" is utilised for producing and superheating the steam required for the production of water-gas, by means of a regenerator with superposed vaporiser. The other part of the waste gases is burned in a second regenerator which is connected to the waste-heat boiler. During the period of steaming ("run") the heat stored in the second regenerator is carried over to the waste-heat boiler by means of a carrying gas, *e.g.*, air or the water-gas produced. When heat is supplied continuously to the boiler in this manner its heating surface can be considerably reduced.

A. B. MANNING.

Gas generators. HUMPHREYS & GLASGOW, LTD., Assees. of J. M. RUSBY (B.P. 334,133, 19.11.29. U.S., 21.1.29).—In a gas generator using bituminous coal as fuel, a number of diaphragms, preferably of metal, extend downward through the upper part of the fuel bed, thereby effecting a better distribution of the gases passing through the carbonising zone. The diaphragms may be tapered in cross-section, and may be given a slow, vertical, reciprocating motion. They may be radially mounted, but do not extend to the walls of the generator.

A. B. MANNING.

Production of combustible gas. SEMET-SOLVAY ENG. CORP., Assees. of C. H. HUGHES and F. W. STEERE (B.P. 312,071, 13.5.29. U.S., 18.5.28).—The formation of clinker on the lining of a gas generator is prevented by introducing a liquid, preferably water, into the fuel bed through orifices in the side of the generator. Water may be injected also into the base of the fuel bed in order to chill the clinker therein and facilitate its crushing and removal by the rotary grates, which are preferably of the type adapted to operate in conjunction with a vertically reciprocating poking device. A flux, *e.g.*, soda, may be introduced into the generator in order to lower the fusing point of the clinker.

A. B. MANNING.

Producer-gas process. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,743,717, 14.1.30. Appl., 31.8.21. Ger., 14.3.18).—A water and steam reservoir is arranged above a producer provided with a water-jacketed metallic shaft, and the two are so connected that water circulates by a thermosiphon system from the reservoir to the bottom of the water-jacket and from the top of the latter back to the reservoir. The water enters the lower part of the jacket at a temperature (about 100°) sufficiently high to prevent condensation of steam on the shaft wall in the ash zone, and so avoid excessive corrosion of the wall. The circulating system is under sufficient pressure to permit steam formation only in the reservoir. The steam, mixed with air, is supplied to the generator for the production of gas therein.

A. B. MANNING.

Gas-purification process. C. J. RAMSBURG, Assr. to KOPPERS Co. (U.S.P. 1,743,479, 14.1.30. Appl., 24.1.23. Renewed 6.12.27).—The gas is purified in two stages. In the primary absorber the greater part, preferably about 90%, of the hydrogen sulphide and hydrocyanic acid is removed by washing with a solution of sodium carbonate (*cf.* U.S.P. 1,390,037; B., 1921,

762 A), or similar process. A portion of the revived solution is treated with ferric oxide or other compound capable of removing the last traces of sulphide therefrom, and is then used to effect a final purification of the gas in the second absorption stage. The used solution may be returned to the primary absorber or may be again treated with ferric oxide and recirculated in the second absorber.

A. B. MANNING.

Gas detector. M. H. ESPES and B. WEISER (B.P. 334,640, 19.6.29).—An electric circuit is closed by the differential expansion of two adjacent wires one of which is provided with pellets of spongy platinum.

B. M. VENABLES.

[Distillation] retort. J. N. VANDEGRIFT, Assr. to C. M. BARNETT (U.S.P. 1,758,770, 13.5.30. Appl., 8.6.23).—The shale or other material to be distilled is fed continuously on to a rotating horizontal table, which forms the floor of the retort, and is spread thereon in an even and comparatively thin layer by means of a vertical blade arranged at an angle to the radius of the table and suitably spaced therefrom. A second blade held close to the surface of the table serves to direct the treated material into a discharge chute. The table, which is heated from below, is formed of two parallel plates, the space between them being filled with molten lead. The walls of the retort, which is of bee-hive form, are of firebrick, or are lined with refractory material. The volatile distillation products are withdrawn through a central flue in the top of the retort and are passed through a treating chamber, which is maintained at such a temperature that only the higher-boiling products, *e.g.*, paraffin wax, are condensed therein.

A. B. MANNING.

Distillation apparatus [for oils]. J. A. GIBB, Assr. to ZIELEY PROCESSES CORP. (U.S.P. 1,755,778, 22.4.30. Appl., 20.11.25).—In a multi-unit still for the continuous distillation of oil in vacuum, means are provided for separating sediment from the oil as it passes continuously from one still unit to another. The oil is pumped up through a vertical separating chamber, the lower end of which opens into a sediment tank, and is sealed by the oil therein. The height of the chamber is sufficient to provide a head of liquid equal to the difference between the atmospheric pressure and the vacuum maintained in the still.

A. B. MANNING.

Treatment of hydrocarbons, including hydrocarbon derivatives. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,759,620, 20.5.30. Appl., 5.11.19).—Mixed hydrocarbons in a finely-divided condition are subjected to partial oxidation to produce alcohols and aldehydic compounds, and the product is passed through a heated reaction zone in which oxygen is present and in the presence of a catalyst containing a compound of a metal having high m.p. and low at. vol. (*e.g.*, oxides of molybdenum, chromium, tungsten), under conditions preventing complete oxidation of the major portion of the material treated.

H. S. GARLICK.

Purification of hydrocarbons. J. C. BLACK and J. R. MCCONNELL, Assrs. to RICHFIELD OIL Co. (U.S.P. 1,759,730, 20.5.30. Appl., 7.6.27).—A hydrocarbon distillate, *e.g.*, gasoline, is treated with aqueous caustic alkali containing manganese dioxide in suspension, at

about 163° and under sufficient pressure to prevent substantial volatilisation of the hydrocarbons. The treated distillate is separated and subjected to steam distillation at not above 163°. H. S. GARLICK.

Refining of hydrocarbons. R. CROSS, Assr. to CROSS DEVELOPMENT CORP. (U.S.P. 1,760,585, 27.5.30. Appl., 21.11.23).—Hydrocarbon oil, heated to a maximum of 315° and under sufficient pressure to maintain it in the liquid phase, is filtered through a bed of bentonite intimately combined with a copper salt adapted to remove sulphur ingredients, and the treated oil collected separately. H. S. GARLICK.

[Continuous] decolorisation of hydrocarbons. I. C. CARPENTER and A. R. MOORMAN, Assrs. to CONTACT FILTRATION Co. (U.S.P. 1,759,343, 20.5.30. Appl., 17.9.26).—Petroleum oil is heated to above 100° by circulation through a system comprising a tank, a pump, and a heater. Mixed oil and decolorising clay are fed to the main bulk of oil, whereby any water in the clay evaporates on contact with the heated oil and is permitted to escape. Any scale-forming material which may be present in the water is deposited in the oil in solid form, and, mixing with the clay, is carried through the heater. Oil and clay are discharged from the heater in sufficient amount to maintain substantially constant the volume of fluid in the tank, and are treated in any suitable manner to cause their separation. The mixture of oil and clay is heated to a temperature at which the effectiveness of the clay is increased, but not sufficient to vaporise any substantial amount of oil. H. S. GARLICK.

Deodorisation of petroleum. A. E. FEW, JUN., Assr. to SUN OIL Co. (U.S.P. 1,761,152, 3.6.30. Appl., 27.8.26).—Lubricating oil distillates are freed from compounds of objectionable odour, not removable in the distillation process, by turbulently flowing such distillate, heated in order to reduce its viscosity, in a thin film over a series of surfaces of substantial area, while subjecting the oil, without the aid of external heat, to reduced pressure to effect the release of the contained gases, which are continuously withdrawn and removed. H. S. GARLICK.

Purification of lubricating oil distillates. M. L. CHAPPELL, Assr. to RICHFIELD OIL Co. (U.S.P. 1,761,328, 3.6.30. Appl., 31.7.28).—Mineral lubricating oil stocks, after treatment with sulphuric acid and separation of the acid sludge, are treated with the requisite quantity of sodium hydroxide solution at approx. 65° to neutralise the free mineral acids alone and the aqueous solution of the mineral acid salts is removed. The lubricating stock is then treated, at 121–137°, and under a pressure of approx. 50 lb./in.², with sufficient caustic potash solution to neutralise all organic acid emulsifying constituents present, and separated from the aqueous solution. H. S. GARLICK.

[Insulating] oil treatment. S. BOYER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,760,539, 27.5.30. Appl., 2.2.25).—Mineral oil intended for electrical insulating purposes is brought into contact with oxycellulose resulting from nitric acid oxidation of cellulose for sufficient time to remove water and acid substances from the oil, and is then filtered. H. S. GARLICK.

Manufacture of paraffin. C. A. WARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,760,096, 27.5.30. Appl., 20.11.25).—A foots oil from the treatment of hydrocarbon oils by the wax-sweating process is reduced approx. 50% by redistillation without substantial cracking to obtain a residue which on cooling is solid at about 38° and can be sweated directly. H. S. GARLICK.

Recovery of organic acids from oxidation products of solid paraffin and like waxes. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 333,904, 14.3.29).—The crude products are treated with an amount of an inorganic alkali sufficient to neutralise exactly the free acids present, and, after drying if desired, the unsaponifiable material and esters are removed by extraction with an organic solvent. The residual salts are then decomposed with a mineral acid. A. B. MANNING.

Cracking and rectifying petroleum oils and the like. J. M. WADSWORTH, Assr. to UNIVERSAL OIL PRODUCTS Co. (Re-issue 17,831, 14.10.30, of U.S.P. 1,692,476, 20.11.28).—See B., 1929, 120.

Distillation of crude [mineral] oils. F. TINKER, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,779,222, 21.10.30. Appl., 9.2.27. U.K., 28.4.26).—See B.P. 274,959; B., 1927, 741.

Coke-oven doors. H. GOURLEY (B.P. 336,544, 13.3.30).

Road-surfacing materials (B.P. 334,336).—See IX. **Ink** (B.P. 334,370).—See XIII. **Mulches** (B.P. 319,783).—See XVI.

III.—ORGANIC INTERMEDIATES.

Decomposition of methyl alcohol in presence of the zinc-chrome catalyst. V. A. PLOTNIKOV and K. N. IVANOV (J. Chem. Ind., Russia, 1929, 6, 940–943).—The catalyst of zinc and chromic oxides (10:1) employed was carried on iron-free asbestos. Decomposition was first observed at 240–250°, and was vigorous at 300°. Increase in the speed and change in the character of the decomposition were observed at 335–350°, as compared with 320–325°. Polymerides of formaldehyde, together with hydrogen, carbon monoxide, and carbon dioxide are formed. The carbon dioxide, although strongly absorbed, does not act as a catalyst poison. The catalyst is very stable.

CHEMICAL ABSTRACTS.

Reaction of diallylmalonylcarbamide (dial). F. LAGARCE (J. Pharm. Chim., 1930, [viii], 12, 364–365).—A freshly-prepared 1% solution of vanillin in sulphuric acid gives with minimal quantities of diallylmalonylcarbamide, on warming, a stable, cherry-red colour which is not given by any other alkyl derivative of malonylcarbamide. C. C. N. VASS.

Nitration of trimethylbenzenes and properties of their nitro-derivatives. N. A. KHOLEVO (J. Appl. Chem., Russia, 1930, 3, 251–254).—Trinitromesitylene and trinitro-*ψ*-cumene are more susceptible to mechanical shock than is trinitrotoluene, although they have a high ignition temperature and do not easily detonate with mercury fulminate. CHEMICAL ABSTRACTS.

Dehydrating action of coal ash. TANAKA. **Synthesis of phenol from tar.** MIZOSHITA. Deter-

mination of naphthalene in presence of phenols and anthracene. CASTILLA.—See II. Condensation of formaldehyde with terpenes of fir oil. USHAKOV and SOKOLOV.—See XIII. Ether for narcosis. V. D. BERG.—See XX.

PATENTS.

Manufacture of [nitrogenous] products from gaseous acetylene hydrocarbons or aldehydes [and ammonia etc.]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,193, 23.2.29).—Acetylenes and/or aliphatic aldehydes are passed with ammonia or amines at 250°–500° over a catalyst comprising compounds of aluminium or of heavy metals (*d* over 4.4) of groups II to VII, and the products are hydrogenated, without isolation, in presence of a hydrogenating catalyst which may be in the same or a second reaction chamber. Hydrogen may be added as required. The hydrogenation of the freshly-formed acetonitrile leads to monomethylamine, instead of the usual mixture of amines. At high gas velocities, *e.g.*, 10 litres per hr. per litre of catalyst, less acetonitrile and more heterocyclic compounds are produced. A mixture of acetylene (7 pts.), ammonia (7 pts.), and hydrogen (20 pts.) is passed first over zinc oxide and thoria on silica gel at 350°, and then over nickel on silica gel at 150°; the product (140 c.c. from 100 litres of acetylene) is mainly piperidine and monoethylamine. The proportion of the former is increased by adding steam to the gas mixture.

C. HOLLINS.

Manufacture of cyclic hydrocarbons from gaseous acetylene hydrocarbons or aldehydes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,203, 23.2.29. Cf. B.P. 334,193, preceding).—In the absence of ammonia or amines cyclic hydrocarbons are obtained; *e.g.*, acetylene and hydrogen (2 pts.) give hydrogenated benzenes when passed over zinc chloride on silica gel at 450° and then over nickel at 200°.

C. HOLLINS.

Manufacture of alcohols from gaseous acetylene hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,223, 23.2.29. Cf. B.P. 334,193, preceding).—In presence of steam and absence of ammonia or amines, alcohols are formed; *e.g.*, acetylene, saturated by passing through water at 90°, is led over zinc oxide on silica gel at 380°, and then with hydrogen over nickel at 200° to give as main product ethyl alcohol.

C. HOLLINS.

Manufacture of condensation products from diolefines and [aromatic] hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 315,312, 5.7.29. Ger., 11.7.28).—Butadiene or other diolefine is condensed with an aromatic hydrocarbon (toluene, xylene, mesitylene, tetrahydronaphthalene) in presence of a metal of the alkali or alkaline-earth series under pressure and at raised temperature. Toluene at 80–90° gives ϵ -phenyl- α -pentene, b.p. 203–204°.

C. HOLLINS.

Manufacture of polymerisation products from diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,184, 25.5.29).—The polymerisation of diolefines (butadiene) in presence of sodium etc. commences readily and proceeds smoothly in dioxan at 40–60°.

C. HOLLINS.

Manufacture of anthraquinonecarboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,692, 11.4.29. Cf. B.P. 321,916; B., 1930, 316).—Aqueous suspensions of benzanthrone, or its derivatives substituted in the anthrone nucleus, are oxidised with oxygen under pressure (50 atm.) above 180° in presence of alkali. Benzanthrone gives anthraquinone-1-carboxylic acid, m.p. 292°.

C. HOLLINS.

Manufacture of formaldehyde. H. WADE. From BAKELITE CORP. (B.P. 336,282, 10.7.29).—See U.S.P. 1,738,745; B., 1930, 601.

Oxidation of acetaldehyde to acetic acid. S. W. ROWELL and H. S. HIRST, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,778,511, 14.10.30. Appl., 20.9.28. U.K., 18.10.27).—See B.P. 304,350; B., 1929, 275.

Obtaining from seaweed a stable form of alginic acid and its compounds. O. REYNARD (U.S.P. 1,778,688, 14.10.30. Appl., 3.5.28).—See B.P. 316,119; B., 1929, 807.

Preparation of [alkylated] phenol compounds from dihydroxydiphenylmethane derivatives. H. JORDAN, Assr. to CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (U.S.P. 1,778,858, 21.10.30. Appl., 5.7.27. Ger., 14.7.26).—See B.P. 274,439; B., 1929, 164.

Dye intermediate from halogenated [o-] benzoylbenzoic acids. J. THOMAS and H. A. E. DRESCHER, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,779,221, 21.10.30. Appl., 7.12.26. U.K., 24.12.25).—See B.P. 273,347; B., 1927, 648.

Evaporating plant (B.P. 334,623).—See I. **Organic acids from waxes** (B.P. 333,904).—See II. **Ethoxides** (B.P. 334,388). **Catalysts** (U.S.P. 1,746,781–3).—See VII. **Flotation of ores** (U.S.P. 1,763,851).—See X. **Aliphatic acids by fermentation** (B.P. 334,900).—See XVIII.

IV.—DYESTUFFS.

Turmeric and annatto tests. WHITE.—See XIX.

PATENTS.

Manufacture of sulphide dyes from azines. R. W. POPE and M. WYLER, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,778,165, 14.10.30. Appl., 19.7.28. U.K., 8.9.27).—See B.P. 299,152; B., 1928, 922.

Manufacture of acid dyes of the phenonaphthasafranine series. P. LAEUGER, Assr. to J. R. GEIGY & CO. ANON. (U.S.P. 1,779,279, 21.10.30. Appl., 7.9.28. Ger., 21.9.27).—See B.P. 297,441; B., 1930, 364.

Manufacture of [azo] dyes containing metal. F. STRAUB, J. GYR, and O. KAISER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,779,298, 21.10.30. Appl., 1.10.28. Switz., 8.10.27).—See B.P. 298,494; B., 1930, 317.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Degumming of silk by proteolytic enzymes. L. MEUNIER and J. VALLETTE (Rusca, 1930, 4, 1513–1521).—Minimum swelling of silk takes place at p_H about 4.2. The degumming activity of pancreatin is maximal at p_H about 8; pancreatin has practically no

effect on fibroin. Freshly-prepared pancreatin should be used; relatively large amounts (1.5–6.3% of the weight of silk) are required for complete degumming. In practice, the silk is treated for 1 hr. with concentrated soap (2% of silk) solution at 100° (silk, silk-cotton) or 50° (silk-cellulose acetate, silk-viscose); the enzyme (750 g. of pancreatin per 100 kg. of fabric) is dissolved in a buffer solution of p_H 8 which is kept at 37°. The goods are treated for 12 hrs. with a pancreatin solution which has been used once, centrifuged, rinsed for 1 hr. at 100° or 50° with 1% soap solution and then with water, and centrifuged again. Pancreatin acts on wool at p_H 8.

CHEMICAL ABSTRACTS.

Rapid determination of raw fibre. U. H. PURANEN and E. S. TOMULA (Suomen Kem., 1930, 3, 85–89).—The finely-divided material (3 g.) is heated under reflux for 30 min. in a 500-c.c. flask with 50 c.c. of 5% sulphuric acid and 150 c.c. of water. After cooling, 20 c.c. of 28% potassium hydroxide solution are added and the heating is continued for a further 30 min. The mixture is filtered through asbestos and washed successively with hot water, 1.25% sulphuric acid, hot water, and alcohol-ether mixture; acetone may replace the alcohol-ether. The residue is dried to constant weight and ignited. Results are returned as ashless fibre.

T. T. POTTS.

Method of obtaining cross-sectional photo-[micro]graphs of textiles. P. V. PERROTT (Ind. Chem., 1930, 6, 325–326, 365–366).—Threads of yarn or untwisted filaments are stretched on wire frames, stained, and embedded in beeswax or in a collodion mixture. Sections are cut with a rocking type of microtome, and mounted in Canada balsam. The photographic apparatus consists of a light-tight box divided by a partition. One half of the box contains the microscope and illuminant. The eyepiece of the microscope projects through the partition into the other half of the box, a right-angled prism serving to project an image of the object on to the floor of the box, upon which first a white card is laid for focussing, and then a photographic plate for the exposure. Using a $\frac{1}{8}$ -in. objective and a 5 \times eyepiece, a magnification of 300 diam. is obtained.

T. T. POTTS.

Utilisation of kaoliang stalk. III. Industrial experiments on the manufacture of soda-pulp and paper. R. YAKAMOTO (Rept. Lab. S. Manchuria Rly., 1929, 26–27. Cf. B., 1928, 443).—In the manufacture of bleached pulp from kaoliang stalk the consumption of caustic soda and bleaching powder is 9% and 7% respectively; giving a yield of 37% of pulp, on the stalk used. It is considered that the requirements could be reduced to 8% and 5% respectively. Good-quality writing paper has been produced, using a mixture of 70% of bleached kaoliang pulp with 30% of sulphite-wood or Manila-hemp pulp. Unbleached kaoliang pulp gives a medium-grade “burning paper.”

H. J. DOWDEN.

Composition of *Diospyros ebenum*, Coen. Y. SHINODA (J. Cellulose Inst., Tokyo, 1930, 6, 205–206).—The following percentage results have been obtained: water content of the air-dry wood meal 10.5, alcohol-benzene extract of water-free meal 15.1, and, of the fat-free material, extract in 0.2% sodium hydroxide 4.5,

lignin content (König) 36.8, cellulose (Cross and Bevan) 36.9, pentosan content 13.8, purity of cellulose 98.5%. Xylose, mannose, and galactose were identified.

B. P. RIDGE.

Characterisation of technical celluloses by the iodine value. M. BERGMANN and H. MACHEMER (Ber., 1930, 63, [B], 2304–2306; cf. A., 1930, 457).—The finely-powdered, air-dried specimen of known water content is brought into a glass mortar and ground with 200 c.c. of 0.25*N*-sodium hydroxide in the presence of hardened porcelain balls until uniform dispersion is attained. After addition of 50 c.c. of 0.1*N*-iodine solution, the grinding is continued during 4 hrs., after which 10 c.c. of chloroform and 10 c.c. of 5*N*-sulphuric acid are added and the excess of iodine is titrated by sodium thiosulphate with starch as indicator. Addition of chloroform ensures the removal of iodine adsorbed by the cellulose. The results are accurate within about $\pm 3\%$. Prolongation of the oxidation does not lead to higher iodine values, whereas it appears possible that a period of 90 min. may be sufficient in technical practice. The iodine value of unbleached cotton is diminished by bleaching. Over-mercerised cotton has no greater iodine value than good technical cellulose, whereas marked increase in the number of aldehydic groups is observed when sulphite-cellulose is subjected to protracted treatment with 5*N*-sodium hydroxide. Technical cellulose for artificial silk is advantageously distinguished by its iodine value, 1.2, from celluloses with higher iodine value used for other purposes, which differ considerably among themselves. Samples of cellulose acetate with differing viscosity have almost identical iodine values.

H. WREN.

Electrolysis of a colloidal solution of hemicellulose. E. LENOBLE (Russa, 1929, 4, 1839, 1841).—Electrolysis of a waste caustic liquor from a viscose plant, employing an asbestos diaphragm, afforded pure hydrogen at the cathode; when sodium carbonate (from carbon dioxide produced by anodic oxidation of dissolved cellulose) crystallises out of the anode liquor unoxidised hemicellulose also begins to precipitate.

CHEMICAL ABSTRACTS.

Beating of [paper] pulp. XIII. Influence of water of imbibition on the physical properties of the paper. M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 183–191; cf. B., 1930, 899).—The effect of water of imbibition, which causes W_a , on the physical properties of papers has been studied by comparing sheets made from W -slate stuffs with those from W_m -slate stuffs. The differences observed are due to W_a , according to the equation $W - W_m = W_a$. Such water increases the bursting strength, transparency, and degree of self-sizing, and decreases the absorbency and thickness of the paper sheets.

B. P. RIDGE.

Application of the de Vains chlorination process to the pulping of birch wood. M. H. KANG and C. E. LIBBY (Cellulose, 1930, 1, 25–29, 50–51, 54–58).

Celotex. LATHROP.—See XVII.

PATENTS.

Treatment of vegetable fibres which are used, in the purification by dialysis of caustic soda and like solutions, as osmotic diaphragms. L. CERINI

(B.P. 318,963, 27.8.29. It., 13.9.28).—Cotton or other vegetable fabric, in the natural state or preheated with concentrated alkalis, is treated with a magnesium salt solution, dried, and further treated with an alkali resinate solution which forms with the salt an organic colloidal composition on the fabric. F. R. ENNOS.

[Production of synthetic board from] fibrous product. T. B. MUNROE and E. C. LATHROP (B.P. 335,052, 14.9.29).—Vegetable fibre, particularly sugar-cane bagasse, is subjected to a controlled fermentation while stacked in bales, digested with water under pressure to remove cementing material, refined in a beater, mixed with 5–20% of paper fibre or close stock, and formed into a sheet containing about 50% of water. The wet sheet is finally dried at 175° and under 500 lb./in.² in a press. F. R. ENNOS.

Manufacture of [fibrous] friction- and heat-resisting articles. W. NANFELDT, Assr. to WORLD BESTOS CORP. (U.S.P. 1,758,055, 13.5.30. Appl., 6.11.25).—Asbestos material is pulped and mixed with (i) a binder, *e.g.*, resin, which is disseminated through the material by precipitation of a soluble resin soap with alum; (ii) a lubricating material, *e.g.*, graphite; and (iii) a material to prevent undue hardening of the binder, *e.g.*, graphite or glycerin. The product in sheet form is suitable for brake linings etc. F. R. ENNOS.

Treatment of [knitted] fabric. BRIT. CELANESE, LTD. (B.P. 335,139, 13.1.30. U.S., 12.1.29).—Knitted fabrics containing yarns of cellulose acetate (*etc.*) are prevented from “laddering,” “running,” or “splitting” by treatment with a shrinking agent, *e.g.*, nitric acid (*d* 1·19) for 1 min. at ordinary temperature. F. R. ENNOS.

Caustic soda solution of cellulose. O. LEUCHS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,755,657, 22.4.30. Appl., 18.7.27. Ger., 30.7.26).—After mild pretreatment with organic or inorganic acids at suitable temperatures and concentrations to render it soluble, cellulose is dissolved in 10% caustic alkali, from which it is reprecipitable in the form of films, threads, etc. F. R. ENNOS.

Manufacture of artificial threads, bands, films, etc. from viscose solutions. ZELLSTOFFFABR. WALDHOF, and A. BERNSTEIN (B.P. 315,405, 27.6.29. Ger., 13.7.28).—The filaments are first coagulated in a salt solution and afterwards fixed in an acid solution, the coagulating and fixing solutions being contained in two different compartments of the same vessel; the filaments are passed from one liquid to the other over the dividing partition, which does not reach to the top of the liquids. F. R. ENNOS.

Production of artificial threads, filaments, ribbons, films, etc. from cellulose derivatives. BRIT. CELANESE, LTD., R. P. ROBERTS, R. I. R. DEAN, and L. W. GREGORY (B.P. 334,838, 9.5.29).—Organic derivatives of cellulose are wet-spun into (a) strong solutions of mineral acids (30–50% aqueous sulphuric acid) or solutions of their acid esters, or (b) a precipitating bath, *e.g.*, diacetone alcohol, followed by subsequent treatment in the acid bath, and are finally washed. F. R. ENNOS.

Cementing together lacquered cellulose foils which swell in water. WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 316,574, 3.7.29. Ger., 31.7.28).—The foils (*e.g.*, of viscose) are cemented with their lacquered surfaces (*e.g.*, of cellulose acetate) juxtaposed by means of a mixture of an adhesive both for the coating and for the cellulose foils (an aqueous solution of gelatin and glycerin) and of a solvent for the lacquer coating (acetone). F. R. ENNOS.

Recovery of volatile plasticisers from celluloid and the like. H. P. BASSETT (U.S.P. 1,756,331, 29.4.30. Appl., 10.2.27).—Celluloid is immersed in boiling sodium acetate solution, and steam is passed through to volatilise the plasticiser, *e.g.*, camphor, without injuring the nitrocellulose. F. R. ENNOS.

Manufacture of paper pulp. A. M. THOMSEN (U.S.P. 1,758,896, 13.5.30. Appl., 1.8.27).—Vegetable fibre is freed from encrusting materials by cooking with alkali sulphide alone at atmospheric pressure; the product after being washed is bleached by treatment with alkali manganate and carbon dioxide. F. R. ENNOS.

Manufacture of a pulp product [from tobacco waste]. R. B. ARNOLD and R. M. HIXON, Assrs. to TOBACCO BY-PRODUCTS & CHEM. CORP. (U.S.P. 1,756,722, 29.4.30. Appl., 12.11.27).—Tobacco waste which has been treated for the extraction of nicotine and other water-soluble constituents is mechanically shredded and the moist mass of matted strips is dried and beaten in water with a smaller proportion of other cellulosic materials, *e.g.*, old burlap, to form a pulp ready for sheeting etc. F. R. ENNOS.

Continuous sulphate process [for pulping of wood]. W. C. MUNRO, Assr. to W. D. MOUNT (U.S.P. 1,756,432, 29.4.30. Appl., 13.4.27. Can., 1.3.27).—Spent liquor from the digestion of wood by the sulphate process is evaporated and smelted, and the product is dissolved to yield “green liquor,” which is freed from impurities by continuous decantation of the sludge and filtration. The purified liquor is causticised with lime, and after decantation is re-used for digesting more wood, whilst the separated lime sludge is calcined for re-use in causticising. F. R. ENNOS.

Manufacture of [wood] pulp and treatment of residual liquors. L. BRADLEY and E. P. McKEEFE, Assrs. to BRADLEY-McKEEFE CORP. (U.S.P. 1,743,080, 7.1.30. Appl., 22.3.26).—Residual liquor from the cooking of wood with caustic soda and a sodium-sulphur compound is concentrated, mixed with calcium carbonate, dried, and heated in a reducing atmosphere at a temperature sufficient to convert the calcium carbonate into oxide and the sodium compounds into carbonate and sulphide; the products of destructive distillation of the organic matter are collected and the waste gases are used to dry other portions of residual liquor. The heated mass when mixed with warm water yields a solution containing caustic soda and sodium sulphide for use again in the pulping process. F. R. ENNOS.

Utilisation of spent lime cooking liquors in the manufacture of paper. A. L. CLAPP (U.S.P. 1,758,247, 13.5.30. Appl., 14.2.27).—Cellulose fibrous material

which has been digested in lime liquor is beaten in the spent liquor, run off on to a paper machine, and the dried paper is treated with sodium silicate solution.

F. R. ENNOS.

Manufacture of coloured crêpe paper with insoluble dyes. G. R. ALDEN, Assr. to DENNISON MANUFACTURING CO. (U.S.P. 1,756,778, 29.4.30. Appl., 12.11.26).—After treatment with a dye solution and removal of any excess, paper in the form of sheets is crêped, treated with a dye-fixing reagent which does not destroy the crêpe and effects fireproofing (concentrated aqueous solutions of aluminium and ammonium sulphates, or an alcoholic solution of Katanol-O and sodium tungstate), and is finally dried.

F. R. ENNOS.

Sizing of paper and compositions therefor. BECKER & CO. (1924), LTD. From B. WIEGER (B.P. 335,902, 1.7.29).—The pulp is mixed with a sizing agent consisting of an aqueous dispersion formed by the gradual addition of a small proportion of a protective colloid, e.g., casein in alkaline solution, to melted and well mixed natural or artificial resins, waxes, solid or semi-solid hydrocarbons, fats, fatty acids or their mixtures, with or without drying oils, the dispersion being afterwards coagulated by addition of alum. F. R. ENNOS.

Preparing hair for felting. H. WAGNER, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,778,473, 14.10.30. Appl., 26.3.26. Ger., 9.4.25).—See B.P. 250,575; B., 1927, 599.

Felting property of animal hair. R. HAYNN, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,779,363, 21.10.30. Appl., 2.1.29. Ger., 9.1.28).—See B.P. 323,364; B., 1930, 236.

Apparatus for manufacture of artificial filaments, threads, etc. H. J. HEGAN and E. HAZELEY, Assrs. to COURTAULDS, LTD. (U.S.P. 1,779,313, 21.10.30. Appl., 25.2.28. U.K., 21.4.27).—See B.P. 294,279; B., 1928, 810.

Manufacture of artificial silk, and apparatus therefor. F. W. SCHUBERT, Assr. to BRYLSILKA, LTD. (U.S.P. 1,778,301—2, 14.10.30. Appl., [A] 19.5.28, [B] 18.7.28. U.K., [A] 15.7.27, [B] 4.8.25).—See B.P. 296,856 and 262,369; B., 1928, 852; 1927, 126.

Recovery of fibre from the leaves of fibrous plants. F. KRUPP GRUSONWERK A.-G. (B.P. 335,114, 15.11.29. Ger., 24.9.29).

Evaporating plant (B.P. 334,623).—See I. Solvents for cellulose ester (B.P. 314,087).—See XIII. Mulches (B.P. 319,783).—See XVI. Wood sugar (B.P. 315,198). Fermentable sugars from cellulose (B.P. 315,403). Sealing-wax substitute (B.P. 334,936).—See XVII. Aliphatic acids by fermentation (B.P. 334,900).—See XVIII.

VI.—BLEACHING ; DYEING ; PRINTING ; FINISHING.

Bleaching tussah silk. J. CLARON (Russia, 1930, 4, 1531).—A method involving treatment with sodium perborate, followed by hydrogen peroxide and sodium silicate, and finally treatment with sodium hyposulphite, is described.

CHEMICAL ABSTRACTS.

Kaoliang starch as finishing material for textiles. J. KAWAMATA (Rep. Lab. S. Manchuria Rly., 1929,

27—29).—From a consideration of the viscosity, permeability into cotton cloth, adsorption of dyes, tensile strength, and elongation of the yarns, and the feel and lustre of the cloth, the opinion is formed that the starch extracted from kaoliang (*Andropogon sorghum*, Broth.) is intermediate in quality between wheat and maize starches.

H. J. DOWDEN.

Enzymatic degradation of cellulose. KARRER.—See V.

PATENTS.

Bleaching methods and compositions [peroxides of higher fatty acids]. R. E. ELLIS. From PILOT LAB., INC. (B.P. 334,531, 27.5.29).—Acid chlorides, such as lauryl chloride or the chloride of any acid occurring in natural fats, oils, or waxes, are treated with aqueous alkali and hydrogen peroxide, preferably in presence of soap solution, at 0°. Stable peroxides are obtained, and these, dissolved in high-boiling petroleum, form efficient bleaching agents for flour etc.

C. HOLLINS.

Method of dyeing. R. R. SLEEPER, Assr. to CALCO CHEM. CO., INC. (U.S.P. 1,758,877, 13.5.30. Appl., 30.8.28).—In dyeing, cheap crude sodium bisulphate ("nitre-cake") is used as a substitute for a mixture of sodium sulphate and sulphuric acid; further, a small amount of a reducing agent, particularly oxalic acid, is added in order to avoid discoloration of certain dyes by impurities in the nitre-cake.

A. J. HALL.

Treatment of fibrous materials. SOC. CHEM. IND. IN BASLE (B.P. 317,019, 8.8.29. Switz., 8.8.28).—Dry cellulosic fibres, particularly cotton, are treated with an organic carbimide, especially phenylcarbimide, in the presence of a catalyst (e.g., zinc chloride or pyridine) and a diluent at ordinary or raised temperatures, the reaction being arrested before their fibrous structure is lost (cf. B.P. 130,277; B., 1918, 714 A). After being freed from excess of the reagents by distillation *in vacuo* and washing with hot alcohol, the treated fibres have increased weight (up to 30%), a nitrogen content of about 2.5%, a resistance to direct dyes, and an affinity for cellulose acetate dyes. Wool similarly treated loses its affinity for acid dyes.

A. J. HALL.

[Production of effects on] textile materials. BRIT. CELANESE, LTD. (B.P. 314,396, 26.6.29. U.S., 26.6.28. Addn. to B.P. 274,841; B., 1928, 782).—Fabrics composed of organic derivatives of cellulose are delustrated by the action of wet steam, then treated uniformly or locally with a paste containing a water-insoluble thickening agent (cellulose nitrate), an effect material (zinc oxide, barium sulphate, together with a dye and a plasticiser, if desired), and a solvent for the thickening agent which is a non-solvent for the cellulose derivative (ethyl alcohol and ether), and are finally relustrated, e.g., with acetic acid or calcium thiocyanate.

F. R. ENNOS.

Production of ornamental effects on fabrics. BRIT. CELANESE, LTD. (B.P. 335,138, 13.1.30. U.S., 12.1.29).—"Seersucker" (crinkled or puckered) fabric consisting of cellulose ether or ester silk is produced by printing or spraying woven or knitted materials with a solution of nitric acid (d 1.05—1.30), allowing it to react for 1—10 min. at 0—60° and then washing;

shrinkage of the treated parts causes puckering of the non-treated parts of the fabric (cf. B.P. 274,584; B., 1927, 747).

A. J. HALL.

Treatment of materials made of or containing cellulose esters. BRIT. CELANESE, LTD. (B.P. 313,404, 10.6.29. U.S., 8.6.28).—Cellulose acetate silk is hydrolysed with an alkali or base in the presence of alcohols other than methyl and ethyl alcohols (cf. B.P. 125,153; B., 1918, 427 A), particularly glycol and other polyhydric alcohols. By such means it is possible to hydrolyse 95% of the ester content of the cellulose acetate, using an amount of caustic soda theoretically sufficient to hydrolyse but 3% of the ester. Alkali ethoxides are preferred as hydrolysing agents.

A. J. HALL.

Mercerisation [of materials containing artificial silks]. H. P. BASSETT and T. F. BANIGAN, ASSRS. to MEIGS, BASSETT, & SLAUGHTER, INC. (U.S.P. 1,758,553, 13.5.30. Appl., 30.1.25).—Cotton or other material containing a cellulose or cellulose ester silk is mercerised without deleterious action on the silk by impregnating with a saturated solution of sodium acetate, then (while wet) treating with the mercerising liquor, and finally removing the alkali by washing with saturated sodium acetate solution.

A. J. HALL.

Treatment [delustring] of films, spun threads, and fabrics made of or containing cellulose esters or ethers. SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 313,072, 28.3.29. Fr., 6.6.28).—The material is treated at 40—80° (higher temperatures are unnecessary) in an aqueous emulsion of organic substances which swell or dissolve it, but which are substantially insoluble in water. Such substances include cyclohexanone, cyclohexanol, di- and tri-acetins, methyl phthalate, amyl acetate, ethyl benzoate, anisole, and nitrobenzene; the concentration should not exceed 5%.

A. J. HALL.

Manufacture [and application] of a moth-proofing material. W. SCHEPSS, E. TIETZE, and A. OSSENBECK, ASSRS. to I. G. FARBEIND. A.-G. (U.S.P. 1,757,222, 6.5.30. Appl., 20.12.28. Ger., 28.12.27).—Animal fibres are exposed to the vapours of boron trifluoride or treated with 1% solutions in organic solvents of boron trifluoride or its addition compounds with organic substances containing carbonyl groups, such as acetophenone, benzaldehyde, acetone, acetoacetic ester, esters of phthalic acid.

A. J. HALL.

Manufacture of [crêped] fabrics of or comprising artificial silk. G. B. ELLIS. From SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 335,020, 14.8.29).—Fabrics containing twisted cellulose acetate threads, not necessarily sized, are treated in a bath, e.g., of alcohols or benzene, having a strong swelling action but little or no peptising effect, the latter being ascertained by the permanent shrinkage after treatment of the untwisted threads which, after drying, should not be greater than 8% of their initial length.

F. R. ENNOS.

Treatment of textile goods for identification. K., O., and E. HAASE (T. HAASE) (B.P. 308,815, 27.3.29. Ger., 31.3.28).—The material, either in the raw state or at some stage in its manufacture, is impreg-

nated with salts of such metals as cobalt, nickel, barium, etc. which do not normally occur in the treating liquids used for the particular material and which can be detected chemically or spectroscopically.

F. R. ENNOS.

Dyeing of pelts, hairs, feathers, and the like. P. VIRCK, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,778,819, 21.10.30. Appl., 6.12.27. Ger., 14.9.26).—See B.P. 290,126; B., 1928, 521.

Treatment of threads or fabrics containing cellulose acetate. G. LARDY, ASSR. to DU PONT RAYON Co. (U.S.P. 1,778,327, 14.10.30. Appl., 6.6.28. U.K., 23.6.27).—See B.P. 301,355; B., 1929, 126.

Mechanically moving skeins of yarn during treatment with liquids. J. SCHLUMPF (B.P. 316,227, 24.7.29. Ger., 25.7.28).

Treatment [polishing] of yarn. A. H. JUNKERS (B.P. 319,793, 28.9.29. Ger., 29.9.28).

Washing and boiling machines [for laundries etc.]. A. and (MRS.) K. M. I. RASMUSSEN (B.P. 336,363, 27.8.29).

[Tumbler] drying apparatus [for fabrics]. E. A. ALLIOTT, and MANLOVE, ALLIOTT & Co., LTD. (B.P. 336,357, 22.8.29).

Coloured crêpe paper (U.S.P. 1,756,778).—See V. Alkaline oxidant and its storage (U.S.P. 1,765,344). **Alkali hypochlorites** (B.P. 334,364 and U.S.P. 1,765,013).—See VII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Purification of commercial hydrochloric acid. V. E. TISHCHENKO and V. M. PERMYAKOVA (J. Appl. Chem., Russia, 1930, 3, 255—259).—After purification with stannous chloride (which does not react instantaneously with the arsenic) and washing with hot zinc sulphate solution, the acid is free from arsenic, tin, and iron.

CHEMICAL ABSTRACTS.

Vapour pressures of carbon dioxide and ammonia in the ammonia-soda process. K. N. SHABALIN and V. S. UDINTZEVA (J. Appl. Chem., Russia, 1930, 3, 165—190).—Vapour pressures of solutions of carbon dioxide and ammonia in sodium chloride solution were determined by a static method.

CHEMICAL ABSTRACTS.

Preparation of the salt-ammonia solution and its treatment in the manufacture of soda by the Solvay process. N. F. YUSHKEVICH, A. V. AVDEEV, J. N. SHOKIN, V. S. OLENEY, and D. Y. ITKIN (J. Chem. Ind., Russia, 1929, 6, 1483—1497, 1570—1585).—The influence of concentration of sodium chloride, temperature, partial pressure of ammonia, and concentration of carbon dioxide in the solution and the gaseous phase on the absorption of ammonia by sodium chloride solution has been studied. The pressure of carbon dioxide in the gaseous phase favours the absorption of ammonia by the brine. Increase in the carbon dioxide content of the solution causes a considerable fall in the partial pressure of ammonia and a slight fall

in the partial pressure of water vapour. The introduction of increased quantities of carbon dioxide into the absorber is recommended. The influence of rise of temperature (30–70°) on the speed of absorption of carbon dioxide by ammoniacal sodium chloride solution is small.

CHEMICAL ABSTRACTS.

Solvay soda process. N. F. YUSHKEVICH (J. Chem. Ind., Russia, 1929, 6, 1087–1098).—The application of the process to a brine containing much calcium and magnesium and very small quantities of iron and aluminium is described.

CHEMICAL ABSTRACTS.

Causticising ammonium chloride with magnesium oxide. K. NISHIDA and S. NAKAMURA (Rep. Lab. S. Manchuria Rly., 1929, 37–39).—An attempt to utilise light-burnt magnesite in the place of lime for causticising ammonium chloride in the Solvay process has shown that the former is weaker than the latter and that for 100% recovery of ammonia the ammonia liquors must be diluted from 3.46*N* to 2.34*N*. From the rate of distillation it is calculated that the amount of fuel required in the magnesite method is thrice that for the lime, and hence the former method is not economical unless the value of the recovered magnesium chloride is sufficient to cover the cost of the fuel.

H. J. DOWDEN.

Manufacture of ammonium sulphate from phosphogypsum. S. I. VOLFKOVICH, V. P. KAMZOLKIN, and A. SOKOLOVSKI (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 64, 5–61).—By treating the gypsum, obtained from a low-grade phosphate rock, with ammonia and carbon dioxide (5–10% excess) at 40–45°, 92–97% of the sulphuric acid was regenerated, ammonium sulphate being obtained in 30% solution.

CHEMICAL ABSTRACTS.

Utilisation of bittern. I. Recovery of Glauber's salt. T. KATO (Rep. Lab. S. Manchuria Rly., 1929, 40–41).—The maximum recovery of Glauber's salt with least contamination by impurities is achieved when to 100 pts. of bittern are added 40 pts. of sodium chloride or 100 pts. of saturated brine, followed by dilution to *d* 1.20. The yield of sodium sulphate (based on the SO₃ content of the bittern) is 45% at –10° and 70% at –19°. The impurities (sodium chloride and magnesium sulphate) increase slowly with increasing concentration of the solution, but rapidly above *d* 1.23. The impurity content is almost independent of the degree of cooling applied.

H. J. DOWDEN.

Volumetric determination of active oxygen in mixtures of hydrogen peroxide and persulphuric acid. G. O. LUBARSKY and M. G. DIKOVA (Z. anal. Chem., 1930, 81, 450–454).—Most of the methods usually employed give low results, but accurate results may be obtained by the iodometric method provided that the solution is set aside in a dark place for 24 hrs. prior to titration of the liberated iodine.

A. R. POWELL.

Analysis of red phosphorus. S. A. TOLKATSCHOV and M. A. PORTNOV (Z. anal. Chem. 1930, 82, 122–133).—In the determination of the total phosphorus in red phosphorus by oxidation to phosphoric acid with nitric acid, oxidation is never complete, the product always

containing lower acids of phosphorus. If, however, the phosphorus is warmed with a saturated solution of bromine in nitric acid of *d* 1.4 instead, it is soon completely oxidised to phosphoric acid, which may then be determined in the usual way. To determine the amount of combined phosphorus originally present, the phosphorus is treated with 2*N*-sulphuric acid and the extract oxidised with bromine and nitric acid; extraction with water alone fails to remove the whole of the combined phosphorus. The amount of white phosphorus present may be found by adding a slight excess of bromine to the carbon disulphide extract, then evaporating off the solvent, and oxidising to phosphoric acid with bromine and nitric acid.

R. CUTHILL.

Determination of silica in silicates. TANANAEV and BABKO.—See VIII.

PATENTS.

Generation of hydrochloric acid gas. G. P. ADAMSON, Assr. to GEN. CHEM. CO. (U.S.P. 1,764,593, 17.6.30. Appl., 5.2.27).—Sulphuric acid of 93–98% concentration is introduced at the top of a packed tower, and hydrochloric acid solution (*d* 1.16–1.18) continuously fed in at the centre. The mixing of the two acids in the lowest section generates sufficient heat to expel the hydrogen chloride, which becomes dehydrated in the upper part of the tower by the downwardly flowing sulphuric acid, and leaves the tower with a concentration of 100%. The sulphuric acid is only slightly diluted, the acid leaving the bottom of the tower having *d* not below 1.71.

W. J. WRIGHT.

Manufacture of ammonia [from hydrogen and nitrogen]. Preparation of ammonia-factor gases. F. H. SNYDER and E. D. NEWKIRK (U.S.P. 1,760,013–4, 27.5.30. Appl., [A] 21.2.27, [B] 22.7.27).—(A) Catalyst poisons from the reacting gases are temporarily removed before the latter enter the converter. The gas mixture from the compressor, after passing through a deoxidiser, is conveyed to a brine-cooled coil, from which it is bubbled into a separator containing liquefied ammonia, and passes on to the converter. The ammonia formed here is liquefied and, together with uncombined gases, mixes with the incoming gases passing through the coil. A constant level of liquefied ammonia is maintained in the separator. (B) A mixture of steam and air, in proportions to give a mixture of hydrogen and nitrogen for ammonia synthesis, after preheating in a heat exchanger, passes down through the air chamber of a furnace into a generator, filled with nodules of sponge iron and maintained at 500°. The iron oxide is continually removed from the bottom, and fresh sponge iron fed in at the top, so as to restrict the zone of oxidation. The hydrogen and nitrogen formed are conducted to the outer chamber of the heat exchanger, and thence to a water scrubber in which is placed a layer of sponge iron. The steam generated by the hot gases heats the sponge iron and causes it to react with impurities in the gases to give removable products; the cooled gases pass first to an ammonia water scrubber, where oxygen is liberated and carbon dioxide removed, and thence to a gas holder. In this the hydrogen-nitrogen mixture is compressed to about 4500 lb./in.² prior to passing to

an oil and moisture separator and finally to the converters.
W. J. WRIGHT.

Production of compressed mixtures comprising air and ammonia. H. PAULING (B.P. 334,448, 12.11.29. Ger., 7.1.29).—The mixture being corrosive, the air only is compressed and is afterwards passed in contact with a concentrated aqueous solution of ammonia. The flow is countercurrent, and when the solution is exhausted its pressure is let down in a reversed pump and it is then regenerated by ammonia gas at ordinary pressure; the pressure is then increased in a pump and the solution re-used. The letting down and increase of pressure may be effected on opposite sides of the same piston; since the work of compression of the ammonia gas has been avoided the solution will be cooled and must therefore be heated at some point of the cycle, preferably while in the high-pressure tower.
B. M. VENABLES.

Alkaline oxidant and [its storage in] aluminium metal [vessels]. A. PIETZSCH and G. ADOLPH, Assrs. to BUFFALO ELECTRO-CHEM. Co., INC. (U.S.P. 1,765,344, 17.6.30. Appl., 16.6.27. Ger., 3.9.26).—The corrosive action of alkalis on aluminium is avoided if a sufficient concentration of hydrogen peroxide is maintained in the solution, so that a protective skin is formed on the metal. In bleaching cotton by means of caustic soda and hydrogen peroxide, the waste liquor may be stored in the vessel without acting on it as long as oxygen is available.
W. J. WRIGHT.

Preparation of [solid or semi-solid compositions comprising] alkali hypochlorites. V. SZIDON (B.P. 334,364, 14.8.29).—The compositions are prepared by adding alkali salts of low-molecular fatty acids, *e.g.*, sodium octoate or dodecoate, and, if desired, sodium carbonate or silicate, to solutions of alkali hypochlorites as free as possible from chlorides and from salts of unsaturated fatty acids.
L. A. COLES.

Manufacture of [solid or semi-solid compositions comprising alkali] hypochlorite in colloidal form. P. R. HERSHMAN, Assr. to C. O. and C. H. SETHNESS and P. RUDNICK (U.S.P. 1,765,013, 17.6.30. Appl., 28.3.27).—A solution of a hypochlorite is mixed with a colloidal substance or with a substance capable of forming a colloidal precipitate, such as a silicate or a phosphate, and the mixture is evaporated to dryness.
W. J. WRIGHT.

Apparatus for dissolving sodium silicate. L. B. EDGERTON, Assr. to PHILADELPHIA QUARTZ Co. (U.S.P. 1,763,845, 17.6.30. Appl., 6.10.27).—A cylindrical tank with convex base has a screen near the bottom for supporting the silicate, access to this being obtainable by a lateral manhole. Projecting centrally upwards from the bottom of the tank is a standpipe, terminating near the top of the tank in a conical spreader. The lower part of the standpipe, from a point level with the screen, is provided with a series of slot-like apertures covered by a gauze screen. Below the tank the standpipe communicates with a steam injector which has a branch to a water pipe entering the bottom of the tank. A bottom discharge pipe and two lateral sampling tubes are provided.
W. J. WRIGHT.

Sodium borate-trisodium phosphate compound and its manufacture. C. F. BOOTH, A. B. GERBER, and P. LOGUE, Assrs. to FEDERAL PHOSPHORUS Co.

(U.S.P. 1,759,152, 20.5.30. Appl., 28.10.27).—In the production of neutral trisodium phosphate, as described in U.S.P. 1,688,112 (B., 1929, 53), addition of sodium borate improves the crystallisation of the trisodium phosphate, and results in the formation of the double compound, sodium borate-trisodium phosphate, which has advantages as a detergent and water-softening agent.
W. J. WRIGHT.

Purification of [removal of boric acid from] monopotassium phosphate. R. D. PIKE and R. CUMMINGS (U.S.P. 1,746,905, 11.2.30. Appl., 9.7.28).—Solutions containing monopotassium phosphate, prepared by the action of potassium carbonate on phosphoric acid, are treated with currents of air and (preferably superheated) steam to volatilise the boric acid, after which they are evaporated to dryness to yield fertilisers.
L. A. COLES.

Manufacture of alkali ethylates [ethoxides]. A. WACKER GES. F. ELEKTROCHEM. IND., G.M.B.H. (B.P. 334,388, 12.9.29. Ger., 24.11.28).—An alkali hydroxide is treated with ethyl alcohol at a temperature considerably above its normal b.p. and in the presence, if desired, of a non-reacting auxiliary liquid capable of forming azeotropic mixtures with the water, with alcohol, or with both. *E.g.*, alcohol vapour is passed at 200° into a suspension of sodium hydroxide in paraffin oil, or a mixture of the constituents is distilled under raised pressure.
L. A. COLES.

Stable effervescent composition. L. T. ANDREWS, Assr. to E. R. SQUIBB & SONS (U.S.P. 1,764,996, 17.6.30. Appl., 12.4.28).—Alkali dihydrogen citrate is used instead of citric acid in effervescent compositions to render these stable even when subjected to high temperature (up to 120°) and humidity.
W. J. WRIGHT.

Separation of soluble substances [*e.g.*, salts]. IMPERIAL CHEM. INDUSTRIES, LTD., and H. E. COCKS-EDGE (B.P. 334,169, 14.3.29).—Mixtures of crystals obtained by the evaporation of a common solution are separated by grading them first according to density and then to size, or *vice versa*. The particle size may, if desired, be controlled during crystallisation, *e.g.*, by the addition of colloids, by seeding, or by the addition of supersaturated solutions.
L. A. COLES.

Catalysts and catalytic processes. W. A. LAZIER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,746,781—3, 11.2.30. Appl., [A] 8.4.26, [B] 12.6.26, [C] 3.11.28).—Catalysts for use in processes such as the synthesis of methyl alcohol are prepared (A) by the decomposition at 200—600° of oxalates or mixtures of oxalates which on heating yield metals, oxides, or carbonates; (B) by calcining chromates at 650—1000° to yield chromites containing trivalent chromium; and (C) by heating exothermic multiple salts of hydrogenating metals and nitrogenous bases. Examples include the preparation of catalysts in (A) from zinc, copper, and chromium oxalates; in (B) from basic zinc chromate, the product being washed with 10% acetic acid to remove free zinc oxide; and in (C) from manganese and basic zinc ammonium chromates and from zinc dichromate tetrapyridine.
L. A. COLES.

Manufacture of cuprous compounds. S. B. HEATH and M. O. KELLER, Assrs. to DOW CHEM. Co.

(U.S.P. 1,763,781, 17.6.30. Appl., 4.2.28).—If chlorine is bubbled through a solution containing cupric, cuprous, and ammonium chlorides in presence of a large surface of copper, the ammonium salt is not decomposed, but the chlorine is absorbed more rapidly than if the ammonium salt were absent. The copper, on account of its large surface, quickly reduces the cupric salts formed and accelerates the introduction of the chlorine, which reacts with the copper to form more cupric chloride for cyclic reduction. During the reaction the temperature of the solution soon reaches the b.p. When the solution is sufficiently concentrated, addition of chlorine is discontinued, and the hot solution is allowed to remain in contact with metallic copper until 98% of the metal is converted into the cuprous state.

W. J. WRIGHT.

Concentration of nitrous gases. J. A. WYLER, Assr. to TROJAN POWDER Co. (U.S.P. 1,765,254, 17.6.30. Appl., 8.12.27).—Nitrogen oxides are recovered from waste gases in which they are present in a diluted form by passing the gases through a solution of an alkali salt, *e.g.*, sodium chromate, molybdate, tungstate, or arsenate. After evaporating the solution, the residue is heated to drive off the nitrogen oxides in a concentrated form and to recover the alkali salt.

W. J. WRIGHT.

Extraction of sulphur [from its ore]. A. J. CROWLEY, Assr. to HUMBOLDT SULPHUR Co. (U.S.P. 1,763,762, 17.6.30. Appl., 6.7.27).—The method is based on the fact that in presence of superheated steam molten sulphur will support the residue, resulting from the extraction of sulphur from ore, to a depth of about 4 in. above its surface, dependent on the relative sp. gr. Suitable apparatus is described.

W. J. WRIGHT.

Preparation of colloidal sulphur. H. HOWARD, Assr. to the GRASSELLI CHEM. Co. (U.S.P. 1,765,307, 17.6.30. Appl., 17.11.26).—Calcium sulphide in the solid form, or lime-sulphur as an aqueous solution, is added to an aqueous solution of sulphur dioxide, an excess of the latter being maintained till the reaction is complete.

W. J. WRIGHT.

Production of alumina. E. C. MARBURG, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,778,083, 14.10.30. Appl., 20.10.25. Ger., 2.10.24).—See B.P. 240,834; B., 1926, 12.

Purification of zinc solutions. R. H. STEVENS, G. C. NORRIS, and W. N. WATSON, Assrs. to RHODESIA BROKEN HILL DEVELOPMENT Co., LTD. (U.S.P. 1,778,987, 21.10.30. Appl., 25.8.27. S. Rhod., 13.8.26).—See B.P. 278,851; B., 1927, 943.

[Ebonite] valves for oxygen containers or bottles. GAS ACCUMULATOR Co. (UNITED KINGDOM), LTD., Assees. of SVENSKA AKTIEB. GAS-ACCUMULATOR (B.P. 336,535, 17.2.30. Swed., 16.2.29).

Fibres for dialysis of caustic soda (B.P. 318,963). **Pulping of wood** (U.S.P. 1,756,432). **Residual liquors from wood-pulp manufacture** (U.S.P. 1,743,080).—See V. **Nickel-iron-silicon alloy** (U.S.P. 1,759,477). **Treatment of arsenious ore** (U.S.P. 1,763,435).—See X. **Electrolysis of water** (B.P. 334,991).—See XI. **Fertilisers** (B.P. 335,600).—See XVI.

VIII.—GLASS; CERAMICS.

Woodall-Duckham continuous circular tunnel kiln. A. N. TARRANT (Trans. Ceram. Soc., 1930, 29, 280—289).—The kiln is about 100 ft. overall diam., has a tunnel 260 ft. in length, and the tray-setting space is 7 ft. high \times 6 ft. wide. As an open-fired kiln for saggars or bricks the setting capacity is 10,920 cub. ft. per cycle, and as a muffle kiln for firing open-set glost materials the capacity is 7980 cub. ft. Producer gas is used for firing, the fuel consumption being one quarter of the amount required for a similar output with intermittent muffle kilns.

R. J. CARTLIDGE.

Influence of iron borate on rate of inversion of quartz in silica bricks. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1930, 29, 299—303).—Three batches of bricks were fired to cone 17 in a commercial silica-brick kiln. Photomicrographs and expansion curves show that bricks with 1.26% Fe_2O_3 and 0.74% B_2O_3 contain more tridymite than those containing 0.87% Fe_2O_3 and 1.13% B_2O_3 , and that bricks with 2.0% Fe_2O_3 contain a still larger proportion of tridymite.

R. J. CARTLIDGE.

Comparison between artificial and natural solarisation and stabilisation of special commercial ultra-violet-transmitting glasses. D. C. STOCKBARGER and L. B. JOHNSON (J. Franklin Inst., 1930, 210, 455—459).—Samples of 47 commercial varieties of such glasses were solarised by exposure (a) for 3 months in the open air; (b) for 200 hrs. at 18 in. from a Uviarc with a thin sheet of "neutral glass with transmission terminating at about 3000 Å." placed close to the sample; and (c) to the Uviarc direct. The percentage transmission was measured by a spectroradiometer with thermopile, and that for a uniform thickness of glass of 2 mm. thence calculated. The results show that (c) was invariably much too severe, whilst results with (a) and (b) agreed substantially, transmission after (b) as compared with that after (a) being slightly greater in 26 cases, smaller in 6, and identical in 14. The effect of sunlight on glass solarised by the bare arc was tested in a few cases, with results approximating towards those of Wood and Leathwood (*cf.* B., 1929, 897), though transmission was always less than after exposure to sunlight alone. Transmission after (a) varied from 31 to 64% in different samples.

C. A. SILBERRAD.

Testing safety glass. H. D. MURRAY and D. A. SPENCER (Ind. Chem., 1930, 6, 399—401).—The following tests are desirable: (1) A fracturing test in which blows are struck by a steel striker descending between guides from increasing heights until fracture occurs; (2) the glass is penetrated by the striker and the broken sample examined, all splinters above a limit being weighed; (3) a crushing test; (4) a test for sealing of the edge in a water-bath. The colour should also be determined after heating at 100—105° for 24 hrs. and after exposure to a carbon arc, while at 60°.

C. IRWIN.

Volumetric determination of silicic acid in silicates. N. A. TANANAEV and A. K. BABKO (Z. anal. Chem., 1930, 82, 145—150).—A method for the volumetric determination of silica in silicates, depending on

the titration of potassium fluosilicate with sodium hydroxide in presence of calcium chloride (cf. Schuch and Möller, B., 1906, 1176), is described. The silicate is opened up by fusion with a considerable excess of potassium carbonate, and then by adding ammonium fluoride and an excess of hydrochloric acid the silica is precipitated as potassium fluosilicate. Excess of acid is required in order to prevent the simultaneous precipitation of aluminium in a form which would interfere with the subsequent titration. The precipitate is then washed with a saturated solution of potassium fluosilicate, and finally titrated hot with 0.5*N*-sodium hydroxide, using methyl-red as indicator. The attainment of the end-point is also indicated by the precipitate coagulating. A blank determination must be made with the reagents used, omitting the silicate, and a corresponding correction applied. R. CUTHILL.

Fire clays. II. Some fundamental properties at several temperatures. R. A. HEINDL and W. L. PENDERGAST (J. Amer. Ceram. Soc., 1930, 13, 725—750; cf. B., 1929, 979).—Extensive tests were carried out on 26 commercial fireclays and 3 samples of quartz and silica sand, the tests including chemical analysis, tempering water and absorption of grog, linear shrinkage and unfired strength, porosity of fired clays, pyrometric cone equivalents, linear thermal expansion, the effect of firing temperature on the mineralogical composition of the fireclays, moduli of elasticity and transverse strength of the fired clays, and the plastic flow. The data are presented in tables and graphs. F. SALT.

Packing of particles. WESTMAN and HUGILL.—See I. **Resistance furnaces.** SCHMIDT.—See XI.

PATENTS.

[Multiple-type, regenerative] tunnel kilns. WOODALL-DUCKHAM (1920), LTD., DOULTON & Co., LTD., and C. BAILEY (B.P. 334,951, 14.6.29).—At the heating and cooling zones of the kiln superposed groups of flues are built into the side walls, and are of varying length with increasing cross-sectional area towards the hotter end. These flues are intended to produce the desired gradation of heating or cooling both in a longitudinal direction and also from floor to crown. C. A. KING.

Kilns or ovens for firing pottery, earthenware, and the like. O. A. VERMORCKEN (B.P. 312,942, 1.6.29. Belg., 2.6.28).—A kiln of the intermittent up-draught or down-draught type is heated by producer gas supplied to a central header beneath the kiln floor, and thence distributed to burner mouths situated at the internal periphery of the kiln and at or above the floor level, the air for combustion being charged to the burners through ducts at the level of the burner mouth. The combustion gases are withdrawn through flues arranged radially between the flues leading the gas to the burners. L. A. COLES.

[Non-tarnishing] optical glass. M. R. SCOTT, Assr. to BAUSCH & LOMB OPTICAL Co. (U.S.P. 1,765,287, 17.6.30. Appl., 11.6.28).—A dense barium crown optical glass containing more than 25% Ba, and having n_D and v values of 1.56—1.63 and 40—64, respectively, is rendered more resistant to surface tarnishing if not

more than 5% of the zinc oxide or other suitable constituent be substituted by zirconium oxide.

M. PARKIN.

Production of laminated glass. F. B. DEHN. From LIBBEY-OWENS GLASS Co. (B.P. 334,378, 30.8.29).—The glass sheets before combination with a non-brittle layer are skin-coated with a medium comprising gelatin, nitrocellulose, water, acetone, and acetic acid.

L. A. COLES.

Resistor material and its manufacture. M. H. HUNT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,764,311, 17.6.30. Appl., 1.11.22).—A mixture of powdered, low-fusing (below 900°) silicate glass 35, powdered quartz 52, plastic clay 10, and lamp black 3 pts., ground together wet or dry, and brought to a suitable consistency by means of water and a suitable electrolyte such as sodium silicate or carbonate, is used in the production of resistor bodies by casting or extrusion processes. After drying at above 150°, the bodies are fired under oxidising and reducing conditions for 5—20 min. at 500—900°, to give a non-vitreous, porous material. M. PARKIN.

[Coloured] refractory products [cements]. S. S. COLE, Assr. to KOPPERS Co. (U.S.P. 1,763,882, 17.6.30. Appl. 23.2.27).—The addition of 0.05—0.5% (preferably 0.2%) of Prussian blue to a refractory silica cement used for patching furnaces etc. gives increased strength, particularly between 800° and 1000°; borax (up to 2%) may be added as a fluxing agent. The use of the pigment enables the extent of the patching to be detected when the furnace has cooled down, and also the colour of the cement after firing furnishes some indication of the temperature to which it had been subjected.

M. PARKIN.

Manufacture of ultra-violet-transmitting glass. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE ST.-GOBAIN, CHAUNAY & CIREY (U.S.P. 1,779,176, 21.10.30. Appl., 24.5.28. Fr., 4.6.27).—See B.P. 291,468; B., 1930, 145.

Moulding of articles of fused quartz. DEUTSCH-ENGLISCHE QUARZSCHMELZE GES.M.B.H. (B.P. 336,493, 6.12.29. Ger., 12.12.28).

Manufacture of unsplinterable glass. T. W. HOLT and J. F. W. STUART (B.P. 336,037, 1.8.29 and 29.4.30).

Application of gelatinous films to glass and the like. S. BARRATT (B.P. 336,219, 2.7.29).

Agglomerating processes (B.P. 334,831).—See IX. **Patterns on metallic articles** (B.P. 335,789).—See X.

IX.—BUILDING MATERIALS.

X-Ray study of the constitution of Portland cement. L. T. BROWN MILLER and R. H. BOGUE (Amer. J. Sci., 1930, [v], 20, 241—264).—The compound $3\text{CaO} \cdot \text{SiO}_2$ has been found in the system $\text{CaO}-\text{SiO}_2$. Calcium oxide does not dissolve in the solid compound $2\text{CaO} \cdot \text{SiO}_2$ in appreciable amounts. Mixtures of compositions between $2\text{CaO} \cdot \text{SiO}_2$ and $3\text{CaO} \cdot \text{SiO}_2$, after fusion, contain an equilibrium mixture of $3\text{CaO} \cdot \text{SiO}_2$ and β - or γ - $2\text{CaO} \cdot \text{SiO}_2$. No compound $8\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ exists in the system $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$ or in Portland

cement; the compounds $3\text{CaO}, \text{SiO}_2$, $\beta\text{-}2\text{CaO}, \text{SiO}_2$, and $3\text{CaO}, \text{Al}_2\text{O}_3$ are formed. In the system $3\text{CaO}, \text{SiO}_2$ - $2\text{CaO}, \text{SiO}_2$ - $3\text{CaO}, \text{Al}_2\text{O}_3$ solid solutions do not occur in appreciable amounts. Free calcium oxide was not found in commercial clinkers, but less than 2.5% could not have been recognised. X-Ray diffraction patterns of 28 Portland cement clinkers have been obtained. The chief constituents are $3\text{CaO}, \text{SiO}_2$ and $\beta\text{-}2\text{CaO}, \text{SiO}_2$. The compounds $3\text{CaO}, \text{Al}_2\text{O}_3$, $4\text{CaO}, \text{Al}_2\text{O}_3$, Fe_2O_3 , and MgO were found, separately or together, in most of the clinkers. The results are in agreement with those obtained by phase-rule, chemical, and microscopical methods. C. W. GIBBY.

Electrical investigation of the setting and hardening of mixed cements containing iron blast-furnace slag. Y. SHIMIZU (Sci. Rep. Tōhoku, 1930, 19, 307—314).—The investigation was carried out by means of an electrical conductivity method, the cements used being blast-furnace and iron Portland cements and also a new kind of rapid hardening ("slag-aluminous") cement. The last-named is made by adding basic slag to aluminous cement or aluminous cement clinker, and fine-grinding the resulting mixture. Although quenched slag itself can behave as a cement, its hydraulic reaction is too slow for use in practice. If mixed with Portland or aluminous cement, however, the reaction of the latter accelerates that of the quenched slag. If the mixture is made in suitable proportions the time of occurrence of the reaction can be regulated, and it can be so arranged that the reactions of the constituents are simultaneous. It was found that for a cement mixed in the correct proportions for simultaneous reaction, as determined by the electrical conductivity method, the strength is a maximum. By a comparison of the strengths of the three cements it was found that the slag-aluminous cement is comparable in strength with aluminous cement. A. J. MEE.

PATENTS.

Heat-conducting cement compositions. BABCOCK & WILCOX, LTD. From FULLER LEHIGH CO. (B.P. 335,086, 15.10.29).—A cement suitable for jointing between metal tubes and refractory lining components, *e.g.*, water-tube boilers, consists of about 35 pts. of sodium silicate solution (*d* 1.41), 13 pts. of glycerin (*d* 1.25), and 2 pts. of castor oil. Graphite exceeding the weight of the other ingredients, *e.g.*, 50—100 pts., is added. C. A. KING.

Manufacture of artificial stone plates of fibrous materials and hydraulic cement. R. KUNZ (B.P. 335,106, 8.11.29. Austr., 23.11.28).—A mixture of hydraulic cement, fibrous material, and water is treated on a paper machine, a mineral oil being incorporated at any stage of the process. C. A. KING.

Manufacture of artificial stone, marble, or the like. J. J. BURKE (B.P. 335,107, 8.11.29).—A 1:2 cement-sand mixture is made into a paste suitable for moulding by the addition of a solution composed of 40 gals. of water, 4 lb. of sodium silicate, 2 lb. of boric acid, and 2 lb. of alginic acid. C. A. KING.

Manufacture of road-surfacing materials. SOUTH METROPOLITAN GAS CO., and H. PICKARD (B.P. 334,336, 23.7.29).—The materials comprise mixtures prepared

at 120—150° of hard stone ($\frac{3}{8}$ in. diam. to dust) with a binder prepared by dispersing at 250—300° 18—25% of coal in tar, which may contain not more than 25% of added pitch, the proportions of the constituents and the range of sizes of stone being such that the sinking rate of a plunger (diam. 1 in.; end hemispherical) is not greater than 1 in. per min. under a weight of 100 lb., nor less than 1 in. in 10 min. under 1500 lb.

L. A. COLES.

Agglomerating processes [for making paving, flooring, refractories, etc.]. "CEMSIL" SOC. ANON. ITAL., and G. BOGGERO (B.P. 334,831, 9.3.29).—A solution of an alkaline silicate (sodium silicate) is treated with a weak acid, *e.g.*, carbonic, sulphurous, or boric acid, under constant stirring, to a point just before coagulation, which point is indicated by a definite decrease in viscosity. An agglomerate, *e.g.*, a rolled foundation for a road or pavement, is then sprayed or coated with this solution, allowed to dry, and is finally sprayed with a fixing solution containing calcium chloride or similar salt. C. A. KING.

Production of well-adhering coatings on asbestos-cement-slate plates. A. FRICKE, Assr. to HAWENTA-PLATTEN GES.M.B.H. (U.S.P. 1,779,229, 21.10.30. Appl., 4.5.29. Ger., 15.7.27).—See B.P. 327,871; B., 1930, 770.

Bituminous emulsions. G. S. HAY, Assr. to FLINT-KOTE ROADS, INC. (U.S.P. 1,778,760, 21.10.30. Appl., 5.11.25. U.K., 16.12.24).—See B.P. 248,859; B., 1926, 409.

Mixing machines [for concrete]. J. M. VER MEHR (B.P. 335,982, 5.7.29).

Heat-insulating material (B.P. 334,884).—See I. **Refractory cements** (U.S.P. 1,763,882).—See VIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Melting of steel in an acid-lined Brackelsberg furnace. P. BARDENHEUER (Stahl u. Eisen, 1930, 50, 1328—1330).—Using an air preheater in which the air blast is heated to 200—300° by means of the outgoing gases from the furnace, the Brackelsberg furnace may be rapidly heated up to 1800°. Steel is readily melted on an acid lining in the furnace, using for a 1-ton charge about the same amount of fuel as is required per ton of charge in a large open-hearth furnace. The charge can be particularly easily cast and the resulting ingots have good mechanical and technological properties. The silicon, manganese, and carbon contents of the castings are appreciably lower and the phosphorus and sulphur contents slightly higher than in the charge added to the furnace. A. R. POWELL.

Tests of an electrical [blast-furnace] gas-purification plant. A. MICHEL (Stahl u. Eisen, 1930, 50, 1356—1361).—The Elga electric filtration plant for the removal of dust from iron blast-furnace gas at the Huckingen works of the Mannesmann Tube Co. is described and illustrated with scale drawings, and some account is given of the results obtained with it. The plant has a capacity of 270,000 m.³/hr. (at N.T.P.) with a gas velocity of 2.3 m.³/sec. at 70°, so that the average duration

of exposure of the gas to the electric field is 1.3 sec. The best results are obtained with a moisture content of about 80 g./m.³ of dry gas (at N.T.P.) and the gas should be cooled to below 80° before passing to the filters. The cleaned gas has a dust content of only 0.02 g./m.³ under these conditions, but if its temperature reaches 110° in the filters it contains 0.1 g./m.³. The average composition of the dust collected in the settlers before the filters is about 43% Fe, 8% Si, 1% Mn, 8% CaO, 3% Al₂O₃, 1.2% P, 6% C, and 3–5% (K₂O + Na₂O). Dust from the filters contains 9% Fe, 1% Mn, 11% SiO₂, 5% Al₂O₃, 11% CaO, 3% MgO, 10–16% (K₂O + Na₂O), up to 1% Pb, and up to 4% Zn. A. R. POWELL.

Structure of heat-treated low-carbon steel. W. J. CROOK and H. S. TAYLOR (Met. and Alloys, 1930, 1, 539–543, 594).—The hardness of a steel containing C 0.20, Mn 0.59, P 0.016, S 0.042, Si 0.13, Cu 0.29, Ni 0.19%, and a trace of chromium decreased uniformly with rise of the drawing temperature. A mechanism for the transformation which occurs when quenched martensitic low-carbon steel is subjected to rising drawing temperatures is proposed.

CHEMICAL ABSTRACTS.

Rate of grain growth in low-carbon steels. H. E. PUBLOW and S. E. SINCLAIR (Mich. Eng. Exp. Sta. Bull., 1930, No. 29, 20 pp.).—Steels containing 0.02–0.08% C reach the same maximum grain size when heated for a sufficiently long time at 871–1038°. Steels heated just above the Ac₃ point for 5 min., and furnace-cooled, are uniformly fine-grained regardless of their previous structure. The grain growth is unaffected by nitrides. There is no relation between the Rockwell hardness and grain size in annealed steels.

CHEMICAL ABSTRACTS.

Normal and abnormal steel. E. HOUDREMONT and H. MÜLLER (Stahl u. Eisen, 1930, 50, 1321–1327).—A critical review of recent American work on the case-hardening of steel.

A. R. POWELL.

Etching figures in iron and steel. V. N. SVETCHNIKOV (Rev. Mét., 1930, 27, 404–411).—Examples are described with reference to photomicrographs of characteristic etch figures produced on the crystal grains of mild steel, silicon steel, and copper steel by etching with Heyn's reagent. The shape of the figures is always the same on any one grain, but varies from grain to grain according to its orientation.

A. R. POWELL.

Thermochemistry of iron. W. A. ROTH, H. UMBACH, and P. CHALL (Arch. Eisenhüttenw., 1930–1, 4, 87–93; Stahl u. Eisen, 1930, 50, 1331–1332).—From measurements of the heat evolved when iron and its various oxides are dissolved in 20% hydrochloric acid at 97° the following heats of formation have been determined: α -Fe + O = FeO + 64.25 kg.-cal.; FeO + Fe₂O₃ = Fe₃O₄ + 7.0 kg.-cal.; 2FeO + SiO₂ (quartz) = Fe₂SiO₄ + 8.1 kg.-cal.; Fe₂O₃ + 3Cl₂ = 2FeCl₃ + 3O + 7.4 kg.-cal. (cf. Roth, A., 1929, 1389).

A. R. POWELL.

Physical properties of manganese steel. A. D. ROSS (J. Roy. Soc. W. Australia, 1926–1927, 13, 73–77).—When cooled in liquid air, manganese steels containing more than 1% Mn exhibit increased magnet-

isation owing to the transformation of γ - into α -iron. A high-carbon steel quenched at 1100° and cooled in liquid air behaved similarly. CHEMICAL ABSTRACTS.

Magnetic properties of permalloy: influence of internal tension, longitudinal stretching, and of [hydraulic] pressure. O. VON AUWERS (Wiss. Veröff. Siemens-Konz., 1930, 9, 262–293).—The effect of artificially-induced internal tension on the magnetic properties of cased wires has been measured. The core consisted of materials having variable expansion coefficients; the outer layer was of permalloy. Longitudinal stretching causes the hysteresis loops to rotate about two fixed points.

W. E. DOWNEY.

Nickel-chromium-copper cast iron ("Nimol"). M. WAHLERT and H. OSTERMANN (Chem. Fabr., 1930, 377–378, 386–388, 402–404).—Nimol is an alloy of cast iron with 20% of monel metal (66% Ni, 34% Cu) and 2–4% Cr; it is non-magnetic, has an austenitic structure with inclusions of graphite and carbide, a tensile strength of 14–19 kg./mm.², and a hardness of over 200 Brinell. The alloy has a high resistance to corrosion by sulphuric, hydrochloric, and acetic acids and is more resistant to the action of the salt spray and to immersion in sea-water than bronze; it is highly resistant to scaling and to structural changes up to 925°. Tables are given showing the relative behaviour of nimol, cast iron of various compositions, and several bronzes in numerous corroding media, and the changes which take place in the hardness of various cast-iron alloys during annealing at temperatures up to 925°.

A. R. POWELL.

Dilatometric study of the transformations and heat treatment of light aluminium alloys. A. PORTEVIN and P. CHEVENARD (Rev. Mét., 1930, 27, 412–435).—The application of dilatometric analysis to the study of the changes which take place during the ageing of quenched aluminium alloys is demonstrated with reference to alloys containing Mg₂Si and Al₂Cu. The results obtained confirm the conclusions of Gayler and Preston (B., 1929, 327) and of von Göler and Sachs (Metallwirtschaft, 1929, 8, 671) with respect to the mechanism of age-hardening of these alloys. The dilatometer shows that the rate of precipitation of Mg₂Si from solid solution in aluminium at 20–100° is an exponential function of the temperature, doubling itself for an increase in temperature of about 10°. In the case of copper-aluminium alloys precipitation of Al₂Cu below 175° is accompanied by an expansion in volume and by a decrease in electrical resistance. The second hardening effect above 175° is, however, accompanied by a small contraction in volume as well as by an increase in resistance, although micrographic and X-ray examinations fail to reveal any structural change.

A. R. POWELL.

Effect of thermal treatment on the resistivity and mechanical resistance of aluminium alloys containing up to 2.5% Si. L. GUILLET and M. BALLAY (Rev. Mét., 1930, 27, 398–403).—Quenching from 500° increases the resistivity of silicon-aluminium alloys containing less than 2.5% Si; on subsequently annealing the quenched alloys the resistivity decreases to a minimum at about 350°. The presence of iron

increases the solid solubility of silicon in aluminium at 500° from 0.44% Si with 0.15% Fe to 0.7% Si with 0.8% Fe. Extrapolation of the resistivity curves to 0% Si and 0% Fe indicates that the resistance of pure aluminium at 16° is 2.62 microhms-cm.²-cm. Quenching increases the resistance to shearing stresses of these alloys, and if the alloys are quenched from 600° subsequent annealing still further increases the shear strength. A. R. POWELL.

Wetting phenomena with blende and lead glance. E. BERL and B. SCHMITT (Kolloid-Z., 1930, 52, 333—341).—The wetting of materials such as blende and lead glance by liquids is determined largely by the presence of surface films of oxidation products. The effect of alkali hydroxides, cyanides, and xanthates on the wettability of blende and lead glance has been investigated. Alkalies repress the wettability of blende by benzene, owing to the formation of a layer of alkali zincate at the surface of the particles, thus increasing the hydrophilic properties of the solid phase. Similarly, alkali cyanides form a hydrophilic surface layer of the complex alkali zinc cyanide, whilst no corresponding compound is formed from lead glance. Blende and lead glance may therefore be separated by flotation with benzene in the presence of alkali cyanides. E. S. HEDGES.

Destructive action of molten zinc, at and above galvanising temperatures, on metals and alloys. VII. Nickel and nickel alloys. W. G. IMHOFF (Amer. Met. Market, 1930, 37, No. 106, 5).—Nickel and chromium are more resistant than their alloys, and than is iron or steel. CHEMICAL ABSTRACTS.

Formation of zinc and alloy layers during galvanising. H. BABLICK (Iron Age, 1930, 125, 1528—1531).—Dissolution of iron in zinc forms brittle crystals of composition corresponding with the formula FeZn_7 ; some of these remain attached to the iron to form the inner layer. In order to produce sheet with good bending qualities the thickness of the alloy layer can be reduced by diminishing the dipping time, lowering the temperature, and reducing interaction by alloying the zinc, e.g., with aluminium. CHEMICAL ABSTRACTS.

Effect of anodic impurities on the electrodeposition of gold. R. W. HARBISON (Deut. Goldschmiedeztg., 1930, 33, 139—140; Chem. Zentr., 1930, i, 2955).—Lead, bismuth, selenium, tellurium, arsenic, and antimony should first be removed. In presence of silver, copper, and the platinum metals the composition of the electrolyte must be altered. The gold content of the electrolyte is continuously determined by addition of ferrous ammonium sulphate and titration of the excess with permanganate. A. A. ELDRIDGE.

Variations in thickness of metal [electro-] deposits. I. L. E. and V. E. GRANT (Metal Ind., N.Y., 1930, 28, 275—276).—A description of the technique employed in a study of the effect of composition of the solution, spacing of the electrodes, p_H , etc. on the thickness of electrodeposits of nickel. CHEMICAL ABSTRACTS.

Setting of cements containing slag. SHIMIZU.—See IX. **Resistance furnaces.** SCHMIDT.—See XI. **Contamination of food in iron alloys.** TITUS and others.—See XIX.

PATENTS.

Cupola furnaces. L. A. BENTLEY (B.P. 334,917, 12.6.29).—The furnace comprises a stack surrounded by outer and inner jackets through which the air for the tuyères is caused to pass, upwards through the outer annular space, and downwards through the inner, and means for supplying part of this preheated air to numerous openings in the brickwork above the charge so as to complete the combustion of the partly-burnt gases in the upper zone of the stack. A. R. POWELL.

Decarburisation of iron and steel and their alloys. SIEMENS & HALSKE A.-G. (B.P. 314,439, 26.6.29. Ger., 27.6.28).—The metal is heated by high-frequency induction currents in an atmosphere of hydrogen at above 1000° and under more than 100 atm. pressure. The methane formed and excess hydrogen are then removed, and the metal is heated in a vacuum to remove the last traces of adsorbed gas and carbonaceous material. The resultant metal is claimed to have a carbon content below 0.005%. A. R. POWELL.

Apparatus for treatment of liquid slags and the like. NORSKE A./S. FOR ELEKTROKEM. IND. (B.P. 335,875, 24.2.30. Nor., 27.2.29).—Liquid slag is passed through a container and by means of stirrers is converted into a liquid foam which is cast in moulds. The container may be made sectionally of cast iron, on which the slag itself forms a refractory lining. C. A. KING.

[Flotation] concentration of ores. B. L. JOHNSON (U.S.P. 1,763,851, 17.6.30. Appl., 31.10.28).—Claim is made for the use as flotation agents of compounds of the general formula: $[(\text{RO})_2\text{PS}_2]_2$, where R is one or more alkyl or aryl radicals, which may be all alike or different. Tetraethyl tetrathiodiphosphate (m.p. 72—73°) is prepared by the action of concentrated sulphuric acid on diethyl dithiophosphate. A. R. POWELL.

Purification of pig iron. F. KRUPP A.-G. (B.P. 312,361, 21.5.29. Ger., 25.5.28).—Molten pig iron in a hearth furnace is covered with a slag containing carbon and the usual basic fluxes for the removal of sulphur and phosphorus. E.g., pig iron with 3.8% C, 1.2% Mn, 0.5% Si, 0.8% P, and 0.06% S is treated with a mixture of 2.5 kg. of carbon, 15 kg. of iron oxide ore, 20 kg. of lime, and 10 kg. of fluorspar per 100 kg. of metal; the refined iron contains 3.7% C, 0.5% Mn, 0.3% Si, <0.01% P, and <0.01% S. A. R. POWELL.

Protection of iron from rust. W. BÖHMER (B.P. 335,868, 27.1.30).—The clean iron surface is coated with a mixture of 10 pts. of red lead, 2 pts. of litharge, 3 pts. of glycerol (d 1.23—1.26), and 9 pts. of sodium silicate solution (d 1.33). After drying, a coating of 10 pts. of zinc oxide in 10 pts. of sodium silicate solution (d 1.26) is applied and finally the article is dipped in a solution of zinc chloride (d 1.52—1.7), which converts the free zinc oxide into zinc oxychloride. A tenacious, imperious, white coating resistant to shock and to the action of steam is obtained. A. R. POWELL.

Improving the physical properties of steel containing copper. W. E. EVANS. From VEREIN. STAHLWERKE A.-G. (B.P. 309,132, 28.3.29).—Steel containing 0.6—10% Cu and up to 0.5% C is subjected

to its final shaping operation at 800–900°, air-cooled, and then annealed for at least 1 hr. at a temperature below the Acl point, *e.g.*, at 600°. In this way the elastic limit is increased to 75–80% of the tensile strength.

A. R. POWELL.

Heat-resisting [iron-chromium-nickel] alloys.

W. ROSENHAIN and C. H. M. JENKINS (B.P. 335,588, 23.4.29).—Alloys containing 30% Ni, 20–30% Cr, 4–20% W, 0.5–1.5% C, 1–1.5% Si, and the remainder iron are claimed. A typical alloy contains 20% W, 27.5% Fe, 30% Ni, 20% Cr, 1% C, and 1.5% Si.

A. R. POWELL.

Copper alloys [hardened with iron silicide].

VICKERS-ARMSTRONGS, LTD., W. MACHIN, and W. B. O'B. GOUDIELOCK (B.P. 335,950, 4.4. and 13.7.29).—Alloys of copper with zinc and iron silicide with or without the addition of other metals are claimed, the iron silicide replacing the tin of ordinary bronzes. The alloying constituents are added in the form of a hardener made by melting cast iron, deoxidising with phosphor-copper, and adding the requisite amount of silicon and ferromanganese; the hardener is poured directly into molten copper and finally the desired amount of zinc is added. The alloys may be heat-treated by quenching from above 900° and annealing at 200–450°. They may be used for all purposes for which the corresponding tin bronze is suitable.

A. R. POWELL.

Alloy of high surface stability comprising nickel, [iron], and silicon. P. A. E. ARMSTRONG and R. P. DE VRIES, Assrs. to LUDLUM STEEL Co. (U.S.P. 1,759,477, 20.5.30. Appl., 7.1.21).—An alloy resistant to acids and to scaling at high temperatures comprises iron with 20–60% Ni or Co, 2–10% Si, and <1% C. The alloys are suitable for electrical heating elements, case-hardening or annealing boxes, and for the manufacture of vessels resistant to sulphuric or hydrochloric (but not nitric) acid.

A. R. POWELL.

Blende-sintering process. SOC. ANON. LA NOUVELLE MONTAGNE (B.P. 335,609, 27.6.29. Belg., 9.3.29).—A mixture of blende and zinc sulphate is sintered under forced draught, the endothermic dissociation of the zinc sulphate controlling the temperature of the mass. About 3 mols. of zinc sulphate and 2 mols. of zinc sulphide in excess of the sulphide necessary to maintain combustion is the proportion recommended.

C. A. KING.

Increasing the grain size of sulphide ores during [blende] desulphurisation. METALLGES. A.-G. (B.P. 335,852, 1.1.30. Ger., 4.6.29).—Clay, loam, or ferric sulphate is added before or during the early stages of roasting, so as to cause the production of a granular sinter.

A. R. POWELL.

Production of zinc and other metals. L. STURBELLE (B.P. 313,858, 17.6.29. Belg., 15.6.28).—Roasted or oxidised zinc ores are extracted with sulphuric acid or spent electrolyte on the countercurrent principle and the solution is purified by sucking it through filter beds containing zinc sulphide. The filter cakes are subsequently caused to circulate in countercurrent to the solution undergoing purification and the purified solution is electrolysed for the recovery of zinc. A. R. POWELL.

Recovering metals [*e.g.*, lead from ores]. R. W. HYDE, Assr. to DWIGHT & LLOYD METALLURG. Co.

(U.S.P. 1,746,945, 11.2.30. Appl., 21.5.27).—Lead sulphide ores are subjected to an oxidising sintering process and the sinter is heated on perforated beds in a reducing atmosphere so that the reduced lead melts and drains away through the porous mass into a sump in the furnace from which it may continuously be siphoned off. Draining of the lead is accelerated by shaking the perforated shelves. The unmelted gangue material may subsequently be smelted for the recovery of the less fusible metals which it may contain.

A. R. POWELL.

Soldering and metal-coating lead alloy. (Sir)

G. C. MARKS. From AMER. MACHINE & FOUNDRY Co. (B.P. 335,545, 19.3.29).—Lead containing 0.01–0.03% P is claimed to be suitable as a solder and for use in the production of lead-coated metal articles. [Stat. ref.]

A. R. POWELL.

Improvement of high-percentage magnesium alloys.

I. G. FARBENIND. A.-G. (B.P. 336,019, 15.7.29. Ger., 25.4.29).—Magnesium alloys containing aluminium, zinc, or lead are heated at a temperature sufficiently high to produce a supersaturated solid solution of the alloying element, and are then allowed to cool slowly in such a way that the time required to cool through the range 300–100° exceeds 3 hrs. After annealing at 410° for 72 hrs. and cooling through the critical range for 24 hrs., the tensile strength of an alloy of magnesium with 8.2% Al increased from 16.6 to 23.3 kg./mm.², the elongation from 2.7–5.6%, and the yield point from 10.3–12.0 kg./mm.²

A. R. POWELL.

Treatment of arsenious ore.

A. R. LINDBLAD (U.S.P. 1,763,435, 10.6.30. Appl., 1.12.27. Swed., 10.6.27).—The ore is roasted in presence of air with substances capable of combining with the arsenic to form an arsenite or arsenate, and is then roasted in the usual manner with slag-forming materials. The arsenic compound is thus taken up by the slag and is readily disposed of.

W. J. WRIGHT.

Production of ductile chromium.

J. W. MARDEN and M. N. RICH, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,367, 27.5.30. Appl., 27.4.26).—Chromium powder, produced by the reduction of chromic oxide with calcium in the presence of calcium chloride, is pressed into bars which are sintered in hydrogen by passing through them 80% of the current required to melt the metal. After cooling in hydrogen, the slugs are swaged at 800–1100° and then at gradually lower temperatures until a ductile wire is obtained.

A. R. POWELL.

Sintered alloys containing tungsten compounds.

R. WALTER (B.P. 335,453, 28.11.29. Ger., 3.12.28).—The alloys contain 50–80% of tungsten carbide and/or tungsten carbonitride together with one or more metals of the iron or chromium groups. *E.g.*, an alloy for the manufacture of lathe tools comprises 60% WC, 26% W, 12.5% Co, and 1.5% Cr₃C₂.

A. R. POWELL.

Production of mercury.

W. GLAESER, Assr. to GLAESER RES. CORP. (U.S.P. 1,762,861, 10.6.30. Appl., 14.11.24).—Finely-ground cinnabar is heated at 60° with a dilute acid or alkali and an electropositive metal, *e.g.*, zinc or aluminium, or with a metal couple, *e.g.*, a zinc-copper couple. The mercury liberated forms an amalgam with the excess of zinc or aluminium, which

can be recovered by water concentration and distilled to obtain mercury. A. R. POWELL.

[Stable] preparation of colloidal silver. T. SATOW (U.S.P. 1,764,417, 17.6.30. Appl., 23.1.26).—A 20% solution of silver nitrate is treated with a solution of dextrin and sodium hydroxide, with or without the addition of lanolin as a protective colloid, and kept for several hours. A. R. POWELL.

Welding rods. (A) W. B. MILLER and (B) S. M. NORWOOD, ASSRS. to ELECTRO-METALLURG. Co. (U.S.P. 1,762,482—3, 10.6.30. Appl., [A] 3.5.27, [B] 24.10.25).—Claims are made for welding rods consisting of alloys of iron with (A) 1.5—4% Mn, 0.2—0.6% Mo, 0.1—0.4 Si, and 0.05—0.5% C; or (B) 10—35% Cr, 0.1—3% Mn, 0.75—3% Si, and 0.1—1% C. A. R. POWELL.

Coating for [iron] welding rods. W. A. WISSLER, ASSR. to HAYNES STELLITE Co. (U.S.P. 1,764,589, 17.6.30. Appl., 22.11.27).—The coating comprises a mixture of equal parts of calcium carbonate, powdered silica, and powdered borax glass, bonded with a solution of shellac in alcohol. A. R. POWELL.

[Copper] welding alloy [for ferrous metals]. A. R. LYTLE, ASSR. to UNION CARBIDE & CARBON RES. LABS., INC. (U.S.P. 1,762,871, 10.6.30. Appl., 20.12.29).—An alloy of copper with 25—60% Ni, < 25% Zn, and 0.05—4% Si is claimed. The preferred composition is 30% Ni, 20% Zn, and 50% Cu, with 0.05% Si. A. R. POWELL.

Production of age-resisting welded seams when welding age-resisting iron or steel alloys by the press-welding process. F. KRUPP A.-G. (B.P. 316,556, 25.7.29. Ger., 30.7.28).—The weld is subjected to an annealing process above the Ac₃ point, followed by air-cooling or chilling and tempering. A. R. POWELL.

Electrodes for spot-welding of copper. F. KRUPP A.-G. (B.P. 335,876, 24.2.30. Ger., 26.3.29).—Electrodes consisting of an alloy of iron, cobalt, or nickel with tungsten carbide, e.g., "Widia" metal, are claimed. The copper surface is covered with borax and the welding takes place in an atmosphere of nitrogen and hydrogen. A. R. POWELL.

Soldering [chain links, metal mesh, etc.]. G. BRUNHÜBNER, ASSR. to E. G. BEK (U.S.P. 1,764,604, 17.6.30. Appl., 24.12.26. Ger., 12.10.25).—The solder comprises a pasty mixture of a metal powder, red phosphorus, boric acid, and an oil. A. R. POWELL.

Electrodeposition of chromium. E. V. HAYES-GRATZE (B.P. 335,161, 13.6. and 24.7.29).—When lead or lead alloy anodes are used in the deposition of chromium, a more even distribution of the deposit over the surface of the articles to be plated is obtained by the use of auxiliary cathodes with or without suitable insulating shields or deflectors for the prevention of gas accumulations. A. R. POWELL.

Electrolytic [perforation] treatment of metals. W. GUSSEFF (B.P. 335,003, 24.7.29).—An apparatus for making holes or cavities in metals by electrolytic action is described. A. R. POWELL.

Furnace for roasting sulphide and other ores. E. BRACQ (U.S.P. 1,778,415, 14.10.30. Appl., 11.2.26. Fr., 9.3.25).—See B.P. 248,711; B., 1927, 583.

Apparatus for roasting or sintering of ores and the like. G. E. STORER, ASSR. to NAT. PROCESSES, LTD. (U.S.P. 1,778,872, 21.10.30. Appl., 18.1.29. U.K., 21.7.28).—See B.P. 306,787; B., 1929, 329.

Improvement of copper or copper alloys and hardener for use therein. W. MACHIN and W. B. O'B. GOUDIELOCK, ASSRS. to P.M.G. METAL TRUST, LTD. (U.S.P. 1,777,174, 30.9.30. Appl., 10.7.29. U.K., 4.4.29).—See B.P. 335,950; preceding.

[Inhibitor for pickling] treatment of metals with acid liquors. M. HARTMANN and W. KLARER, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,778,634, 14.10.30. Appl., 4.6.29. Switz., 7.6.28).—See B.P. 313,135; B., 1930, 617.

Furnaces for annealing metal sheets by a continuous process. E. GRIFFITHS (B.P. 335,234, 21.5.29 and 21.3.30).

Mould for casting metals. A. E. WHITE. From ALUMINIUM, LTD. (B.P. 336,337, 1.8.29).

[Aeration] treatment of sands in foundry practice. J. E. GERNELLE (B.P. 336,248, 4.6.29).

Filtration of liquids (B.P. 334,663).—See I. Alkaline oxidant and aluminium vessels (U.S.P. 1,765,344).—See VII. Carbonaceous coatings on metal (B.P. 334,633).—See XI.

XI.—ELECTROTECHNICS.

Electrical resistance furnaces. E. SCHMIDT (Chem. Fabr., 1930, 409—410, 425—426).—The Wild-Barfield furnace for tempering carbon-steel is provided with a secondary winding by means of which the magnetism of a specimen in the furnace is recorded. When this reaches zero the optimum temperature for tempering has been reached. A furnace suitable for the heat-treatment of metals and for firing glass and enamels at 300—950° is described. It is fitted with chromium-nickel resistance elements on all sides of the heating chamber and is capable of regulation to $\pm 8^\circ$. Other furnaces mentioned are designed for the tempering of steel in baths of fused salt at up to 1300° and for the melting of white-metal alloys, tin, and lead. The latter type is designed so as to minimise atmospheric oxidation, and may be supplied with automatic temperature indicator. Drying ovens for temperatures up to 250° are also described. C. IRWIN.

Theory of the lead accumulator. L. JUMAU (World Power, 1930, 14, 303—304).—The increase in weight of the positive plate of the lead accumulator during discharge is in good agreement with the double sulphation theory, provided that the terminal voltage is not reduced to below about 1.6 volts; discharge to very low voltages causes the increase in weight to be considerably less than that required theoretically. Determination of the *E.M.F.* and of the temperature coefficient of the *E.M.F.* of lead peroxide in sulphuric acid, using mercurous sulphate as auxiliary electrode, yields for the heat of the reaction 46.9 kg.-cal., in good agreement with that calculated thermochemically on the basis of the double sulphate theory (47.1 kg.-cal.). The theory is supported also by measurements of the internal resistances of the two electrodes during charge, discharge, and inversion. H. F. GILLBE.

Transportable electric stirrers. H. WOLLENBERG (Chem.-Ztg., 1930, 54, 804).—An improved clamp, by means of which the stirrer shaft is always held vertically upright in the centre of the vessel, is described.

S. I. LEVY.

Electrolysis of colloidal solution of hemicellulose. LENOBLE.—See V. **Ultra-violet-transmitting glasses.** STOCKBARGER and JOHNSON.—See VIII. **Gas-purification plant.** MICHEL. **Manganese steel.** ROSS. **Magnetic properties of permalloy.** VON AUWERS. **Silicon-aluminium alloys.** GUILLET and BALLAY. **Electrodeposition of gold.** HARBISON. **Thickness of metal deposits.** GRANT and GRANT.—See X. **Action of silent discharge on oils etc.** IWAMOTO.—See XII.

PATENTS.

Electric induction furnaces. C. LORENZ A.-G. (B.P. 335,766, 23.10.29. Ger., 25.10.28).—The refractory, non-conducting crucible of an electric induction furnace, heated by polyphase current, is co-axially surrounded by an annular group of sheet metal laminations having a winding in which polyphase currents flow, so that material to be treated is heated directly by eddy currents and is agitated by the impressed, rotating, electromagnetic field.

J. S. G. THOMAS.

Electric arc carbons or electrodes. COMP. LORRAINE DE CHARBONS POUR L'ELECTRICITE (B.P. 316,130, 8.6.29. Fr., 23.7.28).—An arc carbon suitable for use in panchromatic photography is composed substantially of calcium fluoride 13 pts., strontium fluoride 10 pts., barium fluoride 7 pts., chromium fluoride 1–3 pts., powdered copper 1–3 pts., sodium borate 0–0.05 pt., carbon or graphite 30–40 pts., and, if desired, 1–2 pts. each of magnesium fluoride and calcium fluorotitanate.

J. S. G. THOMAS.

Manufacture of electric cables. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 334,840, 9.5.29).—A conductor is insulated with thermoplastic material, e.g., gutta-percha, balata, rubber, rubber-bitumen mixtures, which is treated so as to develop a cellular structure. Thus, e.g., air or other gas under pressure may be enclosed in the material and the pressure released. Alternatively, a substance from which gas is evolved on heating, e.g., ammonium carbonate, may be incorporated in the thermoplastic material before heating.

J. S. G. THOMAS.

[Electrode for] electric-discharge tubes. EGYE-SÜLT IZZÓLAMPÁ ÉS VILLAMOSSÁGI RÉSVÉNTÁRSASÁG (B.P. 319,206, 17.8.29. Hung., 17.9.28).—A solid insulating core, e.g., of glass, is coated with a metal or carbon, and is provided with a metallic conducting lead, e.g., a metal wire fused into the surface of the core. Such electrodes require no heating and are thermionically and photoelectrically inactive.

Production of carbonaceous coatings upon metallic bodies for use in electron-discharge devices. A. S. CACHEMAILLE. From WESTINGHOUSE LAMP CO. (B.P. 334,633, 15.6.29).—To prevent overheating of the plates and grids of radio valves they are first deoxidised by heating in hydrogen, then coated with a layer of carbon black by heating in an atmosphere of hydrogen

and hydrocarbon vapour, and finally heated in a vacuum to remove occluded gas from the deposit and to cause it to adhere tightly to the metal.

A. R. POWELL.

Photoelectric cell. ARCTURUS RADIO TUBE CO., Assecs. of S. RUBEN (B.P. 317,862, 23.8.29. U.S., 24.8.28).—A light-sensitive electrode, preferably composed of copper coated with cuprous oxide, and a co-operating reticular electrode, preferably containing nickel, make contact with opposite faces of a translucent, electrolytically conducting crystal plate, e.g., of sodium chloride or Rochelle salt. Means are provided for controlling an external electric circuit by the piezoelectric effect produced in the crystal by variation of potential between the electrodes.

J. S. G. THOMAS.

[Alkaline] electrical accumulators or secondary batteries. J. J. DRUM, and CELIA, LTD. (B.P. 335,587, 27.3.29).—A positive electrode, composed of a mixture of silver (or silver oxide) and ceric oxide, and a negative electrode, composed of oxides of iron and/or cadmium, immersed in 25–30% caustic potash solution, are arranged in a container made of rustless or nickel-plated steel.

J. S. G. THOMAS.

Electrolytic apparatus [for electrolysis of water and collection of evolved gases]. A. E. KNOWLES (B.P. 334,991, 6.7.29).—Gas offtake pipes terminate within, and above the bottom of, the gas main, the lower part of which forms a liquid channel below the orifices of the offtake pipes and has no connexion therewith. A return pipe to the cell tank or casing is arranged from each main collecting pipe, which is provided with a cock for removal of scum or froth.

J. S. G. THOMAS.

Temperature indicators [for oil-immersed electric apparatus]. BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 336,431, 14.10.29).

Means for handling objects in baths, particularly for electrolytic baths. SIEMENS & HALSKE A.-G. (B.P. 336,386, 24.9.29. Ger., 28.1.29).

Gas detector (B.P. 334,640). **Insulating oil** (U.S.P. 1,760,539).—See II. **Decarburisation of iron and steel** (B.P. 314,439). **Nickel-iron-silicon alloy** (U.S.P. 1,759,477). **Zinc etc.** (B.P. 313,858). **Ductile chromium** (U.S.P. 1,760,367). **Spot-welding of copper** (B.P. 335,876). **Chromium** (B.P. 335,161). **Treatment of metals** (B.P. 335,003).—See X.

XII.—FATS; OILS; WAXES.

Action of silent discharge on oils, fats, and fatty acids. I. Deodorisation of cod-liver oil. Y. IWAMOTO (J. Soc. Chem. Ind., Japan, 1929, 32, 93–94 B).—Cod-liver oil was subjected to the action of the silent discharge in hydrogen and nitrogen atmospheres under ordinary pressure. The sp. gr. and refractive index of the oil increased somewhat, and the iodine value decreased by some 12–20%. When hydrogen was used the oil was perfectly deodorised after 3 and 1.5 hrs. treatment under pressures of 10,000 and 20,000 volts, respectively, and the vitamin-A potency, as measured by colour reactions and physiological tests, was unchanged.

E. LEWKOWITSCH.

Iodometric acid values of some edible fats.

W. RUCZICKA (*Z. anal. Chem.*, 1930, **81**, 377—379).—The acid value of a fat may be determined by measuring the amount of iodine liberated in the presence of potassium iodide and iodate. The determination can be made after the saponification value has been determined as follows: 2 g. of fat are saponified with 25 c.c. of alcoholic 0.5*N*-potassium hydroxide and the saponification value is determined by titration with 0.5*N*-hydrochloric acid as usual. Further acid is added to neutralise exactly all the alkali and to liberate the fatty acids. The solution is diluted to 100 c.c. with alcohol and, after settling, 25 c.c. are shaken with 200 c.c. of water, 10 c.c. of 10% potassium iodide solution, and 10 c.c. of 3% potassium iodate solution; finally a little starch and an excess of at least 10 c.c. of 0.1*N*-thiosulphate are added and the excess is titrated, after 30 min., with 0.1*N*-iodine. A blank test is made in the same way, and the corrected result, expressed as c.c. of 0.1*N*-iodine is called the "iodometric acid value" of the fat. For butter this varies from 10 to 11, for lard it is about 4, for cacao butter 14.1, and for palm-kernel fat 12.9.

A. R. POWELL.

Determination of the abrasive constituents in consistent fats. E. PYHÄLÄ (*Petroleum*, 1930, **36**, *Motorentrieb*, **3**, 3—4).—Although consistent fats should be free from mineral abrasive constituents, the commercial products may contain chalk, sand, etc., often in a form difficult of detection. The following procedure is suggested for examining such materials: the fat (20 g.) is shaken with light petroleum in a separating funnel and any solid mineral matter which separates is removed, washed with light petroleum, transferred to a filter, dried, and weighed. The petroleum washings are returned to the funnel and the whole is filtered. The filtrate contains the mineral oil and the unsaponified fats. The latter are saponified and removed by refluxing with sodium hydroxide, and the mineral oil is isolated by evaporation of the light petroleum. The fatty acids which pass into the alkaline solution are determined in the usual way. The residual soaps left after the petroleum treatment are extracted with warm 96% alcohol, the soluble calcium soaps being thereby removed; the residue is suspended in light petroleum and treated with warm dilute hydrochloric acid. There are obtained an aqueous layer containing a fine deposit of mineral matter, and a light petroleum layer containing the fatty acids. The mineral matter is collected on a filter, washed, dried, and weighed, and the fatty acids, corresponding with the calcium soaps insoluble in alcohol, are determined in the usual manner. A "cup grease" examined as described contained 0.2% of free chalk and sand, and 3.8% of a grey-brown mineral sludge, probably either kieselguhr or silica. A. B. MANNING.

Analysis of soap containing water-glass. K. SCHERINGA and J. W. AHLRICHS (*Pharm. Weekblad*, 1930, **67**, 1070—1072).—The quantity of silica in soap containing sodium silicate may conveniently be determined by gently igniting the material until inflammable gases cease to be evolved and determining the silica in the usual manner. With a high silicate content separation with alcohol is unnecessary. An apparently normal separation with ether from the acidified soap solution

is not proof that silica is absent, since colloidal silica solutions are readily formed. H. F. GILLBE.

Determination of the hardness of soaps. B. TIUTIUNNIKOV (*Masloboino Zhir. Delo*, 1929, No. 8, 33—36).—The hardness, determined by measurement of the depth of the impression caused by a graduated conical wedge (300 g.) falling through 20 cm., is expressed in absolute units.

CHEMICAL ABSTRACTS.

Lathering power of soap solutions. B. TIUTIUNNIKOV and N. KASYANOV (*Masloboino Zhir. Delo*, 1930, No. 2, 40—44).—Maxima were observed when the concentration of fatty acids was 0.2—0.5% or 0.8—0.9%; variation in temperature has little effect. Comparative observations were made with various soaps.

CHEMICAL ABSTRACTS.

Extraction of soya-bean oil with alcohol. II.

(i) **Solubility of soya-bean oil in alcohol.** M. SATO and H. SAKAI. (ii) **Extraction of soya-bean oil with alcohol.** M. SATO and M. YOKOCHI. (iii) **Separation and recovery of phosphatides and carbohydrates from alcoholic extract of soya beans.** M. SATO and I. SETO (*Rep. Lab. S. Manchuria Rly.*, 1929, 1—3, 3—6, 6—7).—(i) The optimum concentration of alcohol for the extraction is 92 wt.-% or over and the temperature should be near the critical solution temperature. After extraction, cooling to 30° gives the most efficient separation of oil.

(ii) Further study showed that extraction is best carried out with 96 vol.-% of alcohol at temperatures above 75°, the extract being cooled to 25° to separate the oil. An apparatus for large-scale operation is described.

(iii) On cooling, the alcoholic layer contains 1.07% P, 1.27% N, and 27.53% (as invert sugar) of carbohydrates, the total representing about 6% of the soya bean. As methods of recovery, alcoholic extraction, alcoholic distillation, and coagulation with sulphuric acid and with calcium chloride were tried. Distillation with added alcohol gave the best recovery (carbohydrates 91.5%, phosphatides 96.8%), but coagulation by calcium chloride was probably the most practical (carbohydrates 82.7%, phosphatides 90.8%).

H. J. DOWDEN.

Denaturation of the protein in soya beans during the extraction of oil with alcohol.

I. V. K. OKANO and M. NINOMIYA (*Rep. Lab. S. Manchuria Rly.*, 1929, 7—9).—The protein matter is denatured by the action of alcohol, heat, and steam, the last-named being blown into the grains to recover the solvent. The relative proportions of protein, soluble in water, in 10% sodium chloride solution, and in 0.2% caustic soda, contained in soya bean after extraction with alcohol benzene, or alcohol-benzene mixture have been determined. It is shown that the water- and brine-soluble fractions decrease, whilst the alkali-soluble and the insoluble fractions increase. The change produced by steaming is similar and is proportional to the duration and the temperature of the steaming. No change in the amino-acid partitions of the raw bean and the grist could be detected, and digestion of the purified protein with pepsin and trypsin revealed no significant differences in the various preparations.

H. J. DOWDEN.

Occurrence of vitamins-A and -B in soya-bean oil and cake. S. OHTOMO (Rep. Lab. S. Manchuria Rly., 1929, 13—15).—Soya-bean oil has been shown to contain a definite amount of vitamin-A, although it is less effective than cod-liver oil. Oil obtained by extraction was richer than the expressed oil, and also the vitamin is destroyed by bleaching. Soya-bean cake does not contain any appreciable amount of vitamin-A, but does contain vitamin-B. H. J. DOWDEN.

Linseed. S. LEONOV and A. SHCHEPETILNIKOVA (Masloboino Zhir. Delo, 1930, No. 2, 51—57).—The seed contains about 40% of oil having iodine value 178—190.2, n_D^{20} 1.4828—1.4838, d_4^{20} 0.931—0.934; the yield of oil is little affected by variation in soil conditions.

CHEMICAL ABSTRACTS.

[Analyses. of] alimentary cottonseed oils. A. PERDRIGÉAT (J. Pharm. Chim., 1930, [viii], 12, 307—312).—The bulk of the cottonseed oil used in France is now of Egyptian origin, instead of American as formerly, and in consequence samples have been found to exhibit characteristics differing from those usually recorded for good-quality oils. Samples of Egyptian oil, refined at source or in Holland, gave values for n_D^{20} of 1.4710—1.4711 (corresponding to oleorefractometric deviations of 14.0—14.4°) instead of the usual value of 1.4717—1.4732 (oleorefractometric deviation 17—23°). The iodine value of 104.0—105.3 approached very closely the lower limit of the normal range (103—117). No trace of adulteration was found and differences in methods of refining had no significant influence.

H. J. DOWDEN.

Seeds of *Carthamus tinctorius*, L. N. BELIAIEV (Masloboino Zhir. Delo, 1930, No. 2, 16—18).—Two specimens of safflower seeds contained, respectively, moisture 4.60, 5.00; oil 15.35, 11.95; total nitrogen 2.25, 1.71; crude protein 14.08, 10.69; ash 3.29, 2.09%; the oil had d_4^{25} 0.9210, 0.9184, butyrefractometer reading (Zeiss) 77.0, 75.0, saponif. value 189.53, 192.77, iodine value (Hübl) 142.40, 142.69.

CHEMICAL ABSTRACTS.

Thermal treatment of castor oil. M. V. BORODULIN (Trans. State Inst. Appl. Chem., U.S.S.R., 1929, No. 300, 11—24).—The oil is rendered soluble in benzene when heated for 90 min. at 300°; the loss in weight is 19%. The characteristics of oil so treated, or heated for 4 hrs. in an open vessel at 270—280°, are recorded. The increase in acidity is only a secondary factor; the product possesses an ester character. It is considered that fission with elimination of heptaldehyde and then of water takes place, followed by oxidation or fission with formation of free carboxyl groups. Oxidation with formation of hydroxyl groups is then followed by condensation of the non-volatile reaction products. The lubricating properties of the product are improved by partial saponification.

CHEMICAL ABSTRACTS.

Oil viscosimeter. SCHAFFER.—See I. **Oxidizability of hydrocarbon oils and fats.** BÜRSTENBINDER.—See II. **Fat in buttermilk.** PETERSEN and HERREID. **[Oil in] embryo of soya beans.** Soya-bean cake. OHTOMO. **Soy sauce.** YOSHINO and SEKI.—See XIX.

PATENTS.

Obtaining oils and fats of pale colour and of low acid content from fish, flesh, or offal, and producing light-coloured fish, flesh, or offal meals. K. F. WILHELM (B.P. 335,438, 14.11.29. Ger., 19.10.29).—After dehydrating the wet disintegrated material by treatment with dry acetone (at about 50°), the wet acetone produced is drained off and the fat extracted by further treatment with fresh dry acetone.

E. LEWKOWITSCH.

Bleaching compositions (B.P. 334,531).—See VI. **Resins** (B.P. 335,292).—See XIII. **Artificial cream** (B.P. 334,863).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Chalking" [of paints]. II. **Uses of the "stamping process" for testing chalking.** R. KEMPF (Farben-Ztg., 1930, 36, 171—172).—Instances of the application of the method described earlier (cf. B., 1930, 156) are detailed and illustrated photographically. The effects on chalking of the presence or absence of sunlight, the "washing off" action of rain, the revelation of fine cracking and its distinction from wrinkling by the present process, and the relation between pigment concentration and chalking of water paints as indicating their relative "washability" and the binding power of organic hydrophilic colloids are all discussed.

S. S. WOOLF.

Microscopy of the iron oxide pigments. H. WAGNER (Z. angew. Chem., 1930, 43, 861—864).—A continuation of previous work (cf. Wagner and Kesseling, B., 1928, 680) on the correlation of the properties of pigments and the plastic properties of the pastes produced when pigment (especially natural iron oxides) and media are mixed. Microscopical observations, X-ray examination of lattice structure, and adsorptive power of the pigments for certain dyes are correlated with the plastic properties of the pigments in paste form. It is concluded that the pigment properties, as exhibited by those of the paste, are influenced by the ultimate crystal structure, the shape and form of the particles, and the presence of impurities such as silicates (clay), quartz, and calcium compounds.

J. O. CUTTER.

Solvents and non-solvents for nitrocellulose. F. ZIMMER (Farben-Ztg., 1930, 36, 70—71).—A brief account of solvents (esters, ketones, and ethers) and non-solvents (hydrocarbons and alcohols) for nitrocellulose is given, and the process of dissolution is discussed from theoretical considerations. S. S. WOOLF.

Resins. VII. E. STOCK (Farben-Ztg., 1930, 36, 69—70; cf. B., 1930, 779).—The analytical constants, solubilities, and capillary and fluorescence characteristics of samples of fossil resin, camphor resin, and Siam dammar are detailed.

S. S. WOOLF.

Physical properties of synthetic resins in lacquers. A. W. VAN HEUCKEROOTH (Amer. Paint & Varnish Manufs.' Assoc., Sept., 1930, Circ. 369, 354—417).—The following tests were carried out on 54 synthetic resins, representing all the types of "lacquer resins" at present available on the American market:—acid value, softening point, solubilities, colour and

viscosity of 50% solution in toluene, miscibility with cellulose esters, and durability, hardness, abrasion-resistance, tensile strength, elongation, permeability, and yellowing of films of lacquers containing the resin. The details of the tests are summarised and the results tabulated. S. S. WOOLF.

Condensation of formaldehyde with the terpenes of Siberian fir oil. S. N. USHAKOV and A. D. SOKOLOV (J. Appl. Chem., Russia, 1930, 3, 47—67).—Bornyl and isobornyl chlorides condense with formaldehyde in presence of ferric chloride, affording black resinous products which are soluble in benzene, ether, and petroleum, whilst the product obtained in presence of sulphuric acid is soluble in the first solvent only. Condensation of *l*-borneol with formaldehyde yields methylene glycol di-*l*-bornyl ether, oily substances, or resinous products according as small (heating) or large (heating) amounts of concentrated acids or large (cold) amounts of concentrated sulphuric acid are present. Condensation of the oil or its fractions in presence of sulphuric acid affords pitches. CHEMICAL ABSTRACTS.

Mineral-black filler for rubber. SCHIDROWITZ and PHILPOTT.—See XIV.

PATENTS.

[Bactericidal] paints. H. WADE. From LACKWERKE "JAPONIKA" GES.M.B.H. (B.P. 335,242, 19.6.29).—The use in such paints of phenolic compounds other than carbolic and cresylic acids, *e.g.*, thymol or naphthols, is claimed. The proportion of disinfectant should amount to 20% on the dried film. S. S. WOOLF.

Ink [for intaglio printing]. E. WINSHIP (B.P. 334,370, 23.8.29).—A mixture of a syrup of an asphaltic base, *e.g.*, gilsonite in a hydrocarbon solvent, with a substantial amount of water is homogenised by a suitable agent, *e.g.*, glue, and pigmented. S. S. WOOLF.

[Ink for lithographic] printing. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,074, 4.10.29).—Printing inks suitable for lithographic machines having a water-wiping mechanism consist of lacquers of cellulose esters or ethers, all the constituents of which lacquers repel water. S. S. WOOLF.

Manufacture of hard-rubber varnish and articles made therefrom. W. A. GIBBONS and T. V. BINMORE, ASSRS. to MECHANICAL RUBBER Co. (U.S.P. 1,745,533, 4.2.30. Appl., 17.2.25).—Sulphur is added to an approx. 10% solution of vulcanised rubber in an aromatic solvent, *e.g.*, high-flash or solvent naphtha, and the mixture is heated at atmospheric pressure until a free-flowing varnish of suitable concentration and "body," substantially free from uncombined sulphur, is obtained. The quantity of sulphur added is such as to produce a vulcanised rubber containing 15—32% of combined sulphur. S. S. WOOLF.

Lacquer. I. W. HUMPHREY, ASSR. to HERCULES POWDER Co. (U.S.P. 1,746,895, 11.2.30. Appl., 10.6.26).—The use of chemically untreated pine oil (in conjunction with the oxidised product, if desired) in nitro-cellulose lacquers is claimed. S. S. WOOLF.

Manufacture of solutions applicable as lacquers. I. G. FARBENIND. A.-G. (B.P. 318,549, 3.9.29. Ger.,

5.9.28).—Solutions containing polymerised vinyl esters or halides and phenol-aldehyde condensation products are claimed. S. S. WOOLF.

Solvents for and compositions containing cellulose esters. CELLULOID CORP. (B.P. 314,087, 21.6.29. U.S., 23.6.28).—Cellulose ester solvent mixtures containing a lower alkyl acetate in preponderating proportions, a lower aliphatic alcohol, and water are claimed. A preferred composition contains (pts. by wt.) ethyl acetate 82.5, ethyl alcohol 8.5, water 9. S. S. WOOLF.

Manufacture of aniline-sulphur [thermoplastic] resin. R. S. BLY, ASSR. to CUTLER-HAMMER, INC. (U.S.P. 1,761,291, 3.6.30. Appl., 29.6.25).—The easily controllable reaction between aniline (930 pts.) and sulphur (576 pts.) at the b.p. and in the presence of powdered nickel, iodine, or tungstic acid yields a thermoplastic resin, the m.p. of which depends on the period of heating and the proportion of sulphur. When hot-moulded, the resin has a glossy surface. E. LEWKOWITSCH.

Manufacture of resins primarily obtained from polyhydric alcohols and polybasic acids. IMPERIAL CHEM. INDUSTRIES, LTD., W. BAIRD, and E. E. WALKER (B.P. 335,292, 3.7.29).—Resinous compositions of the "glyptal" type containing hydroxylated fatty oils, *e.g.*, castor oil, as vehicles for the introduction of non-hydroxylated fatty oils, especially drying oils, are claimed. S. S. WOOLF.

Production of (A) phenol-aldehyde resins, (B) discs or sheets for gramophone records and the like. CHEMIE & TECHNIK J.M.S., GES.M.B.H., and H. MENZ (B.P. 335,194 and Addn. B.P. 335,219, [A] 18.6.29, [B] 19.6.29).—(A) Pure carbolic acid mixed with powdered sodium carbonate and bicarbonate is boiled with formaldehyde and to the mixture acetic acid is added. If coloured plastics are desired, a portion of the mass is treated with bone oil or glycerin (before the acetic acid treatment) and colouring matter is stirred in. This coloured material is then mixed with the normally treated remainder. (B) Shavings, chips, etc. of the material described in (A) are mixed with the powder produced by grinding old record-discs containing shellac and vulcanite, the mixture is boiled in bone oil at 80—100° with the addition of alcohol, and the resulting composition is suitably moulded. S. S. WOOLF.

Synthetic resin products. BAKELITE, LTD., J. W. CRUMP, and A. LLOYD (B.P. 335,173, 13.6.29).—Resinous condensation products of the urea-phenol-formaldehyde type are prepared by melting the three constituents together without the aid of aqueous solution, and heating to complete the simultaneous condensation. The proportion of the urea constituent is considerably greater than that of the phenol, and the reaction mixture is maintained neutral or alkaline. Derivatives or homologues of urea and of phenol may be employed, and a solid polymeride of formaldehyde is suitable. S. S. WOOLF.

Manufacture of condensation products from formaldehyde, thiourea, and urea. SOC. CHEM. IND. IN BASLE (B.P. 313,615, 14.6.29. Switz., 15.6.28. Addn. to B.P. 275,995; B., 1929, 219).—The solution obtained by

the prior process is concentrated and worked up only after the binding of at least the greater part of the free formaldehyde, this binding being brought about by allowing the solution to remain at approx. neutral reaction and at normal or slightly raised temperatures.

S. S. WOOLF.

Manufacture of resistant masses, lacquer coatings, films, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,961, 15.3.29. Cf. B.P. 333,872, B., 1930, 1015).—The products obtained according to the prior process are subjected to a suitable hardening process.

S. S. WOOLF.

Manufacture of artificial masses. I. G. FARBENIND A.-G. (B.P. 313,569, 13.6.29. Ger., 14.6.28).—A pure vinyl halide is polymerised under pressure at a gradually rising temperature not exceeding 100°, with or without the addition of an organic solvent, *e.g.*, ethyl chloride or vinyl acetate, which may or may not be polymerisable. The reaction may take place under the influence of chemically active rays, and in the presence or absence of catalysts, *e.g.*, organic or inorganic peroxides.

S. S. WOOLF.

Sealing compositions. [Sealing-wax substitutes.] J. LESIUK (B.P. 334,936, 12.6.29).—The compositions, which do not require heating, comprise a gum, paper or similar fibrous material, and a solution of a cellulose compound (ester) in a volatile solvent; sugars, resins, and pigments may also be added. L. A. COLES.

Coating surfaces with cellulose varnishes. E. RICHTER and W. BECKER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,778,813, 21.10.30. Appl., 11.9.28. Ger., 24.8.27).—See B.P. 317,987; B., 1929, 863.

[Resinous] condensation products obtainable from unsaturated higher fatty acids and phenols. A. THAUSS, A. DOSER, and G. MAUTHE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,779,345, 21.10.30. Appl., 1.2.28. Ger., 8.2.27).—See B.P. 310,562; B., 1929, 636.

Composition comprising resin esters. H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,757,104, 6.5.30. Appl., 14.6.29).—See B.P. 327,095; B., 1930, 570.

Manufacture of porous, homogeneous, cloudy, artificial compositions. K. RIPPER, Assr. to KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (U.S.P. 1,779,047, 21.10.30. Appl., 8.11.26. Austr., 14.11.25).—See B.P. 261,409; B., 1928, 531.

Spraying or squirting process and apparatus therefor [for paint etc.]. A. KRAUTZBERGER (B.P. 336,494, 7.12.29. Ger., 10.1.29).

Printing on surfaces coated with nitrocellulose lacquer. Surface ornamentation of metal, wood, etc., particularly for imparting thereto the appearance of rare woods. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 318,554 and 319,006, 5.9.29. Ger., [A] 5.9.28, [B] 15.9.28).

Coating [small objects with] finely-divided and like [edible] material. H. F. and E. C. MAYNES, Assees. of E. A. ZEBULSKE (B.P. 310,855, 1.5.29. U.S., 1.5.28).

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Micro-manipulations with [rubber] latex in the dark field. E. A. HAUSER (Kolloid-Z., 1930, 53, 78—82).—Recent advances in the ultramicroscopical examination of latex are discussed and the conclusions reached are summarised. The particles are enveloped in an elastic sheath of proteins and resins, the thickness of which varies with the age of the tree. E. S. HEDGES.

Processes in the mechanical deformation of rubber. H. MARK and E. VALKÓ (Kautschuk, 1930, 6, 210—215).—The different degree of extensibility of raw rubber and soft vulcanised rubber, the latter with or without the presence of carbon black, is also evident at the temperature of liquid air, although the actual extension at this temperature is naturally of small magnitude. The degree of extension of any sample before freezing is substantially without influence on its ultimate strength; the breaking strength at 195° increases proportionately to the degree of extension before freezing and inversely to the reduction in cross-sectional area. Stretching before freezing can be regarded as yielding an amorphous rubber matrix reinforced by the rubber crystallites, which act as a highly active filler; the strength of a rubber test-piece, however, represents the sum of the breaking strength of the amorphous material and the crystalline material, the latter being about ten times as strong as the former. The earlier observation that the rate of crystallite formation is much less on stretching between 0 and 100% than at higher extensions is confirmed by very sensitive examination of the crystalline interferences, although the rate of increase of strength in the frozen stretched material is comparable over both ranges of elongation. The line of demarcation between the great extensibility of rubber as observed at ordinary temperatures and the low extension at the temperature of liquid air is fairly sharp, the stress-strain curve becoming suddenly shortened near -67°. Between -20° and -40° a remarkable feature is the slow recovery after being stretched, rapid retraction being inducible by warming. Above -66° the rubber strips become turbid when stretched and on retraction recover their transparency. Below -66°, however, the necessary extending force is greater than the intermicellar adhesion and the stretched material develops fissures which persist after warming.

D. F. TWISS.

Mineral black as a reinforcing filler [for rubber]. P. SCHIDROWITZ and M. PHILPOTT (Kautschuk, 1930, 6, 197—199, 204—207; Trans. Inst. Rubber. Ind., 1930, 6, 96—116).—Mineral black or Bideford black has been used for many years in paints and imparts good ageing qualities. The carbonaceous content can be increased by a flotation process, using, *e.g.*, creosote oil as frothing agent. The dried refined material contains up to 80% C, the remainder being finely-divided clay and silica. Using a product (*d* 1.75—1.80, acetone extract 0.38%) containing 65% C as a compounding ingredient for rubber, the results are as good as with a product containing 70% C. It exercises a reinforcing effect approx. 40% of that of carbon black and apparently somewhat greater than that of lamp black. It does not retard vulcanisation, and the ageing qualities of mixtures containing it

are good. Comparative tests indicate that 100% of lamp black and other "soft" blacks and 25% of carbon black in mixtures for rubber tyres can be satisfactorily replaced by mineral black. D. F. TWISS.

Kinetics of the vulcanisation of rubber. B. W. NORDLANDER (J. Physical Chem., 1930, 34, 1873—1902).—The mechanism of the vulcanisation of rubber has been considered in relation to the active form, S_μ , of sulphur. Up to 170° the vulcanisation of crêpe rubber is directly proportional to the equilibrium content of S_μ . The forms $S\lambda$ and $S\pi$ appear to be without effect on the rate of vulcanisation. The effect of accelerators is discussed and the accelerated reaction is considered to be one of zero order. Using the idea of active molecules of S_μ , the rate of vulcanisation as affected by heat and varying amounts of accelerators is quantitatively described. In the cold-vulcanisation process the active agent also appears to be S_μ . L. S. THEOBALD.

PATENTS.

Manufacture of organic materials such as rubber. LIVERPOOL RUBBER Co., LTD., and G. THORNE (B.P. 335,612, 28.6.29).—A surface covered with a layer of rubber latex, after coagulation of the latter, is introduced into an aqueous liquid containing on its surface irregular streams of coloured solutions, e.g., of rubber in petroleum spirit. Coloured ornamentation of a "jazz" type is thereby imparted to the rubber surface. D. F. TWISS.

Manufacture of mixed artificial rubbers. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,616, 29.6.29).—A mixture of a diolefine hydrocarbon, polymerised beyond the dimeric stage, with one or more other diolefine hydrocarbons is submitted to further polymerisation by the same or another process. There are thus obtained homogeneous, mixed, polymerisation products which, on vulcanisation, particularly after compounding with carbon black, yield valuable vulcanisation products. D. F. TWISS.

Preparation of rubber compositions. L. MEILERSH-JACKSON. From NAUGATUCK CHEM. Co. (B.P. 335,597, 20.6.29).—A water-in-oil emulsion containing rubber is prepared by kneading well-masticated rubber (100 pts.) in an emulsion of water (100 pts.) in spindle oil (15 pts.) containing stearic acid (5 pts.) for 2—3 hrs. On treating the resulting sticky doughy mass with sufficient alkali hydroxide to combine with the stearic acid, the dispersion reverses its type and changes rapidly into a smooth oil-in-water emulsion resembling concentrated latex. D. F. TWISS.

Manufacture of rubber articles. SOC. ITAL. PIRELLI (B.P. 335,621, 27.6.29. Addn. to B.P. 292,964; B., 1930, 205).—When ordinary latex containing up to about 38% of rubber is used in the prior process, it is advantageous to subject the latex to a period of moderate heating, e.g., between 40° and 60° for $\frac{1}{2}$ —2 hrs., and then to cool the product, prior to introduction of the reagents which promote coagulation. D. F. TWISS.

Manufacture of [plastic] tyres for vehicles. I. G. FARBENIND. A.-G. (B.P. 334,958, 15.6.29. Addn. to B.P. 299,037; B., 1930, 71).—As raw material the products obtained from butadiene by polymerisation

(a) in the presence of latex rich in resins, e.g., latex from *Euphorbiaceae*, (b) according to the process of B.P. 313,188 (B., 1929, 652), or (c) of mixtures of any plastic or resilient polymerisation product of butadiene with a natural or an artificial resin, are claimed. D. F. TWISS.

Art of preserving rubber. B. F. GOODRICH Co., Assees. of W. L. SEMON (B.P. 317,786, 12.8.29. U.S., 22.8.28).—Rubber is treated with a small proportion of the resinous condensation product of an aldehyde with a secondary arylamine or a primary-secondary amine. Examples are formaldehyde-*s*-diphenyl-*p*-phenylenediamine, formaldehyde-phenyl- β -naphthylamine, aldol-*p*-aminodiphenylamine. D. F. TWISS.

Electrodeposition of rubber. W. A. WILLIAMS (U.S.P. 1,778,841, 21.10.30. Appl., 12.5.27. U.K., 7.2.27).—See B.P. 289,965; B., 1928, 494.

Vulcanisation of caoutchouc and product derived therefrom. L. B. SEBRELL, Asst. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,779,375. 21.10.30. Appl., 13.9.27).—See B.P. 297,051; B., 1929, 949.

Combining [uncured] rubber with solid substances [e.g., bakelite]. B. O. and M. WARWICK (WARWICK'S TIME STAMP Co.) (B.P. 336,292, 12.7.29).

Electric cables (B.P. 334,840).—See XI. **Hard-rubber varnish** (U.S.P. 1,745,533).—See XIII. **Mulches** (B.P. 319,783).—See XVI.

XV.—LEATHER; GLUE.

Determination of small amounts of tannins. J. G. OBERHARD and A. V. SCHALBEROVA (Pharm. Ztg., 1930, 75, 970—971).—The method is based on the colour produced by tannin extracts in the presence of ferruginous albumin, and is applicable to materials containing large amounts of sodium chloride such as salted foodstuffs contaminated by storage in wooden barrels etc. Filter paper is immersed in warm 1% gelatin solution and, after drying, it is dipped in dilute ferric chloride solution (1% Fe), dried in the dark, and cut into strips 8 mm. wide. A standard solution of tannic acid is prepared and a series of 12 test tubes (diam. 15 mm.) is prepared, each tube containing 10 c.c. of the solution at dilutions ranging from 0.1 to 0.001% of tannic acid. The filtered extract to be tested is disposed in similar tubes at normal concentration and diluted 1:5, 1:10, etc. Test strips are immersed in each of the liquids for 1 hr. at room temperature in a shaded position and the colours produced by the extract and by the standards are compared. H. J. DOWDEN.

Determination of insoluble matter in tanning extracts. C. VAN DER HOEVEN (Collegium, 1930, 380—388).—Sedimentation was effected in a cylindrical vessel, 9 cm. in diam. and 4.5 cm. high, through the wall of which at 1.5 cm. from the bottom was fitted a glass tube with its inside end turned up and provided outside with a stopcock. The results obtained on different extracts for the insoluble matter varied very little for variations of the depth of liquor above the tube of 0.7—2.0 cm. Lower results for the insoluble matter in quebracho extracts were obtained by sedimentation than by the Berkefeld filter-candle method, but the concordance, though poor, was about the same.

The insoluble matter was also determined by centrifuging in glass cylinders of 50-c.c. capacity for the period of time required for particles $\rightarrow 1\mu$ to settle out. The results obtained by this method were slightly lower than those obtained by sedimentation, but on increasing the time of centrifuging higher figures were obtained. The degree of concordance was poor by all methods for quebracho extracts. The amount of clarified liquor pipetted after centrifuging is so small as to give rise to large errors. The amount of insoluble matter in a mangrove extract was 7 times as great in a solution containing 36 g./litre as in one containing 12 g./litre.

D. WOODROFFE.

PATENTS.

Preparation of enzymic products [for treatment of hides]. C. J. M. M. LE PETIT, Assr. to ROHM & HAAS Co. (U.S.P. 1,779,243, 21.10.30. Appl., 23.10.28. Fr., 16.11.27).—See B.P. 300,615; B., 1930, 473.

Auxiliary means for leather dressing. B. REWALD (U.S.P. 1,779,012, 21.10.30. Appl., 12.1.28. Ger., 16.7.27).—See B.P. 306,672; B., 1929, 369.

XVI.—AGRICULTURE.

Chemical changes in the organic matter [of soils] during the natural decomposition of the humus layers of woodlands. I. Variation in pentosan content. A. NĚMEC (Z. Pflanz. Düng., 1930, 18A, 65—104).—The decomposition of pine, fir, and larch needles does not commence until after that of the cell-wall substance. In general, the pentosan content (ash-free basis) decreases as humification proceeds, although this relationship is less marked in soils under deciduous trees. Humification precedes pentosan decomposition. Matter soluble in benzene-alcohol is decomposed at a relatively greater rate than the total organic matter, and is greater in pine than in fir soils in a similar stage of humification. The high proportion of resin in the needles tends to inhibit the normal humification process. The iodine consumption of benzene-alcohol extracts varies directly with the amount of material extracted. The nitrifying power of these soils is closely allied to the amount of benzene-alcohol-soluble matter present, the latter becoming toxic when present in quantities exceeding 5% of the ash-free dry matter.

A. G. POLLARD.

Indirect determination of various soil characteristics by the hydrometer method. G. J. BOUYROUCOS (Soil Sci., 1930, 30, 267—272).—The "total colloids" of soils as determined by the hydrometer method (cf. B., 1927, 422, 454) at the end of 15 min. is closely related to the moisture equivalent and unfree water content. These values together with the wilting point may be determined indirectly by this method.

A. G. POLLARD.

Application of filtration analysis in the investigation of factors determining the dispersity of soils. I. N. ANTIPOV-KARATAIEV (Kolloidchem. Beih., 1930, 31, 374—417).—A critical review of work on the velocity of filtration through different soils leads to the view that the principal factors concerned are the degree of dispersion of the soil, the coagulating and peptising effects of the electrolytes present, and the nature of the adsorbed bases. The velocity of filtration of water

through different soils of varying particle size and in the presence of various cations has been measured. With increasing content of particles of colloidal dimensions the velocity of filtration decreases almost linearly. The filtration velocity also depends on the adsorbed cations, increasing in the ratio $\text{Na}^+ : \text{NH}_4^+ : \text{Mg}^{++} : \text{Ca}^{++} = 1 : 6 : 33 : 46$. Numerous experiments were carried out on the effect of base exchange on the filtration velocity.

E. S. HEDGES.

Determination of carbonates in soil. C. J. SCHOLLENBERGER (Soil Sci., 1930, 30, 307—324).—Apparatus is described in which carbonates in soils are decomposed by boiling with hydrochloric acid containing ferrous chloride at low pressures and below 30°. Ferrous chloride serves to prevent the production of carbon dioxide by oxidation of organic matter, e.g., in soils containing manganese dioxide.

A. G. POLLARD.

Carbon-nitrogen relationships in soils. W. R. LEIGHTY and E. C. SHOREY (Soil Sci., 1930, 30, 257—266).—The C:N ratios for numerous soil types are recorded. Values obtained vary widely with the nature of the soil, but those for surface soils are generally higher than for corresponding subsoils. Organic matter in soils cannot be determined as total N \times 20. The C:N ratio of soils may be expected to indicate the availability of organic matter to soil organisms.

A. G. POLLARD.

Rate of loss of replaceable potassium [from soils] by leaching. O. C. MAGISTAD (Soil Sci., 1930, 30, 243—256).—Leaching experiments with artificial zeolites and with soils show that the rate at which potassium is leached may be expressed mathematically. Constants in the leaching equation were fairly uniform for non-calcareous soils, whilst those for calcareous soils were consistent with lower leaching rates. For synthetic zeolites intermediate values were obtained.

A. G. POLLARD.

Proportions of easily and difficultly mobilisable acid within the zone of exchange acidity in soils, and the bearing of buffer values on this. S. GOY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1930, 18A, 104—114; cf. B., 1930, 833).—Differences in the exchange-acidity values of soils as determined by Daikuhara's method and by electrometric titration are attributed to the varying proportions of "easily" and "difficultly" soluble acids present. The latter are not necessarily recorded by Daikuhara's process. Electrometric titration curves are adapted to show base-fixing areas and areas characteristic of easily and difficultly soluble acids. In humus soils there is a greater proportion of difficultly soluble acids concerned in producing exchange acidity than is the case with mineral soils. Buffer values of soil are calculated from electrometric titration curves (in *N*-potassium chloride) over the range p_H 6.8—7.2. Determination of exchange acidity in soils by this means is limited to mineral soils with less than a prescribed buffer value.

A. G. POLLARD.

"Negative values" in Neubauer-Schneider tests of soils. M. GRAČANIN (Z. Pflanz. Düng., 1930, 18A, 115—118).—Negative values for phosphate assimilation in Neubauer tests are ascribed to the transference

of phosphatic material from the germinating seeds to the soil and its subsequent fixation by the soil. The phenomena is characteristic of highly adsorptive soils deficient in phosphate. A. G. POLLARD.

Use of superphosphate, "ammonium superphosphate," and "potash ammonium superphosphate." GERLACH (Superphosphat, 1930, 6, 135—137).—Superphosphate may be used on light sandy soils without risk of appreciable losses by leaching, and the conjoint use of lime or marl does not reduce its efficiency. A. G. POLLARD.

Intake of phosphoric acid by plants. R. RUDEL and KLEBERGER (Superphosphat, 1930, 6, 163—166).—The assimilation of phosphate by barley plants from superphosphate and Rhenania phosphate was more rapid than from basic slag, particularly in the early stages of growth. Differences in total yields were not great, but the lesser efficiency of basic slag was manifest in the smaller grain yield, lower phosphate content of grain, and smaller percentage utilisation of the fertiliser. In these respects Rhenania phosphate occupied an intermediate position between superphosphate and slag. A. G. POLLARD.

Intake of phosphate and potash [by plants] in Neubauer seedling experiments and the law of minimum. A. HOCK (Superphosphat, 1930, 6, 167—172).—The growth of seedlings under the conditions of Neubauer tests was examined in pure sand to which the essential nutrients were added in various combinations. The percentage utilisation of added potash was greater in all combinations than that of phosphate and was less affected by the presence of other nutrients. Growth and root development in cultures containing phosphate alone was poor. The assimilation of phosphate from potassium phosphate was greater than from sodium phosphate, but the assimilation of both potassium and phosphate was frequently less from potassium phosphate than from an equivalent mixture of sodium phosphate and potassium chloride. Nutrients applied in the lower layers of sand were more completely utilised by seedlings than when applied to the surface layers. Seedlings absorbed from potassium phosphate relatively greater proportions of potassium than phosphate and free phosphoric acid remained in the sand. Phosphate applied as monocalcium phosphate was more readily assimilated than tricalcium phosphate. The effect of potash on the assimilation of phosphate is examined in relation to the technique of Neubauer tests and the law of minimum. A. G. POLLARD.

Superphosphate and liquid manure. J. FISCHER (Superphosphat, 1930, 6, 146—147).—The addition of superphosphate to liquid manure used for pumping over grassland considerably increased the hay crops. A. G. POLLARD.

Effect of soil and of manuring on the incidence of "take-all" (*Ophiobolus graminis*) in wheat. SCHMIDT (Superphosphat, 1930, 6, 147—149).—"Take-all" in wheat appears principally in alkaline soils and develops most readily at p_H 7.49. Neither liming nor the use of potash fertilisers tends to eliminate the disease. Phosphatic fertilisers tend to minimise the injury by stimulating root development. A. G. POLLARD.

Manurial trials with sugar beet. ESSKUCHTEN (Superphosphat, 1930, 6, 138—139).—The use of basic slag and of superphosphate in conjunction with a basal nitrogen-potash dressing increased the root yield of sugar beet, but decreased the sugar content. The total sugar yield was increased only in the case of superphosphate. A. G. POLLARD.

The soya-bean question [and agriculture]. W. RIEDE and B. REWALD (Landw. Versuchs-Stat., 1930, 110, 291—304).—Analyses of various samples of soya beans are recorded and their value in German agriculture is discussed. A. G. POLLARD.

Local variation of soil acidity in relation to soya-bean inoculation. G. Z. DOOLAS (Soil Sci., 1930, 30, 273—287).—In pot experiments soya beans were grown with their roots penetrating soil zones of varying acidity. In areas with p_H 3.8—4.6 nodulation was depressed and roots were injured, but at p_H 5.6 growth and nodulation were normal. Apparently healthy plants were produced when part of the root was in soil of p_H 5.6, but part was injured in more acid soil. Depression of nodulation in acid soil results from local acid injury to the plant tissue or from abnormal nutritive conditions. Nodule organisms remain viable in soils of p_H 3.8—8.3. A. G. POLLARD.

Ash constituents of pasture grasses, their standard electrode potentials and ecological significance. H. P. COOPER (Plant Physiol., 1930, 5, 193—214).—A correlation exists between the electrode potentials of potassium, magnesium, calcium, phosphorus, nitrogen, and silica and the amounts of these elements present in the four major soil-fertility levels in New York State. The ways in which radiant energy may affect nutrition are discussed. CHEMICAL ABSTRACTS.

Petroleum oil summer sprays for pine-leaf scale control. H. H. RICHARDSON (J. Econ. Entom., 1930, 23, 753—758).—Comparison of various grades of oil sprays shows that only highly refined "white oils" (d 0.84—0.85, Saybolt viscosity 45—85 sec. at 100° F.) could be applied in toxic concentrations without leaf injury. A. G. POLLARD.

Effect of hydrogen-ion concentration on the toxicity of nicotine, pyridine, and methylpyrrolidine to mosquito larvæ. C. H. RICHARDSON and H. H. SHEPARD (J. Agric. Res., 1930, 41, 337—348).—Larvæ of the house mosquito (*Culex pipiens*) were placed in nicotine solutions of varying concentration and acidity. In solutions of 0.1M—0.001M concentrations the toxicity of the free base was much greater than that of the sulphate adjusted to p_H 5.0, and over the range p_H 2.4—9.7 (free base) toxicity increased with increased alkalinity. Addition of inorganic hydroxides did not increase the toxicity of aqueous nicotine solutions. Similar results were obtained with pyridine and methylpyrrolidine. The rate of toxicity of solutions of the three bases is proportional to the concentration of undissociated molecules. Nicotine ions are less toxic than the molecules. A. G. POLLARD.

Distribution of pasture plants in relation to soil acidity and other factors. W. R. G. ATKINS and

E. W. FENTON (Sci. Proc. Roy. Dublin Soc., 1930, 19, 533—547).

Determination of nitrates in soil. RIEHM.—See VII.

PATENTS.

Mulches or coverings for cultivated land. T. WHITTELEY (B.P. 319,783, 27.9.29. U.S., 28.9.28).—Comminuted fibrous material, *e.g.*, waste paper, bagasse, straw, sawdust, is spread over soil and treated simultaneously or subsequently with a waterproofing or binding material, *e.g.*, oils, asphalts, rubber emulsion, etc. A. G. POLLARD.

Manufacture of urea-containing fertilisers. W. W. TRIGGS. From A. B. LAMB (B.P. 335,600, 27.6.29).—The product obtained by heating ammonium carbonate, bicarbonate, or carbamate in an autoclave, and containing approx. 40% of urea, is partly or wholly neutralised with acid, *e.g.*, sulphuric, nitric, or phosphoric acid. When phosphoric acid is used the N:P ratio of the final product may be raised by distilling part of the residual ammonia in the autoclave charge prior to neutralisation. A. G. POLLARD.

Killing of weeds and preparations therefor. R. N. CHIPMAN (B.P. 315,853, 27.5.29. U.S., 20.7.28).—The weeds are dusted with a 3:2 mixture (by wt.) of sodium chlorate and calcined calcium chloride. L. A. COLES.

Preservation of flowers, foliage, etc. H. J. VALENTINE (U.S.P. 1,779,299, 21.10.30. Appl., 23.12.29. U.K., 12.9.28).—See B.P. 322,806; B., 1930, 163.

Pure monopotassium phosphate (U.S.P. 1,746,905).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Accurate and rapid determination of sugar in beet slices by means of fine pulp obtained by the Herles press. F. HERLES (Z. Zuckerind. Czechoslov., 1930, 55, 31—32).—Recent improvements in the use of the Herles press avoid errors due to evaporation of moisture from the pulp. Only sufficient of the well-mixed sample of cossettes is pressed to provide enough pulp for analysis, so that the pressing occupies but a few minutes; also the receptacle for the pulp fits in the outlet of the press to avoid evaporation. In the analysis of beets a portion of the well-mixed sample obtained by known rasping machines is passed through the press to provide fine pulp suitable for instantaneous cold aqueous digestion. J. H. LANE.

Course of the first carbonatation [of beet juices]. Solutions of sugar and lime. J. DĚDEK (Z. Zuckerind. Czechoslov., 1930, 54, 463—473).—Unaccountably variable results in the experimental study of the carbonatation of beet juices led to the present investigation on pure sugar solutions. In the carbonatation of sucrose solutions containing 0.45 and 0.71% of lime in complete solution, no calcium carbonate separated for a considerable time. Later a voluminous precipitate was formed which settled well, leaving a perfectly clear supernatant liquid; still later this gave place to a much finer precipitate which did not settle well. Throughout a large part of the carbonatation the liquid had a considerably higher alkalinity in the unfiltered than in the filtered

state, indicating the precipitation of lime with the calcium carbonate. This was most marked when the above-mentioned voluminous precipitate had been formed, the filtered solution then showing in some cases only half the alkalinity of the unfiltered liquid. Such differences may be observed in the factory if portions of juice are filtered at various stages in the first carbonatation. The alkalinity of the filtrates may rise during filtration, owing to lime redissolving from the precipitate as the juice cools. In one factory small samples of juice filtered at the end of the first carbonatation had an alkalinity of 0.061% CaO compared with 0.099% in the same juice from the filter presses, the higher value being due to the prolonged contact of the scum cakes with juice. The course of carbonatation of limed sucrose solutions was found to be influenced by the percentage of lime in the solutions, by the rapidity of gassing, and very markedly by the presence of sodium acetate, but it was not affected by addition of starch paste or gelatin, nor by the presence of washed precipitate from a previous carbonatation. J. H. LANE.

Testing [with beet syrup] the activity of decolorising carbons with the Linsbauer-Vašátko filtration apparatus. J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1930, 54, 515—529; cf. B., 1929, 5).—An investigation, carried out in 1926, on the two main methods of using vegetable carbons with beet syrup, viz., as a filtering layer packed between two filter cloths, and as a suspension to be ultimately filtered. The tests were made on a second centrifugal run-off of 65° Brix and 10.5° of colour (Stammer). Preliminary experiments by the filtration method with Standard Norit indicated that for a 62.4% decolorisation of 4.25 hectolitres of syrup, twice as much carbon is required at 60° as at 85° (1.12% on total solids in the syrup compared with 0.56%). Subsequent tests were all made at 75°. The relative amounts of Carboraffin, Supra-Norit 3X, Polycarbon, and Standard Norit required to produce equal decolorisation of a given volume of syrup, filtered at constant speed, were about 1:1.82:2.44:3.50. Carboraffin, unless previously alkalisied by lime, lowered the alkalinity of the first portions of juice. In the filtration method the occasional repacking of the filtering layer increased the decolorisation, especially in the case of very fine carbons, which are apt to become clogged by fine suspended matters from the syrup. No improvement was effected by interrupting the filtration and steaming the filtering layer without disturbing it. Interruption of filtration for long periods (24 hrs.) enhanced the decolorising action in the resumed filtration, but lowered the alkalinity of the filtrate appreciably, possibly owing to decomposition of some sucrose (cf. B., 1928, 29). In the suspension method of use, the four carbons showed activities in the same sequence as in the other method, but the rates of filtration under constant pressure were in the inverse order of the activities. The carbons were stirred with the syrup for 15 min. or longer and then filtered in the Linsbauer-Vašátko apparatus, without the upper filter-cloth, of course. In contrast with the other method, the later portions of filtrate were more decolorised than the earlier portions, but the average decolorisation for the whole syrup was the same as that attained by the other

method, when equal amounts of the same carbon were used. J. H. LANE.

Influence of rate of filtration on the decolorisation of sugar solutions by a layer of active carbon. J. VAŠÁTKO (*Z. Zuckerind. Czechoslov.*, 1930, 54, 531—536).—In the filtration of sugar juices or syrups through a layer of active carbon under factory conditions, a slow rate of filtration has no advantage in economising carbon, since it does not affect the total work of decolorisation which a given amount of carbon will perform before it is exhausted. Rapid filtration has the great advantage of minimising the filtering area required. Tests described show that even with a quantity of juice or syrup insufficient to exhaust the carbon layer, wide variations in the rate of filtration have practically no influence on the average decolorisation of the whole liquid, and very little influence on the decolorisation of the first portions unless the filtration is carried out at low temperatures. J. H. LANE.

Colorimetric determination of p_H of molasses. P. DOPFER (*Bull. Soc. Chim. biol.*, 1930, 12, 1031—1032).—The p_H of molasses may be determined colorimetrically with considerable accuracy after the molasses has been diluted 10 times with distilled water; the results obtained agree within experimental error with those found by electrometric determination. W. O. KERMACK.

Measurements [of colour] with the objective photocolourimeter [in sugar manufacture]. K. ŠANDERA (*Z. Zuckerind. Czechoslov.*, 1930, 55, 33—40).—The author has modified the construction and method of use of his photocolourimeter (*B.*, 1928, 344) in order to obtain consistent results in terms of well-known technical units. Light transmitted through the pure solvent is reduced by a graduated stop having a scale attached, until the photo-electric current is the same as that obtained under similar conditions with the unstopped solution to be tested. The scale readings of the stop are translated into Stammer colour degrees by means of a table or graph. J. H. LANE.

Apparatus for expediting sugar-factory analyses. O. SCHÄFFER (*Z. Zuckerind. Czechoslov.*, 1930, 54, 630—631).—For the dilution of massecuite with an equal weight of water the author and J. Stangl have devised an apparatus consisting of two cylindrical tinned iron receptacles of different sizes but exactly equal weights, the smaller of which fits tightly on the larger one as a lid, forming a closed vessel. The two receptacles are placed on opposite pans of a balance, any suitable quantity of massecuite is placed in the larger one, and water is introduced into the smaller one to balance. This water is then poured on the massecuite and the smaller receptacle, inverted, is fixed on the larger, whereupon the massecuite is dissolved by shaking. The same apparatus can be used in the determination of sugar in beets. The beet sample is weighed into the larger receptacle, the smaller one is fitted as lid, and the basic lead acetate solution is run in from a pipette through a small orifice in the lid which can be opened and closed at will. A type of graduated flask is described in which the lower and upper halves are firmly but detachably connected by a ground-glass surface, so that materials such as molasses and cossettes can be weighed directly into the lower portion. J. H. LANE.

The Celotex and cane-sugar industries. E. C. LATHROP (*Ind. Eng. Chem.*, 1930, 22, 449—460).—Celotex is an artificial building board made from bagasse. The bagasse from the cane mills is compressed into 250-lb. bales which are stored in covered piles. During storage the residual sugar in the bagasse rapidly undergoes alcoholic and acetic fermentation, and in the interior of the piles the fibres become softened and retted. The outer parts of the piles are specially treated to produce similar changes. The bales, halved or quartered, are then cooked under pressure in rotary digesters containing a solution which is buffered to prevent the fibres being unduly weakened by too great an increase of acidity. From the cooker the fibre passes through a tank and is pumped at a concentration of 2—3% through shredders preparatory to washing. The washed fibre is treated in suspension with the usual sizing agents, "refined" to obtain the desired proportions of fibres of different sizes, sheeted, pressed to reduce its moisture content to 50—55%, and finally dried. The material will afterwards re-absorb about 8% of moisture from the air; this "seasoning" process is accelerated by sprinkling water on the boards. The properties of Celotex boards are described. The by-products of the manufacture are pith, for use in the explosives industry, and Celotex dust, which may be used as moulding flour. J. H. LANE.

Physical chemistry of starch and bread-baking. IV. [Analogy between] conversion of starch into paste and mercerisation of cellulose. V. Identity of retrogradation spectra for all types of starch. VI. Effect of drying on X-ray spectra of starch preparations. J. R. KATZ (*Z. physikal. Chem.*, 1930, 150, 81—89, 90—99, 100—109; cf. *B.*, 1930, 1043).—IV. [With J. C. DERKSEN.] X-Ray spectrograms have been made from specimens of wheat starch made into pastes with sodium hydroxide solutions of various concentrations. With a definite minimum concentration of sodium hydroxide (about 0.08*N*) the *V*-spectrum appears, corresponding with the incipient change of β_A -starch into the α -modification. Above this concentration a rather narrow limit exists (up to about 0.104*N*) within which the relative intensity of interference of the *V*-spectrum increases with increasing concentration of sodium hydroxide. The upper limit appears to correspond with complete conversion. There is an apparent analogy between this phenomenon and the volume of supernatant liquid remaining after the swelling of starch in sodium hydroxide solution; the volume is approximately constant up to about 0.6*N*-alkali, and then rises abruptly, reaching a maximum with about 1.0*N*-alkali. After the pastes have been air-dried for some days the *V*-spectrum disappears and a typical retrograde spectrum is observed. These results are closely analogous to those observed in the mercerisation of cellulose by alkali, and it is suggested that identical, or at any rate similar, isomerism of polysaccharides occurs in the two cases.

V. [With T. B. VAN ITALLIE.] Different types of starch may be divided into three classes, according to the type of X-ray spectrum (*A*, *B*, or *C*) which they exhibit. When formed into paste with sodium hydroxide solution, however, all starches give the same spectrum, referred to as the *V* type. This corresponds with the

conversion of the starches into the α -modification. If the pastes are air-dried for considerable periods a retrograde spectrum appears which is always of the *B* type, and corresponds with the formation of the β_n -modification. This spectrum is given by potato, horse-chestnut, and *Canna indica* starches in the native state. Starches vary in the velocity with which the retrograde change from the α - into the β_n -form occurs.

VI. [With J. C. DERKSEN.] The effect of intensive and prolonged drying on the X-ray spectra of native starches and on starch pastes has been investigated. As drying progresses, the *A*, *B*, or *C* types of spectra characteristic of crystalline native starches change into indefinite spectra of the type given by amorphous substances. The ease with which this change occurs depends on the origin of the starch. The moistened starches exhibit on drying a new type of crystalline spectrum (*VT* type). The water in these specimens appears to be zeolitic in nature. F. G. TRYHORN.

Properties of kaoliang starch. I. Adsorption of iodine and chlorine by kaoliang starch granules. S. MIKI (Rep. Lab. S. Manchuria Rly., 1929, 24—26).—Starch granules from kaoliang (*Andropogon sorghum*, Broth.) were obtained by repeated agitation with 0.3% caustic soda, washing, and settling, followed by washing with alcohol, then with ether, and drying at 40°. Known amounts of starch were added to aqueous solutions of iodine and chlorine, and the absorption (x) was determined by titration with thiosulphate or arsenite solutions. The adsorption follows the law $x = kc^n$, and the curve is the combination of two such equations, the point of intersection showing a change of surface tension due to swelling. The abnormally high consumption of bleaching powder in the bleaching of kaoliang starch is mainly due to adsorption of chlorine by the granules. H. J. DOWDEN.

Thermal capacity of sugar-factory products (solutions) and of sugar crystals. V. V. YANOVSKI and P. A. ARKHANGELSKI (Zhur. Sakharn. Prom., 1929, 3, 511—517).

Immersion refractometer. DOLINEK.—See I. **Diospyros ebenum.** SHINODA.—See V. **Kaoliang starch for finishing textiles.** KAWAMATA.—See VI. **Carbohydrates from soya beans.** SATO and SETO.—See XII. **Korean koji.** NAGANISHI.—See XVIII. **Determination of lactose.** CLAVERA and MARTIN.—See XIX.

PATENTS.

Crystallisation of massecuites. R. HADDAN. From F. L. ALLEN (B.P. 335,022, 16.8.29).—The crystallisation is effected in a tank, e.g., rectangular in the upper and hemicylindrical in the lower section, provided with alternating stirring arms and cooling coils, the arms rotating and the coils being set in planes parallel to the ends of the tank. L. A. COLES.

Purification of wood sugar. HOLZHYDROLYSE A.-G. (B.P. 315,198, 8.7.29. Ger., 7.7.28).—To remove the greater part of the hydrochloric acid remaining in wood sugar after it has been dried in an atomised condition, the sugar is treated with organic solvents, e.g., with 3—4 pts. of a mixture of 95% alcohol and benzene (5:1), so that it retains its extended surface, and then is caused to sinter or form a syrupy mass, by warming

or by limited addition of water, whereupon the organic liquid, containing about 80% of the hydrochloric acid originally present, is drawn off. Sulphuric acid may be added during the treatment to ensure the removal of combined hydrochloric acid. J. H. LANE.

Production of fermentable sugars [from cellulose]. SOC. DES BREVETS ETRANGERS LEFRANC & CIE., Assees. of "LE KETOL" (B.P. 315,403, 26.6.29. Fr., 13.7.28).—Cellulosic material such as wood waste, carob beans, or beet pulp is saccharified by sulphuric acid under pressure in two stages, the sugar formed during the first stage being removed before the second stage is commenced, and the pressure during the latter stage being at least as high as during the first stage. E.g., 400 pts. of sawdust containing 50% of moisture are mixed with 400—600 pts. of water containing 10—20 pts. of sulphuric acid, and raised by injection of dry steam to pressures of 4—12 kg./cm.² for periods of 15—3 min. The liquid is then discharged and the solid residue, mixed with 400 pts. of water containing 20 pts. of sulphuric acid, is again heated to a pressure of 12 kg./cm.² for 5 min. J. H. LANE.

Etherification of carbohydrates. O. LEUCHS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,746,663, 11.2.30. Appl., 1.6.27. Ger., 1.6.26).—The carbohydrates (starch, alkali-cellulose, etc.) are treated with etherifying agents, e.g., methyl or ethyl chloride, in the presence of fused calcium chloride kept out of physical contact with the carbohydrate; e.g., the carbohydrate is supported in a perforated basket in an autoclave containing the calcium chloride at the bottom. (Cf. B.P. 302,191; B., 1929, 167.) L. A. COLES.

Filtration of liquids (B.P. 334,663).—See I. **Synthetic board** (B.P. 335,052).—See V. **Mulches** (B.P. 319,783).—See XVI. **Alcohol** (U.S.P. 1,748,791).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Osmosis and fermentation. I. N. C. BEETLESTONE (J. Inst. Brew., 1930, 36, 483—493).—The volume of a yeast cell immersed in water reaches a maximum which is constant for any one race of yeast. The rate of aqueous diffusion into and from the cell is of logarithmic character, and osmotic balance between the cell and surrounding liquid is attained in 4 hrs. at 10°. The osmotic effects of corresponding concentrations of different carbohydrates on the yeast cells are identical, and for dilute solutions of the carbohydrates and of alcohol the osmotic effects are proportional to the concentration. For normal yeast cells, 10—12% solutions of carbohydrate and 5—7% solutions of alcohol have equivalent osmotic effects. This relationship between the carbohydrate and alcohol is such that, during fermentation, the conversion of sugar into alcohol causes no alteration of the osmotic conditions. C. RANKEN.

Korean koji (a kind of so-called Chinese yeast). H. NAGANISHI (Rep. Lab. S. Manchuria Rly., 1929, 41—42).—With a view of preparing a material of higher diastatic activity, an attempt has been made to separate and identify the micro-organisms present in koji. From 18 samples were isolated 37 species of mould fungi, 9 of yeast, and 4 of bacteria. Maximum diastatic activity of the moulds was closely related to the conditions

favouring their growth, viz., temperature 40–60° and p_H 4.3–5.3. Certain of the species produced relatively large amounts of reducing sugars when incubated with starch for 4 days at 35° and p_H 5.1–5.2.

H. J. DOWDEN.

Methods of mashing potato flakes and the effect of "souring." B. LAMPE and W. KILP (Z. Spiritusind., 1930, 53, 267–268).—When the necessary proportion of malt is mashed with potato flakes in one operation, the yield of alcohol after fermentation is greater, and the consumption of steam less, than when the addition of malt is made in two portions to the flakes. These results hold only provided the mashes are vigorously stirred and the flakes are not allowed to "ball." Artificial souring of each type of mash before its liquefaction and saccharification diminishes the yield of alcohol. On the other hand, a mash with an acidity of 0.05–0.1° gives a purer fermentation.

C. RANKEN.

Evaluation of potato flakes according to their yield of alcohol. B. LAMPE (Z. Spiritusind., 1930, 53, 274–275).—Potato flakes should not be valued according to their moisture content, as there is no relationship between the latter and the yield of alcohol.

C. RANKEN.

Determination of the hydrogen-ion concentration of beer and wort. F. STOCKHAUSEN and E. F. ROTHENBACH (Z. anal. Chem., 1930, 81, 392).—The quinhydrone electrode cannot be used for determining the buffering capacity of beer and wort owing to the presence of protein (cf. B., 1928, 871). A. R. POWELL.

Preparation of absolute alcohol, D.A.B. VI. K. R. DIETRICH (Pharm. Ztg., 1930, 75, 846–848).—An apparatus for the continuous production of absolute ethyl alcohol from 80–94% spirit by Young's method is described. Dehydration and rectification are effected in a column, the product being subsequently passed over activated charcoal to remove traces of benzene and other impurities and finally refractionated. The remainder of the apparatus is concerned with the treatment with water of the fractions of lower b.p. and the recovery of the benzene and alcohol which they contain.

H. E. F. NOTTON.

Fermentation of soya beans. YOSHINO and others. —See XIX.

PATENTS.

Manufacture of alcohol. W. L. OWEN, Assr. to CITIZENS OF U.S.A. (U.S.P. 1,748,791, 25.2.30. Appl., 21.12.27).—A mixture of cane bagasse and molasses wort is fermented by yeast until the bagasse fibres are impregnated with yeast cells, and the density of the wort is reduced by one half. The fermenting mixture is then transferred into the main supply of molasses wort and the fermentation allowed to proceed according to ordinary distillery practice.

C. RANKEN.

Production of absolute alcohol. ZELLSTOFFFABR. WALDHOF, and O. LÜHRS (B.P. 335,132, 19.12.29. Ger., 10.1.29).—Dilute alcohol is distilled under pressure with calcined chalk, and the chalk particles are removed from the distillate by passing the vapour without condensation through a layer of absolute alcohol before condensation in the cooler.

C. RANKEN.

Production of aliphatic acids and other fermentation products. H. LANGWELL (B.P. 334,900, 11.6.29).—The acid which is formed during the fermentation of cellulosic materials is neutralised by ammonia, and the ammonia subsequently recovered from the crude liquid by distillation with an alkaline-earth base. After filtration the residual solution is concentrated. A multiple-effect evaporator is utilised for the two distillation stages.

C. RANKEN.

Production of citric acid by fermentation. F. KANHÄUSER, Assr. to MONTAN- U. INDUSTRIAL-WERKE VORM. J. D. STARCK (U.S.P. 1,779,001, 21.10.30. Appl., 31.8.28. Czechoslov., 16.12.27).—See B.P. 302,338; B., 1929, 1029.

Fermentable sugars (B.P. 315,403).—See XVII.

XIX.—FOODS.

Rice storage experiments. H. W. JACK and R. B. JAGOE (Malayan Agric. J., 1930, 18, 447–454).—Under rat-proof and well ventilated conditions rice may be stored in good condition for two years, but should be winnowed before use. It can also be stored in airtight containers for at least two years if fumigated with carbon disulphide (5 c.c. to 18 litres of rice). This fumigant imparts a yellow tinge to the uncooked rice, but does not affect its edibility. The vitamin-B content of rice does not deteriorate on storing. B. W. TOWN.

Composition of Lithuanian wheat. E. SARIN and O. AUSIN (Latvij. Univ. Raksti, 1929, 1, 127–138).—Analyses of 36 different samples of wheat are recorded.

M. S. BURR.

Effect of pasteurisation temperature on the physical properties of milk. W. H. E. REED (Missouri Agric. Exp. Sta. Res. Bull., 1929, No. 126, 3–12).—The viscosity, sp. gr., and surface tension are not materially affected by the use of different pasteurisation temperatures; rise of the temperature retards the separation of fat.

CHEMICAL ABSTRACTS.

Approximate analysis of 1 c.c. of milk. T. UGARTE (Rev. fac. cient. quim. La Plata, 1930, 6, 27–31).—Proteins and mineral constituents precipitated by the addition of absolute alcohol (15 c.c.) are washed with hot alcohol and dried, the ash being determined by ignition. The residue left on evaporation of the alcoholic filtrate is extracted with ether; on evaporation the ethereal solution affords the fat, and the residual lactose is freed from alcohol and weighed.

CHEMICAL ABSTRACTS.

Determination of fat in buttermilk. W. E. PETERSEN and E. O. HERREID (Minnesota Agric. Exp. Sta. Tech. Bull., 1929, No. 63, 2–16).—In the Babcock test the buttermilk (9 g.) is mixed with 10 c.c. of a reagent prepared by dissolving sodium carbonate (110 g.) and salicylate (200 g.) in water, diluting to 1 litre, and adding 50% sodium hydroxide solution (30 c.c.) and butyl alcohol (100 c.c.). The test bottles are kept at 71–82°, with occasional shaking, for 6–7 min., centrifuged at 800 r.p.m. for 5 min., and again for 2 min. after adding warm water to reach the base of the neck; again warm water is added to bring the fat into the graduated neck, and the bottles are centrifuged for 1

min. The reading ($\times 2$) is taken after 5 min. at 57–60°. The accuracy is not affected by addition of lecithin.

CHEMICAL ABSTRACTS.

Extension of certain micro-analytical methods to the determination of lactose. J. M. CLAVERA and F. M. MARTIN (Anal. Fis. Quím., 1930, 28, 1066–1079).—The Folin-Wu, Bertrand, and Hagedorn-Jensen methods have been critically examined and modified with a view of ascertaining their suitability for the determination of lactose in physiological fluids such as milk. The last-named method, as modified by the present authors, yields the most accurate results, takes but 30 min. to complete, and requires only about 1 c.c. of the milk. The following procedure is recommended: to 5 c.c. of the milk are added 10 c.c. of 0.45% zinc sulphate solution and 2 c.c. of 0.1*N*-sodium hydroxide; a similar blank solution is prepared and is heated on the water-bath for 3 min., cooled, and filtered. To 1 c.c. of each solution are added 2 c.c. of alkaline potassium ferricyanide solution (1.65 g. of ferricyanide and 10.6 g. of sodium carbonate per litre), and, after heating for exactly 15 min. on the water-bath, the solutions are cooled rapidly, 2 c.c. of 3% acetic acid and 2 c.c. of a mixture of 4 vols. of a solution containing 10 g. of zinc sulphate and 50 g. of sodium chloride in 160 c.c. with 1 vol. of 12.5% potassium iodide solution are added, and the solutions are titrated with 0.005*N*-sodium thiosulphate. A table for calculating the lactose content of the milk is given. H. F. GILLBE.

Analysis of chocolate. I. Determination of the components of pure chocolate consisting entirely of cocoa, sugar, and cacao butter. R. LÉCOQ (J. Pharm. Chim., 1930, [viii], 12, 101–109).—Analyses of various chocolates containing only cocoa, sugar, and cacao butter are given and interpreted in terms of these components. If *B* be the quantity of cacao butter added as such, and *C* the quantity of cocoa used, then the total butter present = $B + (C \times 0.54)$. The insoluble material other than cacao butter = $\text{cocoa} \times 0.3$. The total content of sugar is only slightly greater than the amount of sugar added.

W. J. BOYD.

Classification of materials extracted from coffee. VON CRUPKA (Chem.-Ztg., 1930, 54, 803).—The extracts from raw coffee are called natural extracts; the further extracts obtained from raw coffee which is roasted, after treatment with solvents to remove the natural extracts, are called pyrogenetic extracts. The amounts of material removed by successive solvents are given as totals, and as fractions removed by successive extractions with different solvents of the solute removed by the original extractions.

S. I. LEVY.

Proteins and vitamins in the embryo of soya beans. S. OHTOMO (Rep. Lab. S. Manchuria Rly., 1929, 15–17).—Soya bean contains 5.95% of rind, 91.96% of endosperm, and 2.09% of embryo. An analysis of the embryo showed that the oil content is almost one half that of the endosperm. The amounts of nitrogen extracted from the embryo by water, 10% saline, 0.2% caustic soda, and that found in the residue are also given. A classification of the water-soluble proteins and the distribution of nitrogen (van Slyke)

are recorded. From nutritional experiments on rats and pigeons it is concluded that there are higher concentrations of vitamins *A* and *B* in the embryo than in the endosperm.

H. J. DOWDEN.

Nutritive value of soya-bean cake and purified soya-bean cake. S. OHTOMO (Rep. Lab. S. Manchuria Rly., 1929, 10–12).—Although the protein constituent of soya beans is denatured by the extraction of oil, it has been found that the cake still possesses nutritive value. Extracted cake is of less value than the expressed, and purified cake than the original. Artificial digestion with pepsin and trypsin showed that the nutritive value is lowered by alcoholic extraction.

H. J. DOWDEN.

Preparation of soy sauce from soya-bean cake. II. E. YOSHINO and K. SEKI. III. Composite Y-type fermentation. E. YOSHINO, K. SEKI, and K. YAMAMOTO (Rep. Lab. S. Manchuria Rly., 1929, 21–22, 22–23).—II. As a potential outlet for soya-bean cake an attempt has been made to prepare soy sauce. The product brewed from the cake and kaoliang, wheat bran, etc. at 24–32° for 100 days was comparable with the commercial material. At room temperature the rate of decomposition was too low to be practicable. The yield was improved by the addition of soya-bean oil.

III. Koji was prepared from soya-bean cake and wheat separately, and the fermentation products of different ages were united to form a material of satisfactory colour, flavour, etc.

II. J. DOWDEN.

Determination of sandy matter in soya-bean cakes. S. USAMI (Rep. Lab. S. Manchuria Rly., 1929, 23–24).—A sample (10 g.) of air-dried soya-bean cake ground to pass 20-mesh is shaken with 50 c.c. of carbon tetrachloride (d_{20}^{25} 1.58) and after keeping for a few minutes the volume of settled sand is measured in a special wedge-shaped vessel. The sand may be filtered, dried, and weighed.

H. J. DOWDEN.

Mineral feeding with dairy cattle. O. E. REED and C. F. HUFFMAN (Mich. Agric. Exp. Sta. Tech. Bull., 1930, No. 105, 3–63).—A supplement of calcium is not essential even when timothy or other low-calcium roughage is fed. Raw rock phosphate is harmful.

CHEMICAL ABSTRACTS.

Determination of sand in the presence of indigenous silica in feeding-stuffs. W. LEPPER (Landw. Versuchs-Stat., 1930, 110, 305–311).—The sample (5 g.) is ashed in a platinum dish over a micro-burner and the residue heated on a water-bath with 20 c.c. of 15% sodium hydroxide solution for 30 min. The mixture is transferred to a beaker, diluted to 400 c.c. with water, and 100 c.c. of dilute (1:1) hydrochloric acid are added. The undissolved residue is collected on a filter, washed, ignited, and weighed; this weight represents "sand." Filtration is hastened by the use of filter pads supported on a porcelain filter disc.

A. G. POLLARD.

[Determination of] chlorine by the "open Carius" method. W. B. WHITE (Ann. Rep. N.Y. Dep. Agric., Leg. Doc., 1930, No. 37, 100–102).—The addition of potassium permanganate (5 g.) prior to digestion is recommended.

CHEMICAL ABSTRACTS.

Turmeric and annatto tests. W. B. WHITE (Ann. Rep. N.Y. Dep. Agr., Leg. Doc., 1930, No. 37, 97—98).—In the modified procedure the substance to be tested is treated with concentrated ethyl alcohol and a few drops of alcoholic boric acid, slightly acidified with tartaric acid, and placed in a small porcelain crucible or on a porcelain plate. The characteristic rose colour is carried to the edge. CHEMICAL ABSTRACTS.

Contamination of food cooked or stored in contact with nickel-chromium-iron alloys. A. C. TITUS, H. B. ELKINS, H. G. FINN, L. T. FAIRHALL, and C. K. DRINKER (J. Ind. Hygiene, 1930, 12, 306—313).—Determinations of the amounts of metal dissolved from strips of different nickel-chromium-iron alloys in contact with various foodstuffs during cooking and storage showed that such alloys are hygienically suitable for the manufacture of culinary utensils. The average amounts of the metals dissolved by various foods cooked for 1 hr. in contact with the iron alloy containing 15% Ni and 18% Cr was 1.01 mg. of iron, 0.05 of chromium, and 0.06 mg. of nickel per 4 dm.² of surface.

G. F. MARRIAN.

Fungi found in butter. M. GRIMES, V. C. E. KENNELLY, and H. A. CUMMINS (Sci. Proc. Roy. Dublin Soc., 1930, 19, 549—569).

Edible fats. RUZICZKA. **Phosphatides from soya beans.** SATO and SETO. **Denatured soya beans.** OKANO and NINOMIYA. **Vitamins in soya-bean cake.** OHTOMO. **Cotton-seed oils.** PERDRIGEAT.—See XII. **Determination of tannin.** OBERHARD and SCHALBEROVA.—See XV. **Green fodders.** HASELHOFF and others.—See XVI. **Starch and bread-baking.** KATZ.—See XVII.

PATENTS.

Manufacture of artificial cream. A. BERGSVIK, VITACREAM, LTD., and R. HELLERUD (B.P. 334,863, 8. and 22.6.29 and 8.3.30).—Artificial cream which can be whipped is prepared by injecting under pressure, through a small orifice, vegetable fat of m.p. 32° into a mixture of unskimmed milk, spray-dried egg-yolk, and fresh egg-yolk. E. B. HUGHES.

Manufacture of pectin. A. F. BROOKE (B.P. 334,974, 24.6.29. Austral., 3.5.29).—Waste citrus material, e.g., the pith, seeds, etc., is extracted twice by simmering with 1½ times its weight of water, strained, and the solution (about 5% of pectin) concentrated to 20—30% pectin or dried to a powder.

E. B. HUGHES.

Detection and determination of moisture (B.P. 335,308). **Deodorisation etc. of fluids** (B.P. 314,022).—See I. **Bleaching compositions** (B.P. 334,531).—See VI. **Oils and fats from fish etc.** (B.P. 335,438).—See XII. **Improving the flavour of water** (B.P. 335,687).—See XXIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Simple preparation of ether for narcosis from commercial ether. L. M. v.D. BERG (Pharm. Weekblad, 1930, 67, 1029—1033).—The properties and reactions of ether which has been three times extracted with conductivity water, distilled over calcium chloride, and collected in three fractions of, respectively, 10, 80,

and 10% are recorded; the middle fraction, amounting to about 52% of the crude ether taken, is found always to conform to the specification for ether *ad narcosin*.

S. I. LEVY.

Analysis of [medicinal] alcoholic solutions of nitroglycerin. H. CARON and D. RAQUET (J. Pharm. Chim., 1930, [viii], 12, 109—118).—Various methods of evaluating such (1%) solutions of nitroglycerin are discussed. The saponification method of D.A.B. VI, with or without the use of hydrogen peroxide, is unsatisfactory owing to the complicated nature of the reaction. As a rapid approximate method it is recommended to add 0.1 c.c. of the sample to 1 c.c. of a 10% solution of phenol or salicylic acid in concentrated sulphuric acid and, after mixing, to add 10 c.c. of water and 10 c.c. of ammonia solution and to compare the coloration obtained with that given by a standard solution of potassium nitrate similarly treated. For more accurate analysis 10 c.c. of the sample are treated with 2—3 g. of Devarda's alloy, 100 c.c. of water, and 25 c.c. of caustic soda solution, and, when the evolution of hydrogen slackens, the ammonia formed is distilled into 20 c.c. of 0.1N-sulphuric acid, the excess acid being titrated with 0.1N-alkali, using as indicator sodium alizarinsulphonate sensitised according to the process of Mestrezat (B., 1920, 316 A).

W. J. BOYD.

Determination of nicotine in oriental tobaccos. J. BURMANN (Helv. Chim. Acta, 1930, 13, 785—787).—Dry, powdered tobacco (3 g.) is triturated with 30% potassium hydroxide solution (3 c.c.) and plaster of Paris (3 g.) is added gradually to the mixture. The resultant powder is extracted with ether (75 c.c.; freshly distilled from phosphoric oxide), the extract (50 c.c.) evaporated, and the residue dissolved in three successive portions of ether (5 c.c.); the solution is evaporated in each case in order to remove traces of ammonia. The final residue is dissolved in ether (20 c.c.), water (50 c.c.) added, the ether evaporated, and the resulting aqueous solution titrated with 0.01N-hydrochloric acid, using one drop of a 1% alcoholic solution of bromocresol-purple as indicator. The percentage of nicotine is given by multiplying the number of c.c. of acid used by 0.081. The method gives results identical with those obtained using the silicotungstate method. H. BURTON.

Physico-chemical analysis of an essential oil from *Eucalyptus globulus*. T. BATUECAS and E. MORALES (Anal. Fis. Quím., 1930, 28, 1036—1044).—The oil was completely miscible with ethyl alcohol, and contained 56.5% of cineol and only traces of water. It had $d_{20}^{21.5}$ 0.9148, d_4^{16} 0.9192, $n_D^{19.3}$ 1.4664, b.p. 168—190°, f.p. below —20°, cal. value 9705—9112 g.-cal., and viscosity (Engler) at 20.8° (or 54.3°) 1.18° (1.06°), flash point (Pensky-Martens) 47°.

H. F. GILLBE.

Essential oil of the leaves of *Laurus reobilis*, L. B. N. RUTOVSKI and N. N. MAKAROVA-SEMLIANSKAJA (Trans. Sci. Chem. Pharm. Inst., Moscow, 1928, No. 19, 157—165).—Laurel leaves yielded 0.38—1.38% of an oil having d_{20}^{20} 0.9128—0.9332, α_D —14.0° to —23.26°, n_D^{20} 1.4641—1.4722, acid value 0.44—4.26, ester value 30.57—59.0, ester value after acetylation 51.86—70.5, cineole content 50—53.8%, phenols 4.0—6.6%.

Eugenol, acetyleugenol, α -pinene, and *l*- α -terpineol were present.

CHEMICAL ABSTRACTS.

Composition of Caucasian geranium oil. B. N. RUTOVSKI and N. N. MAKAROVA-SEMLIANSKAJA (Riechstoffind., 1929, 4, 172—173, 193—195; Chem. Zentr., 1930, i, 2175).—The aqueous distillate of *Pelargonium roseum* is acid, and contains 0.15—0.4 g. of oil per litre. The oil contains (by wt.) citronellol 64.2%, geraniol 1.2%, linalool, 7.4%, terpene 1.5%, menthone 4.6%, sesquiterpenes 1.4%, sesquiterpene alcohol, and acetic, butyric, and tiglic acids as the esters.

L. S. THEOBALD.

Zdravet oils. Y. R. NAVES (Parfums de France, 1929, 7, 304—305; Chem. Zentr., 1930, i, 2176—2177).—The products of distillation and extraction of *Geranium macrorrhizum*, L., are recorded. L. S. THEOBALD.

Carrot oils. B. N. RUTOVSKI and N. N. MAKAROVA-SEMLIANSKAJA (Parf. mod., 1929, 22, 807; Chem. Zentr., 1930, i, 2175).—The ethereal oils of the seeds of *Daucus carotta* and of *D. gingidium* have a strong odour like that of iris. L. S. THEOBALD.

Germicidal powers and capillary activities of certain pure constituents of essential oils. E. K. RIDEAL and A. SCIVER (Perf. Ess. Oil Rec., 1930, 21, 341—344).—In continuation of the investigation previously described (B., 1928, 730), the capillary activities (as given by the "drop numbers") and the germicidal values (as given by the Rideal-Walker coefficients) of the pure constituents of a number of essential oils have been determined. The results for linalool, santalol, menthol, geraniol, borneol, eugenol, thymol, citronellal, cinnamaldehyde, citral, menthone, carvone, cineole, terpineol, anethole, and safrole are tabulated and classified according to their chemical constituents.

H. J. DOWDEN.

Oil viscosimeter. SCHAFFER.—See I.

PATENTS.

Treatment of extract of cascara sagrada. A. ROBERTSON, and J. TAYLOR (TRONGATE), LTD. (B.P. 335,115, 25.11.29).—Practically tasteless extracts of cascara sagrada are obtained by treatment of the concentrated aqueous extract with oxygen produced by the addition of oxygen-generating substances, e.g., hydrogen peroxide, preferably in presence of reducing substances such as charcoval and at temperatures above 37°.

E. H. SHARPLES.

Production of caffeine [from theobromine]. H. E. POTTS. From MONSANTO CHEM. WORKS (B.P. 334,741, 7.10.29).—Theobromine in the form of its sodium salt is methylated in a closed vessel with methyl chloride at 70—85°. The yield of caffeine is 181—186 lb. from 180 lb. of theobromine. C. HOLLINS.

Extracting natural alkaloids and simultaneously forming alkaloid salts by a double circulation of non-miscible liquids. C. PALMERI (B.P. 314,498, 21.6.29. It., 28.6.28).—A continuous process for the extraction of alkaloids from vegetable substances containing them consists in the maceration of these substances in a solution of a salt of high electrolytic property (sodium or calcium chloride) rendered alkaline by the addition of a base such as calcium, sodium, or potass-

ium hydroxide, or ammonia, whereby organic acids and colloidal substances are precipitated. Such solutions are caused to circulate countercurrent through an organic solvent of different density, such as vaseline, which, in turn, circulates through an acid solution of different density, so that only the alkaloid passes from the salt solution into the organic solvent and from the latter to the acid solution. E. H. SHARPLES.

Preparation and separation of the hormones of the anterior lobe of the hypophysis and of the ovary. SCHERING-KAHLBAUM A.-G. (B.P. 313,923, 18.6.29. Ger., 19.6.28).—First the placenta or a preparation thereof is fractionally extracted with an organic solvent miscible with water, such as methyl alcohol, in the presence of a certain amount of water; then the ovarian hormone is separated from the solvent-free extract by extraction with immiscible solvents; and finally the hormone of the anterior lobe of the hypophysis is precipitated from the residual liquors after concentrating, if necessary, or the latter substance may be precipitated first and the ovarian hormone obtained from the residual liquors. E. H. SHARPLES.

Preparation of a pure hormone from the anterior lobe of the hypophysis. SCHERING-KAHLBAUM A.-G. (B.P. 313,924, 18.6.29. Ger., 19.6.28).—Salts and ballast materials are removed from aqueous preparations of organs or excreta containing the hormone of the anterior lobe of the hypophysis by treatment with alkaline-earth or heavy-metal salts, and the pure hormone is isolated from the residual liquor in the usual manner. E. H. SHARPLES.

Extracting and separating the active substances from the posterior lobe of the hypophysis. W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 334,898, 11.6.29).—The fresh or dried parts of the glands are extracted with a mixture of an alcohol and an inorganic acid, the active substances are wholly or partially separated by neutralisation or by addition of an organic precipitant and, in the case of partial separation, the portion remaining in solution is isolated by shaking the solution with water or by removing the precipitant after having neutralised the greater part of the acid present. The fractions obtained as above may be further purified and separated by dissolution in alcoholic mineral acid followed by fractional precipitation of albumin and cleavage products with organic precipitants.

E. H. SHARPLES.

Manufacture of hydroxypyridine compounds. C. RÄTH, Assr. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,778,784, 21.10.30. Appl., 10.4.28. Ger., 14.4.27).—See B.P. 288,628; B., 1929, 709.

Pulp from tobacco waste (U.S.P. 1,756,722).—See V. **Effervescent compositions** (U.S.P. 1,764,996).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photomicrography of textile sections. PERROTT.—See V.

PATENT.

Arc carbons or electrodes (B.P. 316,130).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Nitration of trimethylbenzenes. KHOLEVOL.—See III. **Solvents etc. for nitrocellulose.** ZIMMER.—See XIII. **Celotex [pith].** LATHROP.—See XVII. **Analysis of dilute solutions of nitroglycerin.** CARON and RAQUET.—See XX.

PATENT.

Treatment of glycerin. H. WEBER and F. NIEMEYER (U.S.P. 1,763,451, 19.6.30. Appl., 12.1.27. Ger., 26.1.26).—The formation of undesirable polymerisation products when glycerin is heated to form diglycerol is avoided if compounds containing silica, *e.g.*, silica gel or alkali silicates, are added. Thus a mixture of glycerin and 0.2–0.25% of hydrated water-glass, heated under reflux for 6 hrs. at 220–240°, yields a mixture containing glycerol 86%, diglycerol 13%, residue 1%, whilst by the addition of 0.25% of crystallised sodium metasilicate to glycerin the proportions are 80%, 18%, and 2%, respectively.

W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Atmospheric pollution (Dept. Sci. Ind. Res., Rep. 15, 1930, 64 pp.; cf. B., 1928, 732).—During the year ended March 31, 1929, an improvement is shown in most stations, but a falling off in others. The maximum deposits of tar and sulphates were recorded at the City Rd. Station, Newcastle-on-Tyne. Tar equivalent to 5.04 and sulphates equivalent to 4.03 metric tons per sq. km. were deposited in January, 1929, the yearly totals being 12.39 and 30.11 metric tons, respectively.

C. JEPSON.

Antimony method for the determination of sulphide [in sewage]. A. J. SALLE and E. A. REINKE (Calif. Sewage Works J., 1929, 2, 208–209).—The sample (100 c.c.) is treated with 1 c.c. of a solution containing 50 g. of potassium antimonyl tartrate per litre and 0.5 g. of sodium chloride followed, after mixing, by 1 c.c. of hydrochloric acid (1:1). The mixture is then compared colorimetrically with standards, corrections for turbidity and colour being applied. The preparation of the potassium dichromate-cobalt chloride-silica standards is described. Volatile sulphides are determined by difference after aeration.

CHEMICAL ABSTRACTS.

Filtering materials for water and sewage works. ANON. (Proc. Amer. Soc. Civil Eng., 1930, 56, 1851–1890).—In order to predict rapidly the way in which any kind of medium could be expected to behave under the conditions appertaining in sewage trickling filters, a test was devised in which the sample was immersed in a saturated solution of sodium sulphate for 18 hrs. at 32°, heated at 100° for 5 hrs., then re-immersed, and the cycle repeated 20 times. Fairly concordant results were obtained from 12 laboratories in which the test was applied to a series of duplicate samples, and the possibility is indicated of the sodium sulphate soundness test becoming a practicable and reliable method of determining the soundness of proposed trickling-filter materials.

C. JEPSON.

Purification of drinking water with sodium aluminate. P. I. VASILIEV (J. Appl. Chem., Russia,

1930, 3, 207–219).—When a mixture of sodium aluminate and aluminium sulphate is used instead of aluminium sulphate alone, the time required for flocculation and precipitation of the impurities is shortened, the amount of active carbon dioxide in the water is reduced, and the utilisation of aluminium is more complete.

CHEMICAL ABSTRACTS.

Micro-determination of potassium in [potable] waters. M. H. GRIFFON and A. BERNARD (J. Pharm. Chim., 1930, [viii], 12, 118–124).—The cobaltinitrite micro-method of Leulier, Velluz, and Griffon (A., 1928, 1205) for determination of potassium has been applied to water analysis. For waters containing at least 0.080 g. K per litre 2–5 c.c. of sample suffice, but in analysing samples of lower content 100–2000 c.c. of sample are evaporated in a platinum capsule to dryness on the boiling water-bath and the residue is taken up in 10 c.c. of dilute (1%) acetic acid. The resulting solution is filtered through cotton wool and 2–4 c.c. are used for the analysis. The results obtained for various natural waters by the cobaltinitrite method are compared with those of other authors when alternative methods were used. The former are concordant among themselves, but differ considerably from the latter, which are non-concordant for different authors.

W. J. BOYD.

Toxicity of nicotine etc. RICHARDSON and SHEPARD.—See XVI.

PATENTS.

[Settlement tanks for] sewage purification. C. J. HARTLEY (B.P. 334,851, 10.4.29).—In the type of settlement tank described the mixed liquor is admitted into a central chamber, the sides of which extend to a suitable distance from the tank bottom. The effluent is discharged over cills in the usual way and the sludge is deposited on the sides of a shallow inverted cone which forms the bottom of the tank. In order to prevent an undue retention of the deposited sludge thereon, which in the case of the activated sludge process would interfere with the purification, the sides of this cone are traversed by scrapers which gather the sludge into the centre, whence it may be discharged by hydraulic head through a vertical, centrally situated pipe. This pipe may be arranged to revolve and carry the scrapers, and to provide for this it is connected by ports to a loose annular chamber through which the sludge passes to a fixed discharge pipe.

C. JEPSON.

Preventing incrustation by and improving the flavour of water. GROECK WASSERVEREDLUNG GES. M.B.H., Assees. of H. GROECK (B.P. 335,687, 15.8.29. Ger., 22.11.28).—To the water to be used for drinking and cooking purposes are added acids or acid salts, *e.g.*, lactic acid, in the presence of a sparingly soluble substance, *e.g.*, calcium carbonate, capable of combining with any excess of acid which may be added.

C. JEPSON.

Disposal of sewage and other waste organic matter. C. G. WIGLEY and C. PORTS (B.P. 335,682, 12.8.29).—See U.S.P. 1,730,489; B., 1929, 998.

Alkali hypochlorites (B.P. 334,364 and U.S.P. 1,765,013). **Sodium borate-trisodium phosphate** (U.S.P. 1,759,152).—See VII. **Bactericidal paints** (B.P. 335,242).—See XIII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 19 and 26, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Formation and properties of boiler scale. E. P. PARTRIDGE (Dept. Eng. Res., Univ. Michigan, 1930, Bull. No. 15, 170 pp.).—Chemical analysis and crystallographic examination show that the chief constituents of boiler scale are anhydrite, calcite, brucite, and magnesium and calcium silicates, whilst aragonite and calcium hydroxide are occasionally present. The solubilities of calcium sulphate, magnesium hydroxide, and calcium hydroxide, and probably also of calcium carbonate, decrease with rise of temperature. Calcium and magnesium silicates are also believed to have solubility curves with negative slopes; hence it appears that all the substances found in boiler scale are characterised by negative solubility curves. The maximum value of the heat conductivity coefficient of boiler scales is about 2.0 B.Th.U./ft.²-hr.-ft.-1° F. Dense, compact scales such as those formed by calcium sulphate have a coefficient of 1.3, whilst porous scales, owing to the pores becoming filled with a film of poorly-conducting steam, have coefficients as low as 0.5. The actual heat loss due to scale is only about 2% for scale $\frac{1}{8}$ in. thick, but such a scale has a serious effect on the raising of the metal temperature when the heating surface is exposed to direct radiation; with a rate of heat transfer by radiation of 75,000 B.Th.U./ft.²-hr. non-porous scales 0.05—0.1 in. thick soon produce failure of the tubes by overheating. The present knowledge of the mechanism of scale formation is reviewed, and it is shown that the rate of scale growth may be expressed by the equation $dD/dt = -K \times (dS/dT)^m \cdot (dQ/dt)^n$, where S is the solubility of the scaling substance, T the temperature of the boiler, Q the quantity of heat transferred in time t to deposit an amount of scale D , K a coefficient which is constant for each scale-forming substance, and m and n are constants, both approximately equal to unity. The prevention of scale formation depends either on complete softening of the feed water or careful control of the chemical equilibria in the boiler water to precipitate solids which will form scale only slowly if at all. Any one of the methods in use for external softening at the present time is satisfactory in the first case, but probably only the phosphate method is reliable in the second. The rate of formation of calcium sulphate scales has been measured in an experimental boiler unit; at pressures up to 25 lb. hemihydrate is found in the scale, but at pressures above 50 lb. only anhydrite is formed. Equilibria in the system calcium sulphate-water have been investigated by X-rays; only three crystal forms of the salt exist, viz., gypsum, hemihydrate, and anhydrite, the transformation point for gypsum to hemihydrate being 98° and for gypsum to anhydrite 37°. In contact with water at 11—15° anhydrite is converted into gypsum

with setting similar to that of plaster of Paris. The so-called "soluble anhydrite" is identical in crystal structure with hemihydrate, and should be called, therefore, dehydrated hemihydrate, as this substance apparently belongs to the zeolitic type which loses and regains water of hydration without change in crystal structure. The solubilities of hemihydrate and of anhydrite have been determined at 100—200°, a tentative equilibrium diagram for the system calcium sulphate-water has been prepared, and a new theory of scale formation on a boiler heating surface, based on photomicrographic examination of early stages of calcium sulphate scale formation, is presented.

A. R. POWELL.

Pebble-mill linings. R. TWELLS (J. Amer. Ceram. Soc., 1930, 13, 669—674).—The relative advantages and disadvantages of using flint, fine-grained quartzite, porcelain, and rubber for pebble-mill linings are discussed. Cost data for the various types of linings are given.

R. J. CARTLIDGE.

Nature of flow. E. C. BINGHAM and B. LOWE (Coll. Symp. Ann., 1929, 7, 205—212).

Forensic investigations of firearms, maliciously damaged trees, and forged coins. O. MEZGER (Chem.-Ztg., 1930, 54, 753—755, 774—776, 830—831, 851—853).

Smelting furnace. COOKE.—See VIII.

See also A., Nov., 1396, **Standardisation of a modified Ostwald viscosimeter** (CHADWELL and ASNES).

PATENTS.

Reverberatory furnace. F. F. LINES (U.S.P. 1,763,471, 10.6.30. Appl., 11.7.21).—The gas uptake of the furnace is outside the air uptake and a slidable water-cooled sleeve carries the gas across the air uptake to a throat, leading into the furnace, where air and gas mix. The result of sliding the hollow damper is that the annular space for passage of air from the uptake to throat is varied while the gas passage remains constant. When, however, the sleeve is fully retracted, the gas uptake is shut off altogether and an uptake fully open; this position may then be used at the outlet end of the furnace for the time being, only the downtake (which was the air uptake) leading to regenerators.

B. M. VENABLES.

Tunnel kiln. T. LARSSON, Assr. to NORTON CO. (U.S.P. 1,763,624, 10.6.30. Appl., 26.3.28).—The kiln is of the muffle type, combustion takes place in narrow spaces between the main and muffle walls, these spaces being continued to the end of the kiln at which the goods enter, to effect preheating. The waste gases are then passed through a separate recuperator for the air, and the latter is further preheated in a second exchanger by a current of air drawn through passages round the goods

* The remainder of this set of Abstracts will appear in next week's issue.

leaving the kiln. The muffle walls are formed of corrugated overlapping plates which are free to expand and contract; they may be made of, *e.g.*, crystalline alumina bonded with highly refractory clays. Provision is made for admitting cold air to the muffle spaces and heat exchangers to regulate the temperature.

B. M. VENABLES.

Rotary-hearth oven. F. BARTLING (U.S.P. 1,765,196, 17.6.30. Appl., 27.6.27. Ger., 15.1.27).—The goods to be heat-treated are spread in a thin layer on a slowly rotating annular hearth, which is heated from below, during part of the circle, by surface-combustion devices. Since the latter occupy the space ordinarily taken by the rails and carriage, the hearth is formed as an arch between carriages on inner and outer sets of rails.

B. M. VENABLES.

Continuous method of calcining pulverulent materials, chemicals, pigments, or the like. R. KRAUSSE (B.P. 335,659, 11.7.29).—The material is treated in a muffle, which is jiggged to cause the material to travel through it. The suspending links and driving means are outside the furnace.

B. M. VENABLES.

[Water-cooled] retort. W. F. RIEGER, Assr. to STOKER Matic Corp. (U.S.P. 1,763,539, 10.6.30. Appl., 27.8.28).—A device for the pre-coking of fuel in the furnace of a boiler is described.

B. M. VENABLES.

Drying apparatus. H. A. GILL. From NICHOLS PRODUCTS CORP. (B.P. 335,942, 3.7.29).—The material is subjected in a closed vessel to steam which is under a pressure sufficient to prevent the evaporation of the "moisture" fluid and which is superheated by internal electrical resistors. After soaking, the pressure is released at such a rate that the material becomes uniformly dry throughout.

B. M. VENABLES.

Conveyor-dryers. B. J. OWEN and R. O. DAVIES (B.P. 336,009, 10.7.29).—In apparatus of the type described in B.P. 334,940 (B., 1930, 1096), the main division of the air currents is effected by providing separate fans for each zone. The flow starts at the dry end and the same air passes in turn through each zone to the wet end, but provision is made for heating by steam and/or admixture of furnace or flue gases at the beginning and intermediate points. Application in the sugar industry is indicated.

B. M. VENABLES.

Centrifugal dryer. C. W. TERWILLIGER (U.S.P. 1,764,909, 17.6.30. Appl., 28.11.27).—A resilient bearing for the shaft of a centrifuge is described.

B. M. VENABLES.

Drying of liquid materials. D. J. VAN MARLE, Assr. to BUFFALO FOUNDRY & MACHINE CO., INC. (U.S.P. 1,762,250, 10.6.30. Appl., 7.4.25).—A method of feeding the liquid to a heated roller is described. A sheet of the fluid is allowed to flow, in excess, down an inclined shoot, so that it contacts with the lower part of the roller, the excess being allowed to fall freely away.

B. M. VENABLES.

Exclusion of unfiltered air from tunnel and like drying apparatus. TOMLINSONS (ROCHDALE), LTD., and J. N. TOMLINSON (B.P. 336,049, 22.8.29).—To the outlet end of a dryer, which cannot be maintained airtight owing to the continual exit of goods, is supplied an

excess of filtered air, so that there is always an outward draught.

B. M. VENABLES.

Carrying out exothermic reactions. [Production of calcium chloride.] K. STAIB, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,764,582, 17.6.30. Appl., 12.5.27. Ger., 10.7.26).—In reactions which are exothermic, more especially those between gases and solids, the temperature is regulated by admixture of inert solid material with the charge, the size of the inert particles being different from that of the active particles, so that they may afterwards be separated by screening. *E.g.*, in the production of calcium chloride, small bricks may be formed of lime and coal with a small quantity of calcium chloride as binder; these are mixed with "retort coal" as inert material, and then treated with chlorine gas after a slight preheating.

B. M. VENABLES.

Apparatus for carrying out reactions continuously in the liquid phase, under increased pressure. IMPERIAL CHEM. INDUSTRIES, LTD., and K. H. SAUNDERS (B.P. 336,136, 14.11.29. Addn. to B.P. 329,260; B., 1930, 690).—A vessel as described in the original patent is provided with a thin, non-corrodible lining, *e.g.*, of stainless iron; a non-corrosive constituent of the reaction sweeps through a narrow space between the lining and the pressure-resisting wall on its way to the reaction zone.

B. M. VENABLES.

Heat-exchange apparatus. B. F. STURTEVANT Co., Assees. of G. C. DERRY (B.P. 315,865, 5.7.29. U.S., 21.7.28).—A method of connecting in groups the horizontal tubes of an economiser or other type of heat exchanger is described.

B. M. VENABLES.

Manufacture of heat-insulating masses. I. G. FARBENIND. A.-G. (B.P. 336,163, 15.12.29. Ger., 11.3.29).—A solution of water-glass (*d* 1.38) or other alkali silicate is heated at 130–150° in air for 1 hr., and then at 450° in carbon dioxide for 3 hrs.

H. ROYAL-DAWSON.

Heat-insulating coverings. R. T. KEMPER (B.P. 335,747, 7.10.29. Ger., 27.3.29).—A tank or other vessel is covered with overlapping slabs composed of sheet metal, netting, and insulating material which are held in place by friction only, *i.e.*, without drilling the wall of the vessel.

B. M. VENABLES.

Pulverising machine. B. A. O'NEILL, Assr. to SCHUTZ-O'NEILL Co. (U.S.P. 1,765,309, 17.6.30. Appl., 28.9.23).—A disintegrator and fan are mounted on the same overhanging portion of the driving shaft, and can be removed therefrom without disturbing the main bearings; the fan volute is attached by flanges to the disintegrator casing, and can be rotated so that its outlet points in any direction. On the first disc of the disintegrator at a less radius than that of the hammers are side projections, which in conjunction with the corrugated end of the casing serve as preliminary crushers and deflect uncrushable pieces into pockets formed in the end of the casing.

B. M. VENABLES.

Pulverising or crushing machines. F. K. WOODROFFE (B.P. 336,021, 17.7.29).—A ball mill is run at a speed rather above the critical centrifugal speed, so that the balls will be carried right to the top; they are less

in number than usual and are deflected downward by a stationary internal plate, so that they will fall through a greater distance than usual and the falling stream will be separate from the rising mass. B. M. VENABLES.

Pulverising apparatus. L. V. ANDREWS, Assr. to RILEY STOKER CORP. (U.S.P. 1,763,496, 10.6.30. Appl., 5.7.29).—The rotating disc of the disintegrator divides the casing into coarse- and fine-grinding zones which communicate only at the circumference. A resilient liner for the upper part of the casing takes the form of a steel half-hoop which is secured only to the lower half of the casing, and is lined with wearing pieces of harder material. A pocket for uncrushable pieces is formed at the bottom of the casing, and at this point the passage from coarse to fine zones is guarded by a shelf which is of frangible material, so that in the event of a serious breakage of the disintegrator the loose parts may break through the shelf and enter the pocket.

B. M. VENABLES.

Grinding mills. A. W. McALLISTER (B.P. 336,005, 9.7.29).—A machine suitable, *e.g.*, for the ingredients of chocolate is provided with main grinding surfaces comprising a cylinder rotating within a cylindrical casing, each surface being provided with rifling, of which the section is shaped like a blunted saw tooth, and the direction of rotation being such that the action is one of wiping, not of scraping. The material is admitted at one end through a preliminary coarse grinder, comprising a disc with circumferential grooves and teeth formed on the ridges rotating against the end of the casing, which is of similar form.

B. M. VENABLES.

Apparatus for comminuting materials. C. H. BIRCH (U.S.P. 1,762,381, 10.6.30. Appl., 17.10.27).—The material is flung by disintegrator hammers into the nip between two rollers. One of the rollers is built up of annular sections which, except the end ones, have a limited radial freedom on the shaft, and attached to the lower roller is a scraper device which also forms a pocket to guide the material into the nip.

B. M. VENABLES.

Apparatus for pneumatic separation of materials. R. H. KIRKUP (B.P. 336,055, 29.8.29).—The material is stratified by upward-pulsating air currents which are at all times uniform over the whole surface; a single fan is therefore used, and the pulsations are produced by a number of louvers rotating in line and in phase across the main air stream. B. M. VENABLES.

Mixing and discharging apparatus. H. S. BEERS, Assr. to TURBO-MIXER CORP. (U.S.P. 1,764,498, 17.6.30. Appl., 24.10.29).—In the lower part of a tank containing viscous fluid is a bladed rotor which runs between fixed deflecting blades and acts as a mixer. When mixing is complete the rotor can be lowered through the bottom of the tank, and then runs within a volute casing and acts as a centrifugal pump to discharge the fluid.

B. M. VENABLES.

Centrifugal extractor. J. M. KREIDER (U.S.P. 1,764,214, 17.6.30. Appl., 3.10.28).—A small, simple, and robust machine is described, suitable, *e.g.*, for drying small metal parts which have been plated or lacquered.

B. M. VENABLES.

[Laboratory] filtration apparatus. A. W. PETREY, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,762,738, 10.6.30. Appl., 7.6.29).—A device for filtering and washing a large number of exactly similar assays is described.

B. M. VENABLES.

Method of filtering. C. D. MORTON, Assr. to MORTON SYSTEM, INC. (U.S.P. 1,762,560, 10.6.30. Appl., 15.7.27).—The method is suitable for partially de-watering sewage or other fluid in which the proportion of solids is small. The prefilter flows endways into a horizontal cylindrical chamber, and the thickened sludge is drawn off from the lower part of the other end. A cylindrical filter having an axial outlet for filtrate is rotated within the casing, leaving only a narrow annular space so that the sludge, even when thickened, has considerable longitudinal, or rather spiral, motion over the surface of the filter. The pressure and rate of feed and the speed of rotation are so co-ordinated that the filter remains clear indefinitely.

B. M. VENABLES.

Apparatus for separating solids from liquids. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 335,975, 4.7.29).—The apparatus is of the combined centrifugal basket-and-bowl type. An inner, perforated, conical wall of the rotor retains the coarser solids, and the finer solids and liquid slide down an outer, imperforate, conical wall to an outermost, imperforate, vertical wall, where the remaining solids are retained and over the upper edge of which the liquid is continuously siphoned. The solids are discharged at intervals through suitable doors, preferably while the feed is cut off.

B. M. VENABLES.

Separation of solids from liquids [by filtration]. A. H. GODBE, Assr. to UNIVERSAL PROCESS CO. (U.S.P. 1,763,773, 17.6.30. Appl., 1.11.26).—The filter membrane forms a cylindrical or conical division in a tank. Before use, it is thoroughly cleaned from grease or other matter tending to make air adhere to it and is then dipped in water or previously obtained filtrate; during use, air is allowed to pass freely away. Clogging of the pores is thus prevented.

B. M. VENABLES.

Apparatus for removing the separated solid matter from strainers for liquids. DORR CO. (B.P. 335,767, 24.10.29. U.S., 9.11.28).—A scraper suitable for cleaning a bar screen for sewage is caused to move in an oval path by means of a pantograph mechanism having no submerged bearings.

B. M. VENABLES.

Apparatus for emulsification. L. S. and E. M. RICHARDS, and CREMAC MARKETING CO., LTD. (B.P. 335,949, 4.4.29).—The fluids, melted if necessary, are forced by a hand-operated piston through restricted passages such as those formed by a loosely fitting crew plug; no outlet valve is necessary and the inlet is through a port in the cylinder wall, which is uncovered by the piston at the end of the suction stroke. Beyond that port is a leakage-collecting recess, so that no packing is required for the piston.

B. M. VENABLES.

Evaporators [for generating steam from brine]. D. A. QUIGGIN (B.P. 335,911, 30.4.29).—An evaporator of the marine type is provided with conical baffles below the heating coils, which deflect sludge to the

bottom and cause a circulation of brine upwards at the centre and downwards at the sides; the circulation is also aided by admitting the sea-water feed through a ring-pipe with holes on its underside and by exhausting brine through a similar pipe upwardly pierced. Scale is removed from the steam coils when the evaporator is blown down by allowing them to become hot and suddenly chilling them by sprays of cold feed.

B. M. VENABLES.

Treatment of boiler water. H. KRIEGSHEIM, ASSR. to PERMUTIT Co. (U.S.P. 1,762,784, 10.6.30. Appl., 25.4.27).—The treatment is suitable for alkaline make-up water. Sulphur is burned in a deficiency of air and the raw water treated with the products of combustion; the sulphur dioxide removes both dissolved oxygen and alkalinity and forms sulphates which are useful in preventing caustic embrittlement, and the residual nitrogen is used to form a neutral atmosphere over the hot well and other places where feed-water both new and returned is stored. The amount of sulphur should be insufficient to neutralise all the alkaline salts.

B. M. VENABLES.

Gas washer. H. KREISINGER, ASSR. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,762,338, 10.6.30. Appl., 10.10.27).—In an air-borne pulverising system the vented air only is scrubbed by steam and water moving concurrently downwards in a tower to a tank at the bottom which acts as a separator for the sludge and as a return bend for the air which is exhausted upwards through an adjacent tower.

B. M. VENABLES.

Apparatus for washing gas. H. A. BRASSERT & Co., LTD. From H. A. BRASSERT (B.P. 335,648, 6.7.29).—The apparatus is of the disintegrator type, the bars being T-shaped with the head of the T radial.

B. M. VENABLES.

Liquid and gas contact apparatus. R. P. MASE (U.S.P. 1,765,087, 17.6.30. Appl., 2.8.28).—In a bubbling tower the trays each comprise a bottom and roof which are concave to each other. A number of annular baffles alternately depend from the roof and have notched bottom edges, whilst others dip into the liquid and allow free passage for the gas above them and liquid below them, so that the gas bubbles through the liquid several times in each tray. On account of the high back-pressure thus set up, non-return flaps are provided in the downflow pipes for liquid.

B. M. VENABLES.

Decomposition of gas mixtures. GES. F. LINDE'S EISMASCHINEN A.-G., F. POLLITZER, and H. KAHLE (B.P. 335,599, 26.6.29).—A mixture of gases, e.g., coke-oven gas, is separated, by partial condensation after cooling, in several stages, each of which comprises several heat exchangers through which the gas being cooled flows in parallel and in a downward direction; such heat exchangers provide separate paths for the returning products which effect the cooling. In the case of coke-oven gas the desired product (hydrogen) is not condensed and is kept separate throughout the return flow, but the liquefied nitrogen, carbon monoxide, methane, etc. are added to stages corresponding to their b.p. and emerge together. The withdrawal of heat to compensate for losses is effected in one of the cooler exchangers by

means of liquid nitrogen from an external source which has its partial pressure reduced by admixture of a little hydrogen from the circuit.

B. M. VENABLES.

Apparatus for compressing gases. N. GRIBOJEV (B.P. 314,801, 27.6.29. Fr., 2.7.28).—An apparatus involving both piston and centrifugal compression is described, the reciprocating action being obtained from rollers running in a sinuous groove.

B. M. VENABLES.

Pumps for obtaining high vacua. E. R. GROTE (B.P. 336,001, 9.7.29).—A disc rotates at the highest possible speed with small clearance between two plates which form a gastight casing. A circular groove is cut in each plate about $\frac{1}{4}$ in. away from the edge of the disc and each groove is stopped at one point, the inlet and outlet being on either side of the stop, which is shaped so as to guide the molecules of gas to the outlet. Though the surfaces are actually smooth, they are rough in comparison with the size of a molecule, and the rebound of the molecules will have an average direction towards the outlet. The grooves may be connected in series or parallel according to the requirements of the pump.

B. M. VENABLES.

Apparatus for determining the sp. gr. of so id or liquid substances or bodies. W. & T. AVERY, LTD., and W. A. BENTON (B.P. 336,043, 14.8. and 21.11.29).—The apparatus described in B.P. 320,416 (B., 1930, 4) is simplified and extended to determine the sp. gr. of solids lighter than water and of liquids. In each case there are only two vertical sliders and one swinging arm, no knife edges or parallelogram being required.

B. M. VENABLES.

Thermometers. G. BREWER. From Z. MATSUMOTO (B.P. 335,825, 29.11.29).—To afford easier reading, the inside or outside, back or front of the thermometer tube is roughened by painting, etching, or forming serrations.

B. M. VENABLES.

Repairing heated structures such as furnaces, retorts, etc. A. McD. DUCKHAM (U.S.P. 1,780,120, 28.10.30. Appl., 15.5.29. U.K., 24.5.28).—See B.P. 313,728; B., 1929, 664.

Tube mill. W. JAEDEL, ASSR. to MASCHINENBAU-ANSTALT HUMBOLDT (U.S.P. 1,780,132, 28.10.30. Appl., 19.4.29. Ger., 23.4.28).—See B.P. 310,342; B., 1930, 798.

Grinding mills. G. F. PETTINOS (B.P. 317,710, 19.8.29. U.S., 18.8.28).—See U.S.P. 1,758,010; B., 1930, 1008.

Pulverising machine. P. A. HIRSCH (B.P. 336,776, 3.10.29. U.S., 15.11.28).—See U.S.P. 1,744,895; B., 1930, 490.

Emulsifying and analogous apparatus. V. G. WALSH (U.S.P. 1,780,853, 4.11.30. Appl., 26.12.29. U.K., 4.10.28).—See B.P. 323,534; B., 1930, 224.

Filtering device. F. W. YOUNG (U.S.P. 1,780,777, 4.11.30. Appl., 27.7.26).—See B.P. 280,170; B., 1929, 79.

Sealing rings for rotary kilns and dryers. HURON INDUSTRIES, INC., and B. E. GREEN (B.P. 336,729, 28.8.29).

[Domestic] apparatus for [electrically] heating liquids. L. F. THOMPSON (B.P. 336,011, 11.7.29).

[Domestic, electrically driven] hot air drying apparatus. A. HOFFMANN (B.P. 337,337, 20.3.30. Ger., 20.3.29).

Refrigeration apparatus. SILICA GEL CORP., Assecs. of E. B. MILLER (B.P. 317,471, 16.7.29. U.S., 18.8.28).

Working a continuously-acting absorption refrigerating machine. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 337,188, 26.10.29. Ger., 26.10.28).

Production of cold. K. BERGL (B.P. 336,557, 11.6.29).

[Fixing the plates in] centrifugal mills. C. TILLOY, Assee. of M. B. DAVEZIES (B.P. 336,121, 28.10.29. Fr., 28.3.29).

[Roller machine for] moulding of plastic substances. A. W. SIZER (B.P. 336,912, 25.9.29).

[Non-leaking] atomisers for liquids. R. HADDAN. From T. J. HOLMES Co., INC. (B.P. 336,123, 29.10.29).

Spray producers. I. W. P. BUCHANAN (B.P. 337,064, 30.5.29).

[Combined charging and discharging fixture for] compressed-gas containers. C. J. JOHANN (B.P. 319,760, 15.8.29. U.S., 28.9.28).

[Cleaning of bags in] air filters. I. L. BRAMWELL, C. W. H. HOLMES, and BIRTLEY IRON Co., LTD. (B.P. 335,922, 26.3.29).

Machine for dissolving cellulose xanthate etc. (U.S.P. 1,763,379).—See V. Precipitator for gases (U.S.P. 1,760,461). Pyrometer (U.S.P. 1,762,380).—See XI. Heating apparatus for milk etc. (B.P. 335,964).—See XIX.

II.—FUEL; GAS; TAR; MINERAL OILS.

Relation between ash content and calorific value of Chikuhō coal. K. TAKEI (Fuel, 1930, 9, 393).—Examination of a large number of coal samples of widely varying ash content from the Chikuhō field shows that the calorific value of such coals can be calculated from the percentage ash content of the dry coal (*A*) with an error of not more than 5% by means of the formula: cal. value (in kg.-cal.) = $8080 - 92A$. A. B. MANNING.

X-Ray stereoscopic examination of coal. A. N. WILSON (J. Inst. Fuel, 1930, 4, 64–65).—The actual extent and thickness of inclusions in coal such as pyritic masses cannot be determined with certainty from the inspection of a single radiographic view, but this information may be obtained by making successive identical exposures with slight rotation of the specimen between each. The application of this method is described and radiographs of typical mineral inclusions are given.

C. B. MARSON.

Possible improvements in gas producers. J. RUDE (Gas World, 1930, 93, 320–322).—The reactions occurring in a gas producer, using coke, and the effects of steaming are described. Theories are advanced to account for the larger amounts of carbon dioxide which are formed near the producer walls. It is suggested that greater efficiencies can be obtained by preheating the

blast of air and steam; the amount of steam which can be used is thereby increased, the reaction zone is extended, greater decomposition of steam is obtained, the quality of the gas is improved, the capacity of the plant is increased, and the amount of unburnt fuel remaining on the ash is diminished. C. B. MARSON.

Resin formation in benzols. I. Factors governing and mechanism of resin formation. W. H. HOFFERT and G. CLAXTON (Fuel, 1930, 9, 359–366).—Tests have shown that little, if any, resinification of the unsaturated constituents of benzol takes place when used in internal-combustion engines under normal conditions. The deposits in the induction system are due chiefly to the non-volatile resinous material already present in the benzol. No appreciable formation of resin occurs in benzols on storage at ordinary temperatures in the absence of oxygen. The first step in the formation of resin appears to be the production of organic peroxides, but the further mechanism of the process is still obscure. The main factors governing the resinification of benzols on storage are the nature and relative proportion of the unsaturated hydrocarbons present, the material of the containing vessel, and the presence of small quantities of substances having the power of promoting or retarding the process. Resinification proceeds more rapidly in glass than in tinned iron or copper vessels. The addition of phenols, amino-compounds, pyridine, etc. inhibits the formation of resins; the optimum concentration of cresol, one of the most effective of the inhibitors, is 0.03%. Sulphur dioxide, thiophenol, alkyl sulphates, etc. accelerate resinification. A. B. MANNING.

Resin formation in benzols. II. Prevention of resinification: technical-scale storage tests and road trials. W. H. HOFFERT and G. CLAXTON (Fuel, 1930, 9, 440–447; cf. preceding abstract).—The reactions involved in the formation of resins are extremely sensitive to traces of impurities, which may either accelerate or inhibit the process. The stabilisation of motor spirits by the addition of "inhibitors" has been tested on the technical scale, using alkali-washed gas-works' and coke-oven benzols, to which 0.03% by wt. of "tricresol" had been added. The treated benzols showed no appreciable increase in resins on storage for 34 weeks, and gave very little more deposit than normal in the induction manifold, inlet valves, etc. of the engine used in the road trials. The resin content of the gas-works' benzol with no inhibitor increased from 4–9 mg./100 c.c. after one week's storage to 36–37 mg./100 c.c. after 34 weeks. The results of a similar examination of the coke-oven benzol were not so satisfactory, owing to the difficulty of removing traces of phenols already present in the crude spirit. In using inhibitors for stabilising motor spirits there is a danger of subsequent accidental contamination of the spirit by impurities, e.g., zinc chloride used in soldering the containers, which accelerate resinification. A. B. MANNING.

Accelerated tests of asphalts. O. G. STRIETER (Bur. Stand. J. Res., 1930, 5, 247–253).—Methods are described for testing asphalt by accelerated weathering by the action of arc-lamp radiation, sudden chilling, and water sprays; the results obtained correspond

closely with those of natural weathering. Four to six types of asphalt may be recognised, according to their behaviour. The deterioration may be followed by the chemical changes which occur: in general, the oil and resin contents decrease, and the asphaltene content increases. Certain grades of asphalt contract during weathering.

H. F. GILLBE.

Vegetable oils [as lubricants] in aviation and in automobile engines. N. CHAMPSAUR (Chim. et Ind., 1930, 24, 519—525).—Compared with mineral oils, vegetable oils, *e.g.*, castor or olive oil, possess the advantages of having lower temperature coefficients of viscosity, of undergoing less carbonisation in the engine with the formation of carbon of lower abrasive action, and of producing less fouling of the parts of the machine lubricated. They have the disadvantages of more rapidly gumming, of becoming acid on storage, and of setting in cold weather. Olive oil possesses the highest "oiliness" and the lowest viscosity-temperature coefficient of the vegetable oils examined. The mean mol. volumes of both castor oil and olive oil are higher than those of most mineral oils; the vegetable oils are also relatively more homogeneous in composition. Road tests on small cars have shown a marked increase in power, combined with cooler running of the engine, when the mineral oil was replaced by olive oil. No difficulties were experienced, in a short test, due to gumming or acidity.

A. B. MANNING.

Conversion of methane into acetylene. K. PETERS (Z. angew. Chem., 1930, 43, 855—860).—A detailed discussion of the decomposition of the methane molecule, including the change of molecular structure, the energy changes, and the physical conditions under which a maximum production of acetylene can be produced. Details are given of experimental apparatus by which the desired decomposition has been achieved and the results obtained by various workers.

J. O. CUTTER.

[Separation of graphite in] Canadian laboratory. CARNOCHAN and others.—See VII.

See also A., Nov., 1366, **Ultrafiltration of petroleum** (ZAHARIA and LUCATU). 1378, **Rates of combustion of mixtures of gases** (TAMMANN and THIELE). 1380, **Mixed catalysts** (MITTASCH). 1408, **Salts and esters of humic acid** (FUCHS and HORN). 1425, **Berginisation of anthracene** (ORLOV and LICHATSHEV). 1436, **Mellitic acid and its derivatives** (MEYER and RAUDNITZ).

PATENTS.

Drying of coal in the mill. H. KREISINGER, ASST. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,760,148, 27.5.30. Appl., 11.8.28).—A fan and cyclone separator are arranged in circuit with the mill through which a constant-lifting stream of air is circulated. To dry the coal a current of hot gas is introduced into the mill in such a manner as to form a stratified layer above the entering current of relatively cooler circulating air. By this means a layer of cool air is interposed between the hot gas and those parts of the mill in which there is a collection of relatively stationary material, and ignition due to overheating of the latter is avoided. The moving

coal does not remain in the mill long enough to become overheated.

A. B. MANNING.

Hydrogenation of coal. A. MENTZEL (B.P. 334,389, 14.9.29. Ger., 12.10.28).—The material is heated in layers not more than 5 mm. thick in contact with a catalyst and in the presence of hydrogenating gases. The material is preferably carried in a loose layer, *e.g.*, by means of a current of gas or by gravity, over surfaces coated with the catalyst. [Stat ref.]

A. B. MANNING.

Apparatus for low-temperature carbonisation of bituminous and oil-bearing materials. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,443, 6.11.29. Addn. to B.P. 306,723; B., 1929, 347).—The internal diameter of the tubes through which the material is passed is in this modification made greater at the inlet than at the outlet end. The tubes may be uniformly conical in shape, or may be made up of two or three sections of different diameter joined together.

A. B. MANNING.

Standpipe for coke ovens. J. VAN ACKEREN, ASST. to KOPPERS Co. (U.S.P. 1,747,610, 18.2.30. Appl., 13.12.23).—The gases from the coking chamber pass through a short, slightly tapering gas outlet into a vertical standpipe and thence through a lateral conduit into the collecting main. As they ascend the standpipe they are sprayed with a liquid, *e.g.*, ammonia liquor, from a nozzle at the top of the pipe. The liquid is prevented from passing into the coking chamber by an umbrella plate or baffle mounted over the end of the gas outlet. The liquid is discharged from the annular space between the gas outlet and the standpipe. By this arrangement the gases are freed from ammonium chloride and formation of graphitic material in the standpipe is prevented.

A. B. MANNING.

Carbonisation and distillation of carbonaceous materials such as lignite, coal, wood, peat, etc. W. W. ODELL, ASST. to G. W. TRAER, JUN. (U.S.P. 1,747,731, 18.2.30. Appl., 29.11.22. Renewed 24.7.25).—The material is passed continuously down a vertical retort wherein it is carbonised by the combustion within the mass of part of the distillation gases and part of the material itself. For this purpose air is supplied through tuyères at two levels in the retort, the diameter of which is constricted at the corresponding hot zones. The retort is open at the top where any excess gas evolved may be burned. Traversing the retort below the lower tuyère is a perforated gas offtake pipe through which some of the gas produced may be withdrawn. Below the retort is a zig-zag cooling chamber, provided with inlets for steam or water, and having at its lower end a revolving paddle mechanism for removing the carbonised material from the retort.

A. B. MANNING.

Production of [distillation] products from vegetable substances. E. L. RINMAN (B.P. 334,724, 24.9.29. Swed., 17.7.29).—Vegetable substances are digested under pressure at about 180° with an aqueous solution of barium and/or strontium hydroxide, and the product, preferably after the addition of a neutral substance, *e.g.*, carbon powder, is evaporated to dryness and dry-distilled. Other inorganic substances, *e.g.*, oxides of calcium, magnesium, or aluminium, may be added

before the boiling; they facilitate the digestion and also the subsequent regeneration of the barium hydroxide. The distillation products obtained include alcohols, aldehydes, ketones, hydrocarbons, and hydrogen.

A. B. MANNING.

Distillation of resinous woods. J. C. SCHAEFFER, Assr. to AMER. PINE PRODUCTS CORP. (U.S.P. 1,757,144, 6.5.30. Appl., 11.10.26).—The wood is distilled in a retort the upper portion of which is heated externally by the hot gases from combustion chambers while the lower portion forms the top of a steam generator heated by the same combustion chambers. The wood is stacked in the retort in the form of elongated sticks with the grains extending upwards, and is progressively heated, first to remove the highly volatile constituents, and then to expel the rosin and effect charring of the wood. The charring progresses downwards along the sticks, while the gums and rosin trickle into inclined troughs in the bottom wall of the retort and are discharged therefrom through spouts at the ends of the troughs. A controlled amount of steam from the boiler is admitted to the retort. The volatile products evolved pass from the retort to a suitable condensing system and are collected therein.

A. B. MANNING.

Manufacture of carbon electrodes and the like. ROESSLER & HASSLACHER CHEM. Co., Assees. of H. N. GILBERT (B.P. 312,940, 1.6.29. U.S., 1.6.28. Cf. B.P. 251,982; B., 1927, 805).—A mixture of graphite and a carbonaceous binder, *e.g.*, pitch, containing up to 80% of the former, is heated with agitation to a temperature at which the condensable vapours evolved from the binder displace any occluded air. The mixture is then transferred to a mould, air being excluded during the process either by dropping liquid benzol into the mould or by subjecting the material therein to a vacuum, and compressed therein at 250–450°. If desired, the baked article may be subjected to further heating to graphitise the carbon.

A. B. MANNING.

Production of mixed oil gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (B.P. 334,485, 27.1.30. U.S., 11.7.29).—The apparatus comprises a generator, a carburettor, a superheater, a wash-box, a condenser, and a second wash-box, and the process includes the following steps: storing heat in the carburettor and superheater by burning the blow gases therein with secondary air; introducing a hydrocarbon oil alternately into the carburettor and superheater; passing the gases and vapours formed through both vessels, and in succession through the first wash-box; the condenser, the fuel bed, and the second wash-box. Water-gas may be produced in the generator by steaming the fuel bed in either direction to utilise the deposited carbon.

A. B. MANNING.

Retorts for the generation of oil gas. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT Co., LTD. (B.P. 334,346, 1.8.29).—In the upper part of an externally heated vertical retort for the production of oil gas, a truncated cone is suspended and the oil is fed in at the top of the retort in such a manner that it flows down the surface of the cone in a thin film. The oil-gas outlet is near the lower end of the retort wherein an inclined grid is provided upon which any coke or solid

formed is deposited. Below the grid is an outlet for unvaporised oil, which is returned to the upper end of the retort for re-treatment. One or more such retorts may be arranged at the top of a vertical coal-carbonising plant in such a manner as to be heated by the waste gases therefrom.

A. B. MANNING.

Gas-making apparatus. A. SCHWARZ, Assr. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,758,858, 13.5.30. Appl., 18.2.27).—One or more oil-gas retorts are inserted into the top of a water-gas generator, and are so placed as to lie in the path of flow of the air-blow gases and/or the water-gas. Heavy oil and superheated steam are fed into the retorts. By mixing the oil gas so produced with the water-gas in suitable proportions, a mixed gas of any desired calorific value is obtained.

A. B. MANNING.

Gas-producing apparatus. A. SEELEY (U.S.P. 1,760,782, 27.5.30. Appl., 18.7.25).—The mouthpieces of each retort in a setting can be connected either to a vertical standpipe or to a waste-heat flue lying adjacent and parallel to the standpipe. The connexion is controlled by means of a sliding cut-off movable within a casing which communicates on the one side with the mouthpiece and on the other with openings in the standpipe and flue, respectively; the cut-off closes each of the latter openings alternately. The device permits the retorts and the connexions to the standpipe to be burned out and cleared of carbonaceous deposits while the retort is being recharged.

A. B. MANNING.

Centralised control for gas producers. C. H. SMOOT (U.S.P. 1,755,567, 22.4.30. Appl., 16.4.26).—A device is provided which is controlled by the pressure of the gas produced and which varies in accordance therewith the supplies of air and steam to the producer, as well as the speed of rotation of the grate. *E.g.*, the damper in the air main, the throttle valve in the steam pipe, and the resistance in series with the motor operating the grate are controlled by levers connected to diaphragms, the movements of which are, in turn, controlled by the air pressures in a number of compartments of the central controlling device. Additional diaphragm controls serve to maintain the air and steam supplies constant as long as the pressure of the gas produced remains the same.

A. B. MANNING.

Automatic oil control for carburetted water-gas sets. M. PARSONS, Assr. to U.G.I. CONTRACTING Co. (U.S.P. 1,758,891, 13.5.30. Appl., 28.3.27).—A gas calorimeter is used in which the difference in temperature between two electrical resistance thermometers varies with the calorific value of the gas. The thermometers form two arms of a Wheatstone bridge, in which the movement of the galvanometer needle automatically controls, by means of a suitable electrical device, the valves of the oil supply to the carburettor. A gas of substantially constant calorific value is thereby obtained.

A. B. MANNING.

Gas purifier. Z. W. WICKS (U.S.P. 1,759,819, 20.5.30. Appl., 28.1.25).—An apparatus for treating a gas with a liquid purifying agent consists of a series of inclined pipes connected in zig-zag fashion by V-shaped elbows. The gas and liquid are passed together through the apparatus in the ascending direction. At

spaced intervals within the pipes are spiral plates which invert the stream of gas and liquid first in one direction and then in the other. The issuing mixture is led to suitable separating apparatus. A. B. MANNING.

Purification of gaseous mixtures. W. J. BANISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,757,826, 6.5.30. Appl., 25.1.26).—Gaseous mixtures, consisting principally of oxides of carbon, are passed over iron oxide at 400–850°, and are thereby freed almost completely from sulphur compounds. The process may be carried out under pressure. The iron oxide is preferably mixed with an inert supporting material, or with steel wool. A. B. MANNING.

Recovery of ammonia from coal gas and like industrial gases containing ammonia. GAS LIGHT & COKE CO., H. HOLLINGS, S. PEXTON, and W. K. HUTCHISON (B.P. 334,619, 11.6.29).—The dilute ammoniacal liquor obtained by cooling the gas to 45–55° is treated with a gas containing carbon dioxide, *e.g.*, flue gas, in order to convert the ammonia present into ammonium carbonate; the liquor is then used to scrub the gas for the recovery of ammonia therefrom. A. B. MANNING.

Purification of sulphur-bearing gases and recovery of sulphates and sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,526, 2.3.29).—The hydrogen sulphide in the crude gas is partly or completely converted into sulphur dioxide by catalytic oxidation and the hot gases are then brought into contact with a liquid containing thiosulphates, the reaction occurring being represented by the equation: $3(\text{NH}_4)_2\text{S}_2\text{O}_3 + 6\text{NH}_4\text{HSO}_3 = 6(\text{NH}_4)_2\text{SO}_4 + 3\text{H}_2\text{O} + 6\text{S}$. The treated gases, still containing sulphur dioxide, are cooled and scrubbed with the same solution in the presence of hydrogen sulphide and ammonia in order to produce a further quantity of thiosulphate. The solution is recirculated until sufficiently concentrated, when it is drawn off and the sulphate and sulphur are recovered therefrom. A portion only of the crude gases may be subjected to the oxidation treatment, and the remainder may be scrubbed directly with the sulphate-thiosulphate liquor. [Stat. ref.] A. B. MANNING.

Recovery of cyanogen compounds from gases. J. C. HAVN, Assr. to MERRILL Co. (U.S.P. 1,742,505, 7.1.30. Appl., 23.3.27).—The gases are scrubbed with water, which absorbs the hydrogen sulphide and hydrogen cyanide. The dilute solution so produced is passed through a tower wherein the dissolved gases are boiled off under reduced pressure. The hydrogen cyanide is separated from the hydrogen sulphide, and recovered by passing the vapours from the tower through a condenser at a temperature below the b.p. of hydrogen cyanide. A. B. MANNING.

Production of pitch and like material from tar. C. F. BROADHEAD and R. S. ANDREWS (B.P. 334,380, 2.9.29. Austral., 29.11.28).—Plastic pitch or artificial bitumen is produced from tar by digesting it for 8–16 hrs. at not above 95° with an aldehyde, preferably formaldehyde, and a catalyst, *e.g.*, ammonia. Air or other oxidising gas is subsequently passed through the hot material, preferably at a higher temperature (100–200°), until it has acquired the desired degree of hardness.

The amount of aldehyde required is approx. 1.25–4 vol.-% of the tar. A. B. MANNING.

Bituminous emulsions. N.V. DE BATAAFSCHE PETROLEUM MAATS., Assecs. of L. KIRSCHBRAUN (B.P. 312,580, 27.5.29. U.S., 26.5.28).—Asphalt or bitumen emulsions of a predetermined degree of stability, and therefore of any desired setting time, are prepared by mixing unstable emulsions, *e.g.*, those in which soap is the emulsifying agent, with stable emulsions, *e.g.*, those containing gelatin or clay, in suitable proportions. By mixing such emulsions, in which water constitutes the external phase, it is also possible to produce a compound emulsion of thinner consistency than that of either of the component emulsions. It may be necessary to adjust the p_H of one or both of the emulsions before mixing. A. B. MANNING.

Manufacture of emulsions with the aid of humic acid or the like. I. G. FARBENIND. A.-G. (B.P. 334,426, 21.10.29. Ger., 20.10.28).—By the addition of a small proportion of a volatile organic solvent, *e.g.*, methyl alcohol, to emulsions of fatty acids, tars, bitumen, etc. prepared with the aid of humic acid, they are rendered stable to frost. A. B. MANNING.

Refining of wood-spirit oils. W. QUERFURTH, Assr. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,759,369, 20.5.30. Appl., 25.2.28. Ger., 3.3.27).—The oils are passed in the form of vapour over heavy-metal oxides, *e.g.*, nickel or manganese oxide, preferably supported on a carrier such as pumice or rusted spongy iron, at above 300°. It is sometimes advantageous to dilute the vapours with steam, hydrogen, or other gases. A. B. MANNING.

Refining of hydrocarbon oils. E. W. GARD, B. G. ALDRIDGE, H. J. MULTER, and R. T. HOWES (U.S.P. 1,759,744, 20.5.30. Appl., 12.9.27).—Dehydration of hydrocarbon emulsions is accomplished by passing them through a chamber containing spaced, solid, foraminous blocks, from between each block of which a portion of the liquid is withdrawn and allowed partly to separate. The separated oil and water are removed and the remaining emulsion is returned through the same series of spaced filter elements. The separated oil is mixed with sulphuric acid, settled to separate as much acid as possible, and the remaining acid oil is passed through a filter chamber similar to that described above. The filtered oil is treated with a clarifying and decolorising agent, then with a sweetening agent, washed with caustic soda solution, and finally washed with water. H. S. GARLICK.

Removal of ash-forming constituents from [hydrocarbon] oil. H. G. M. FISCHER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,760,129, 27.5.30. Appl., 8.4.26).—Sodium sulphate and other ash-forming hydratable substances are removed from hydrocarbon oils (of the cylinder-stock type) by first decomposing by regulated heating the alkali soaps formed in the usual neutralisation of the oil, and then adding to the oil a 1% aqueous solution of, *e.g.*, isopropyl alcohol at 65–93°. Sufficient solution should be added to enable the sodium sulphate to form the decahydrate, which, after agitation, is allowed to separate. H. S. GARLICK.

Cracking still and process. M. J. TRUMBLE (U.S.P. 1,762,433, 10.6.30. Appl., 24.7.24).—Hydrocarbon oil is vaporised under pressure and in contact with a gaseous reagent in a two-phase oil-treating apparatus consisting of a vertical, heat-insulated still through which extends an inwardly and upwardly tapering flue, the lower part of which is connected to a furnace. Arranged in the still are a number of inverted gas or vapour cups, in the uppermost of which, out of contact with the oil in the still, is provided a suitable catalyst, *e.g.*, a platinum coil. Hydrogen or other treating gas is delivered under pressure to these cups. A helical scraper suitably supported is used to remove carbon and tarry matter from the sides and bottom of the still to an outlet. Connected to the top of the still is a vapour outlet and pressure-control valve.
H. S. GARLICK.

Apparatus for cracking oil. G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,759,446, 20.5.30. Appl., 28.3.21. Renewed 18.3.29).—Cracking stock is passed initially through a heating coil situated in a furnace into an expansion chamber, where vaporisation takes place. When vapours begin to pass into the dephlegmator connected to the expansion chamber, the stream of raw oil is diverted from the heating coil and passed in a number of independent streams through nozzles, which produce a fine spray, into separate portions of the dephlegmator, valves controlling the volume and velocity of the oil being so introduced. The reflux condensate and raw oil from the dephlegmator are passed to the heating coil, while vapours are taken off from separate portions of the dephlegmator to be condensed and the resulting distillate collected. Heavy residuum is withdrawn from the expansion chamber and the whole apparatus is operated under a regulated pressure.
H. S. GARLICK.

Cracking of hydrocarbon oils. E. W. ISOM, ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,762,229, 10.6.30. Appl., 20.4.27).—The mixture of vapours and gases from a cracking operation is subjected to condensation and the uncondensed gases and vapours are scrubbed first with the condensate or an oil mixture containing condensate, before the condensate is redistilled, and then with raw oil to be supplied to the cracking plant. The oil mixture from the first scrubbing operation is redistilled for separation of the desired product.
H. S. GARLICK.

Treatment of gasoline and the product thereof. A. P. BJERREGAARD, ASSR. to DOHERTY RES. CO. (U.S.P. 1,761,810, 3.6.30. Appl., 30.7.26).—A non-gumming, light-stable motor fuel consists of sodium plumbite-sweetened gasoline to which has been added 0.008–0.016% by wt. of an aromatic benzene homologue, preferably anthracene.
H. S. GARLICK.

Manufacture of liquid fuels. I. G. FARBENIND. A.-G. (B.P. 334,181, 27.4.29. Addn. to B.P. 226,731; B., 1925, 163. Cf. B.P. 252,019; B., 1927, 357).—The fuels described in the prior patents, *i.e.*, gasolines containing at least 0.2 g. of iron carbonyl per gal., are improved by the addition of up to 15%, generally about 1%, of an aromatic amine containing at least one alkyl radical in the molecule, *e.g.*, toluidine, monomethylaniline, with or without the further addition of organic

halogen compounds or organic compounds of metals or metal-like elements, *e.g.*, alkyl or aryl halides, boric esters, lead tetraethyl, etc.
A. B. MANNING.

System of lubrication. J. F. WERDER, ASSR. to E. ROGERS (U.S.P. 1,762,902, 10.6.30. Appl., 20.4.26).—A mixture of light paraffin and kerosene oils has dissolved therein carbon dioxide or other inert gas in such proportions that the resulting product exerts a pressure of 50–75 lb./in.² in a closed container fitted with a valve.
H. S. GARLICK.

Production of nicotine carbolates from crude shale oil. D. T. DAY (U.S.P. 1,762,471, 10.6.30. Appl., 12.11.24).—In a cyclic process, crude shale oil is intimately mixed with alcohol (preferably a liquid monohydric alcohol), which dissolves only the oily bases (pyridine and nicotine as their carbolates) and acids. The mixture of solvent with dissolved material is separated from the insoluble oil, and the alcohol separated by distillation from the cresylic acids. The product is useful as an insecticide.
H. S. GARLICK.

Treatment [cleaning] of carbonaceous materials. R. LESSING (U.S.P. 1,780,830, 4.11.30. Appl., 23.5.27. U.K., 27.5.26).—See B.P. 276,723; B., 1927, 866.

Manufacture of fuel briquettes. H. F. MAUREL, ASSR. to MAUREL INVESTMENT CORP. (U.S.P. 1,780,205, 4.11.30. Appl., 24.3.28).—See B.P. 325,869; B., 1930, 448.

Manufacture of high-purity carbon. D. GARDNER (U.S.P. 1,780,154, 4.11.30. Appl., 10.10.27. U.K., 5.3.27).—See B.P. 292,798; B., 1928, 631.

Apparatus for hot purification [desulphurisation] of gases. A. JOSEPH, ASSR. to COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (U.S.P. 1,780,159, 4.11.30. Appl., 30.7.28. Fr., 6.10.27).—See B.P. 298,236; B., 1929, 842.

Splitting up of mineral oils. M. MELAMID (U.S.P. 1,779,486, 28.10.30. Appl., 12.3.25. Ger., 22.3.24).—See B.P. 231,190; B., 1926, 622.

Burners for firing boilers, furnaces, and the like with gaseous fuel. J. E. WEYMAN (B.P. 336,722, 23.8.29).

Burners for combustion of pulverised solid fuel. F. K. WOODROFFE (B.P. 336,952, 18.7.29).

Liquid and gaseous fuel burners. M. KNUPFFER (B.P. 336,617, 16.5.29).

Decomposition of gas mixtures (B.P. 335,599).—See I. Urea (B.P. 335,421).—See III. Waterproofed paper (B.P. 335,559).—See V. Nitrogen-hydrogen mixtures (B.P. 335,524).—See VII. Fireproofing of wood (B.P. 334,408). **Treatment of sawdust** (U.S.P. 1,764,249).—See IX. Bituminous paint (B.P. 336,117).—See XIII.

III.—ORGANIC INTERMEDIATES.

Manufacture of synthetic borneol and synthetic camphor. J. SCHWYZER (Pharm. Ztg., 1930, 75, 1275–1276).—The conversion of α -pinene into bornyl ester by oxalic acid occurs in about 1 hr., whilst with salicylic acid several hours are necessary. The latter

acid, however, is exclusively used in the industry since the by-products (limonene, fenchene, etc.) are formed in smaller amount (30%, in place of 70%, of the weight of borneol produced). A laboratory process giving 95–100 g. of borneol from 700 g. of α -pinene (of which 500–520 g. are recovered) is described; the recovery of salicylic acid is at most 90%. For the conversion into camphor by boiling with copper powder in xylene, the borneol must be very pure and dry, the xylene dry and water-clear, and the catalyst highly active. The latter is prepared from precipitated cupric hydroxide by reduction in a quartz tube with a slow stream of hydrogen at 160–180°. The yield of camphor is 92–94 g. from 100 g. of pure borneol. On the large scale the α -pinene fraction of turpentine oil is heated with salicylic acid in a homogeneously welded or enamelled, steam-heated, iron vessel, fitted with stirrer, manometer, and condenser, but having no reflux condenser. The reaction vessel, separated by a cock from the condenser, is kept at 0.25–0.5 atm. excess pressure during the esterification. The unused pinene and by-products are then distilled off and fractionated separately, and the bornyl salicylate is hydrolysed, again under slight pressure, with dilute soda lye. The borneol is collected on a metal gauze, washed, centrifuged, and recrystallised from alcohol. For dehydrogenation an enamelled stirring vessel, with reflux condenser and a blow-off tube reaching nearly to the bottom, is used. The copper powder, after reaction, is allowed to settle, and after removal of the clear camphor solution it is ready for re-use. In the production of 100 kg. of camphor 180–200 kg. of turpentine oil are used, and the loss of salicylic acid is 10–12 kg.; the limonene by-products are sold at half the price of turpentine oil. The manufacture is profitable only when the synthetic camphor commands a price at least 4–5 times that of turpentine oil. C. HOLLINS.

Phytin from mustard. BELIAIEV.—See XIX.

See also A., Nov., 1380, Mixed catalysts (MITTASCH). 1409, Volumetric determination of acetone (MEYER and MATHEY). 1420, Purification of acetamide (WAGNER). 1425, Berginisation of anthracene (ORLOV and LICHATSCHEV). 1427, Azo-dye intermediates (RUGGLI and others). 1430, Naphthalene-green (FRISCH). 1439, Anthraquinoneazine and anthraquinoneazhydrin (SCHOLL and LAMPRECHT). Synthesis of anthraquinones related to morindone and emodin (MITTER and others).

PATENTS.

Production of methanol [methyl alcohol from carbon oxides and hydrogen]. BRIT. CELANESE, LTD., W. BADER, and E. B. THOMAS (B.P. 334,924, 12.4.29).—Zinc sulphide, alone or with chromium oxide, is used as catalyst, preferably at 360–400° and 100 atm.

C. HOLLINS.

Manufacture of primary and secondary amines. I. G. FARBEININD. A.-G. (B.P. 334,579, 5.6.29. Ger., 1.11.28. Addn. to B.P. 306,414; B., 1930, 808).—The process of the prior patent is extended to the conversion of alcohols generally into amines. *E.g.*, *n*-butyl alcohol and ammonia, led over nickel on pumice at 300°, give

mainly butyl- and dibutyl-amines; aniline and alcohol, and cyclohexylamine and alcohol diluted with hydrogen or nitrogen, at 185–200° give the secondary with only little tertiary amine. C. HOLLINS.

Manufacture of urea. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,421, 28.10.29. Holl., 13.11.28).—Carbon dioxide and ammonia are introduced into an inert, viscous liquid, *e.g.*, transformer oil, until a paste containing about 50% of oil is produced. This paste is pumped continuously into a high-pressure chamber maintained at 150–200° and under 100 atm. pressure. The reaction mixture is transferred to another chamber in which unconverted carbamate is distilled off, the ammonia and carbon dioxide being used again in the process, and the aqueous urea run off from the oily layer.

W. J. WRIGHT.

Manufacture of urea [carbamide] or products containing it. J. Y. JOHNSON. From I. G. FARBEININD. A.-G. (B.P. 334,564, 2.4.29).—Ammonium carbamate is heated at 150–250° in presence of at least 3 equivalents of ammonia (calc. on the combined carbon dioxide) and at a partial pressure of ammonia which is above the critical pressure. Thus 1 pt. of ammonium carbamate and 5 pts. of liquid ammonia heated in a monel-lined autoclave ($\frac{3}{4}$ full) at 150° for 1 hr. gives urea in 95% yield. The ammonium carbamate may be produced *in situ*, *e.g.*, from other carbamates, or from ammonium hydrogen carbonate etc. C. HOLLINS.

Manufacture of acetic anhydride. IMPERIAL CHEM. INDUSTRIES, LTD., H. HEPWORTH, and F. D. LEICESTER (B.P. 334,986, 4.7.29).—The vapours containing acetic acid, anhydride, and water are passed over active charcoal maintained at 140°, whereby the acid and anhydride are absorbed, and may be recovered as 96.5% acetic acid and a mixture of acetic acid and anhydride by raising the temperature and reducing the pressure.

C. HOLLINS.

Manufacture of aliphatic [acetic] acid anhydrides. H. DREYFUS (B.P. 334,533, 1.6.29).—Acetic or other acid vapour is passed through a copper tube filled with copper turnings at 400–700°. Catalysts, *e.g.*, an alkaline-earth tungstate, may be mixed with the turnings.

C. HOLLINS.

Manufacture of glacial acetic acid from dilute acetic acid. I. G. FARBEININD. A.-G. (B.P. 315,397, 12.6.29. Ger., 14.7.28. Addn. to B.P. 312,173; B., 1930, 1015).—The 60% acetic acid obtained by freezing is further concentrated by known means other than the double-compound method described in the prior patent.

C. HOLLINS.

Manufacture of highly stable sulphonic acids or their salts. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 313,453, 11.6.29. Ger., 11.6.28).—Fat acids or neutral fats are converted, *e.g.*, by means of phosphorus tri- or penta-chloride, into the corresponding acid chlorides, which are treated with concentrated sulphuric acid, oleum, or chlorosulphonic acid at about 30°. The products are probably sulphonated mixed carboxylic-sulphuric anhydrides. The sulphonation of acid chlorides from castor oil acids, acetylated castor oil acids, and olein is described. C. HOLLINS.

Manufacture of basic products from imino-ethers of higher fatty acids. I. G. FARBERIND. A.-G. (B.P. 308,218, 18.3.29. Ger., 19.3.28).—The imino-ethyl ether from stearic or other fatty acid above C_9 is condensed with an aliphatic diamine in which at least one amino-group carries an unsubstituted hydrogen atom. Ethylenediamine reacts with stearic imino-ethyl ether with elimination of ammonia and alcohol to give a compound, m.p. 93–95° (hydrochloride, m.p. 123–125°; acetate, m.p. 70°). A similar base is obtained from β -amino- β' -diethylaminoisopropyl alcohol. The products have therapeutic properties. C. HOLLINS.

Production of unsaturated esters. RÖHM & HAAS A.-G. (B.P. 313,877, 7.6.29. Ger., 18.6.28).— β -Hydroxy- or β -alkoxy-nitriles or -amides are heated at 120–170° with an alcohol in presence of concentrated sulphuric acid. Ethylene cyanohydrin [β -hydroxypropionitrile] with methyl alcohol gives methyl acrylate; the production of methyl cinnamate from β -hydroxy- β -phenylpropionitrile, ethyl acrylate from β -hydroxypropionamide, and methyl acrylate from β -methoxypropionitrile is also described. C. HOLLINS.

Manufacture of 6-halogeno-2 : 4-di(nitrophenyl)-1 : 3 : 5-triazines. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 334,887, 10.6.29).—6-Hydroxy-2 : 4-di-*m*-nitrophenyl-1 : 3 : 5-triazine, m.p. 280–281°, heated with 10 pts. of phosphoryl chloride, gives the 6-chloro-compound, m.p. 180°, in 90% yield. C. HOLLINS.

Manufacture of naphthalene derivatives. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 334,700, 28.8.29).—A 6- or 8-arylamino- β -hydroxy-3-naphthoic acid [5- or 7-arylamino-3-hydroxy- β -naphthoic acid], obtainable from 3 : 5- or 3 : 7-dihydroxy- β -naphthoic acid by condensation with an arylamine (cf. B.P. 326,971; B., 1930, 604), is boiled with bisulphite solution and a *p*-aminophenol, whereby the 2-hydroxyl group is replaced by a *p*-hydroxyarylamino-group and the carboxyl group is lost. The products yield indophenols by oxidation with hypochlorite or chromic and acetic acids. The condensation of *p*-aminophenol with the following 2-hydroxy-3-naphthoic acids is described : 6-anilino- (product, m.p. 205°), 6-*p*-toluidino-, 6-*p*-anisidino- (product, m.p. 207°), and 8-anilino-. From 2 : 6-dichloro-*p*-aminophenol and 6-anilino-2-hydroxy-3-naphthoic acid there is obtained a product, m.p. 182–183°. C. HOLLINS.

[Catalytic] oxidation of acenaphthene, acenaphthylene, and their substitution products. SELDEN Co., Assees. of A. JAEGER (B.P. 318,618, 16.8.29. U.S., 7.9.28).—Naphthalic anhydride is obtained smoothly by catalytic air-oxidation of acenaphthene etc. if steam be added to the reacting gases. If desired, sufficient steam may be used to convert the anhydride (condensed at 140–150°) into acid, which is thus obtained free from impurities volatile in steam. C. HOLLINS.

Manufacture of 4-hydroxyquinaldines. L. LIMPACH (G.P. 455,387, 17.2.26).—The yield of 4-hydroxyquinaldines from arylamines and acetoacetic esters rises from 30% to 90% when the β -arylaminoacrotanic ester first formed is heated in a solvent (paraffin oil)

at 240–250° for 15–20 min. Products from aniline, *p*-phenetidine, and α -naphthylamine are described.

C. HOLLINS.

Manufacture of isatoic anhydride and derivatives thereof. I. G. FARBERIND. A.-G. (B.P. 311,336, 7.5.29. Ger., 9.5.28).—An aqueous solution of an *o*-aminocarboxylic acid, acid to Congo-red, is phosgenated below 100°. Isatoic anhydrides from anthranilic acid, 3-amino-*p*-toluic acid, benzidine-3 : 3'-dicarboxylic acid, and 2 : 3-aminonaphthoic acid are described.

C. HOLLINS.

Manufacture of [di]hydroindole-indophenols. F. BALLAUF, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,058, 28.10.30. Appl., 25.10.26. Ger., 2.11.25).—See G.P. 443,685; B., 1928, 225.

Condensation product of the benzodiazine [quinazoline] series. W. HENTRICH, M. HARDTMANN, and R. KNOCKE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,879, 4.11.30. Appl., 8.3.28. Ger., 18.3.27).—See B.P. 287,179; B., 1929, 747.

Distillation products from vegetable substances (B.P. 334,724). **Refining of wood spirit oils** (U.S.P. 1,759,369).—See II. **Polymethine dye intermediates** (B.P. 334,706). **Compounds of anthraquinone series** (B.P. 334,565). **Intermediates of anthraquinone series** (B.P. 334,872).—See IV. **Treatment of sawdust** (U.S.P. 1,764,249).—See IX.

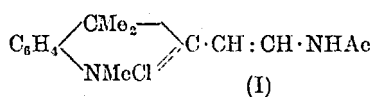
IV.—DYESTUFFS.

***ms*-Dichloroanthracene and its β -sulphonic acid as starting materials for production of alizarin.** V. I. MINAEV and B. P. FEDOROV (Rev. Gén. Mat. Col., 1930, 34, 330–332, 376–382).—Investigation of Perkin's method of making alizarin shows a 70% yield of *ms*-dichloroanthracene on chlorinating 93% anthracene in nitrobenzene (*o*-dichlorobenzene is less satisfactory); an 81.5% yield of sodium *ms*-dichloranthracene-2-sulphonate on sulphonation with 20% oleum in nitrobenzene at 10–15° (cf. G.P. 292,590; B., 1916, 921); an 82% yield of anthraquinone-2-sulphonic acid on oxidising in dilute nitric acid, or 90% with nitrogen peroxide in nitrobenzene, and a 90% yield of alizarin on fusion with sodium hydroxide and potassium chlorate. Curves are given of the rate of sulphonation; the optimum quantity of free sulphur trioxide is found to be the theoretical. The authors eliminate the isolation and purification of the *ms*-dichloroanthracene-2-sulphonic acid and increase the yield of anthraquinone-2-sulphonic acid from *ms*-dichloroanthracene to 92.2% by adding the nitric acid directly to the sulphonation melt. L. J. HOOLEY.

See also A., Nov., 1422, **Plant colouring matters—lycopene and carotene** (KARRER and others). 1427, **Azo dyes and their intermediates** (RUGGLI and others). 1430, **Naphthalene-green** (FRISCH). 1440, **Plant colouring matters—xanthophyll from dandelion flowers and its esterification and methylation** (KARRER and others). 1442, **Colouring matter of wild and cultivated blackberry** (KARRER and others). 1451, **Dyes from 2-aminothiazole-4 : 5-dicarboxylic acid** (DE). 1483, **Distribution of lutein in the vegetable kingdom** (KUHN and WINTERSTEIN).

PATENTS.

Manufacture of [polymethine] dyes and intermediates therefor. SOC. CHEM. IND. IN BASLE (B.P. 334,706, 2.9.29. Switz., 29.11.28).—An ammonium salt containing a reactive α -methyl group, or the corresponding ψ -base, is condensed with 1 mol. of a compound $X \cdot CH:NH$ (e.g., formimino-ethyl ether hydrochloride, formimino-chloride, formamidine hydrochloride, formiminoacetic esters, etc) in acetic anhydride. The intermediate so formed (e.g., I) or the corresponding



methylene base obtained from it by the action of alkali, may then be condensed with a second mol. of

cyclic ammonium salt etc. (the same or different) to give polymethine dyes. The intermediate (I) from 2-methylene-1:3:3-trimethylindoline (iodide, m.p. 240–247°; perchlorate, 228–229°) is condensed, e.g., with 2-methylene-1:3:3-trimethylindoline or with 2-methylene-1-methyl-I:2-dihydroquinoline. By using 2 mols. of cyclic ammonium salt or of ψ -base to 1 mol. of $X \cdot CH:NH$, the polymethine dye is formed directly.

C. HOLLINS.

Triarylmethane dyes. IMPERIAL CHEM. INDUSTRIES, LTD., E. H. RODD, and F. L. SHARP (B.P. 334,689, 19.8.29).—Violet triarylmethane dyes are obtained by condensing *m*-2-xylylidine with a diaralkyldialkyldiaminobenzhydrodisulphonic acid and oxidising the leuco-compound so obtained. Preferably the hydrol is produced *in situ*, e.g., by oxidation of dibenzyl-diethyldiaminodiphenylmethanedisulphonic acid, in presence of *m*-2-xylylidine, and the leuco-compound is oxidised in the same operation.

C. HOLLINS.

Vat dye enolic sulphuric esters, and their use. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 334,902 and 334,921, [A] 7.3.25, 3, and 10.5.29, [B] 7.3.29).—(A) A sulphuric ester of leuco-2-aminoanthraquinone (or its derivatives) is oxidised in presence of alkali, e.g., with hypochlorite, to form probably a tetrasulphuric ester of a leucoindanthrone, which may be isolated and purified by salting out with potassium chloride and, if desired, by washing with charcoal. The tetra-esters from 2-amino- and 3-chloro-2-aminoanthraquinones are described. (B) The tetra-esters, isolated or not, may be oxidised on the fibre for blue shades.

C. HOLLINS.

Manufacture of compounds of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,565, 6.4.29).—One or both halogens in 5:8-dihalogeno-1:2-benzanthraquinone are replaced by amino-, thiol, or hydroxyl groups which may or may not carry substituents. Toluene-*p*-sulphonmethylamide gives, after hydrolysis of the primary product, 5:8-di(methylamino)-1:2-benzanthraquinone, m.p. 249–252° (greenish-blue on acetate silk). The 5:8-diamino-compound, m.p. 224–226° (reddish-blue on acetate silk), 5:8-di-*p*-toluidino-compound, m.p. 204–206° (sulphonated for a green wool dye), 5:8-di-*(p*-tolylthiol) compound, m.p. 306–308° (sulphonated for red wool dye), 5:8-di- $(\alpha$ -anthraquinonylamino)-compound, and 5:8-diphenoxo-compound, m.p. 192–194° (sulphonated for orange wool dye), are also described. C. HOLLINS.

Production of dyes and intermediates [of the anthraquinone series]. W. SMITH, S. G. WILLIMOTT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 334,872, 5.3.29).—An aminoanthraquinone is condensed with a halogenobenzoylbenzoic acid, or a halogenoanthraquinone with an aminobenzoylbenzoic acid, the amino-group or halogen atom being *ortho* to a nitro-group. The products may be cyclised to give indanthrones. Methyl 2-*p*-chloro-*m*-nitrobenzoylbenzoate is condensed with 1-aminoanthraquinone, and the product is reduced, hydrolysed with alcoholic sodium sulphate, and cyclised to a blue vat dye. C. HOLLINS.

Manufacture of vat dyes [of the alizarin-indigo series]. I. G. FARBENIND. A.-G. (B.P. 335,043, 4.9.29. Ger., 28.6.29. Addn. to B.P. 318,107; B., 1930, 1060).— α -Anthrol is condensed with a 5-halogeno-7-alkoxy-4-methylisatin α -chloride (etc.), e.g., 5-chloro- or 5-bromo-7-methoxy-4-methylisatin chloride, for greenish-blue vat dyes. C. HOLLINS.

Manufacture of indigoid vat dyes. I. G. FARBENIND. A.-G. (B.P. 313,493, 7.6.29. Ger., 12.6.28).—A 5-halogenothioindoxyl is condensed with a 5:7-dichloro- or α -dibromo-isatin α -anil (etc.) for greenish-blue vat dyes. [Stat. ref.] C. HOLLINS.

Manufacture of stable reduction compounds of [indigoid] vat dyes. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 334,878 and Addn. B.P. 334,920, 11.5.29).—(A) A 6:6'-dihalogeno-4:4'-dimethylthioindigo is reduced with hyposulphite (or other reducing agent except alkali hydrogen sulphide) in presence of less alkali than is used for vatting. A brownish product, probably ketonic, separates. (B) 6:6'-Diethoxythioindigo gives a similar product. The products are stable and insoluble in alkali. C. HOLLINS.

Manufacture of azo dyes [acid dyes and lakes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,754, 12.10.29).—A 2:5-dialkoxyarylamine is diazotised and coupled with a 1-aroylamino-8-naphthol-4:6-disulphonic acid to give violet acid dyes for wool, which give barium lakes. Examples are: 2:5-dimethoxyaniline \rightarrow 1-benzamido-8-naphthol-4:6-disulphonic acid or the 1-(2':5'-dichlorobenzamido)-derivative; 2-amino-5-benzamidoquinol diethyl ether \rightarrow 1-benzamido-8-naphthol-4:6-disulphonic acid. C. HOLLINS.

Manufacture of soluble compositions for dyeing and printing. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,429, 1.5.29).—A water-soluble diazo-amino-compound, prepared according to B.P. 320,324 or 324,041 (B., 1930, 233 or 364), is incorporated with an azo coupling component free from sulphonic or carboxylic acid groups, particularly a 2:3-hydroxynaphthoic arylamide. The dry mixtures are stable, and are dissolved for use in water or dilute alkali. Fifty-two examples of yellows, oranges, scarlets, reds, bordeaux, violets, blues, browns, and blacks are given, including the following: diazo-amino-compound, 2:5-dichloroaniline \rightarrow 4-sulphoanthranilic acid, with coupling component, bisacetoacetylolidine (yellow); 4-chloro-*o*-toluidine \rightarrow 4-sulphoanthranilic acid, with 2:3-hydroxynaphthoic *o*-phenetidine (scarlet); *m*-chloroaniline \rightarrow α -naphthylamine-2:4-disulphonic acid, with *o*-anisidine (red); 4:5-dichloro-*o*-toluidine \rightarrow 4-sulphoanthranilic acid, with

m-nitroanilide (Turkey-red); 6-nitrocresidine \rightarrow *N*-methylglycine, with β -naphthylamide (bordeaux); 4-amino-6-benzamidoresorcinol dimethyl ether \rightarrow *n*-butylglycine, with *o*-toluidide (violet); 3-amino-6-benzamidoquinol diethyl ether \rightarrow methylglycine, with *o*-toluidide (blue); 4-nitro-4'-amino-2':5'-dimethoxyazobenzene \rightarrow methylglycine, with *o*-toluidide (black). [Stat. ref.]

C. HOLLINS.

Preparation of sulphonated water-soluble dyes of the diaminodiphenyl series. K. THIES and B. DIECKE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,048, 28.10.30. Appl., 10.2.28. Ger., 18.2.27).—See B.P. 285,504; B., 1929, 637.

Dye [for domestic use]. W. KRITCHEVSKY, H. C. PRUTSMAN, and E. MORRILL, Assrs. to PRODUCTS CORP. (U.S.P. 1,752,184, 25.3.30. Appl., 3.12.28).—See B.P. 331,491; B., 1930, 942.

Manufacture of sulphide dyes. M. WYLER, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,779,508, 28.10.30. Appl., 23.5.29. U.K., 26.2.29).—See B.P. 329,716; B., 1930, 811.

Preparation of a reddish derivative of flavanthrone. B. WYLAM, J. E. G. HARRIS, and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,779,791, 28.10.30. Appl., 19.4.26. U.K., 30.4.25).—See B.P. 260,638; B., 1927, 39.

Rosin soap lakes (B.P. 334,874).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Enzymic degradation of native and precipitated cellulose, of artificial silk, and of chitin. P. KARRER (Kolloid-Z., 1930, 52, 304—319).—The resistance to enzyme action offered by cellulose depends on the method of packing and orientation of the cellulose units. Resistance decreases in the order cotton, filter paper, mercerised cotton, regenerated cellulose, hydrocellulose (degraded cellulose). Stretching causes an orientation of the particles, and hence artificially-prepared threads are resistant. Cotton thread mercerised under tension is more resistant than that mercerised without tension. Bleaching causes little change in the resistance to enzyme action. Comparison of the rate of enzymic decomposition with the intensity of the X-ray diffraction ring reveals a parallelism which indicates the importance of orientation. Viscose silks show a very variable behaviour towards the action of enzymes, some being decomposed very readily and others only with difficulty. The resistance offered appears to bear no relation to chemical composition or physical properties, but depends on the shape of the cross-section of the fibres. If this is round and smooth the resistance is low, but if the fibres have a very irregular cross-section they are attacked only with difficulty. Irregular fibres are produced in acid precipitating baths and smooth fibres in concentrated salt baths, and experiments showed a direct connexion between the resistance to enzyme action and the composition of the precipitating bath. This shows again a connexion between resistance and structure and orientation of the coagulum. It is also found that the resistant viscose silks are more weakly

coloured by substantive dyes than those silks that are readily attacked, probably because the silks have the same relative adsorption capacities for the enzyme and the dye. The rate of decomposition increases with the concentration of the enzyme, as a result of the higher degree of adsorption, and agrees approximately with the course of a unimolecular reaction. Chitin from lobster shells can be hydrolysed by means of an enzyme contained in the liver and pancreas of the vineyard snail, and investigation of this reaction indicated many points of similarity to the phenomena observed with cellulose products. E. S. HEDGES.

Rapid determination of cellulose in viscose. H. HAWLIK (Chem.-Ztg., 1930, 54, 755).—About 1—1.5 g. of viscose is transferred to a glass plate, 9 \times 12 cm., and uniformly spread out by means of another superimposed plate. The plates are then separated and placed in a saturated solution of sodium or ammonium chloride, the viscose layer being uppermost. The xanthate film produced, which readily floats off the plates, is purified by immersing it two or three times in fresh salt solution, and then treated with 2% hydrochloric acid. The film of pure cellulose thus obtained is transferred to a flask with 25 c.c. of water, 15 c.c. of potassium dichromate solution (90 g. per litre) are added, followed by 25 c.c. of concentrated sulphuric acid, and the mixture is boiled for 4 min., cooled, and titrated with ferrous ammonium sulphate solution, using potassium ferricyanide as indicator. A blank test must be carried out. The determination is completed in $\frac{1}{2}$ hr.

W. J. WRIGHT.

Lilienfeld silk (Nuera silk). E. GEIGER (Helv. Chim. Acta, 1930, 13, 1114—1116).—Contrary to the statement made by Fierz-David and Brunner (B., 1930, 235), a warm spinning bath can be used in the manufacture of Lilienfeld silk. The shape of cross-sections of the fibres (various photomicrographs are given) is conditioned by the osmotic relations prevailing in the bath and the viscose sheath. When the osmotic pressure in the bath and the viscose is the same, the cross-sections are round; such silks are of the best quality and have the greatest tensile strength. The observed striation (cf. *loc. cit.*) on the fibres is due to light reflexion from the surface and not to the structure; ordinary viscose silk, spun without stretching, shows a more marked, but finer, striation. The addition of various substances to the spinning bath does not affect the stretching of the fibre; the surface tension is lowered thereby, causing more favourable coagulation and fixing conditions. The sample of silk examined by Fierz-David and Brunner has not the characteristics of the strongest Lilienfeld silk which can be produced. H. BURTON.

Test for strained viscose threads. A. J. HALL (Rayon Record, 1930, 4, 1001).—When stretched, viscose threads have an increased reactivity towards an alkaline silver nitrate solution containing silver nitrate 1 g., sodium thiosulphate 4 g., and caustic soda 4 g., per 100 c.c. When immersed in the nearly boiling solution for 1 min. stretched threads become brown and unstretched threads remain nearly colourless. The test is suitable for detecting excessively stretched viscose threads in woven and knitted materials. A. J. HALL.

[Detection of] strained viscose threads. J. M. PRESTON (Rayon Rec., 1930, 4, 1053).—Hall's test for stretched viscose threads (preceding) is also applicable to nitrocellulose silk, but not to cuprammonium silk, and it is therefore concluded that the test depends on the greater accessibility of the reducing groups present as produced by stretching. A. J. HALL.

Central Asiatic reeds as material for preparation of pulp. N. L. KARAVAEV and I. M. KRIWOZIAS (Papier-Fabr., 1930, 28, 654—656).—The stalks of *Phragmites communis* have been found to be similar to straw as papermaking material. Digestion with a 10% solution of sodium hydroxide at 160—165° and under 6—8 atm. yields pulps of quality sufficient for the manufacture of medium grades of paper. The resultant fibres are of the order of 1.5 mm. long and 17—18 μ wide. As in straw pulps, the ash contains an appreciable amount of silica, and the pentosan content is 6—8% in the best pulps obtained. T. T. POTTS.

See also A., Nov., 1415, Celluloses of some Australian plants (ARNEMAN and EARL). 1416, Dissolution of cellulose fibres in ammoniacal copper solution (SAKURADA). 1417, Kinetics of the "coppering" of cellulose fibres (SAKURADA). Copper oxide-ethylenediamine-cellulose (TRAUBE and others; TROGUS and SAKURADA). 1483, Determination of cellulose in straws (JENKINS).

PATENTS.

Degumming of silk. L. WALLERSTEIN, ASST. to WALLERSTEIN Co., INC. (U.S.P. 1,763,112, 10.6.30. Appl., 2.2.27).—Silk is washed in a dilute soap or weak alkali solution, and then degummed by immersion at 50° in a liquor containing a proteolytic enzyme, such as papain, and an accelerator, such as potassium cyanide or hydrogen sulphide. A. J. HALL.

Manufacture of felt. G. D. BEAL and R. R. MCGREGOR (U.S.P. 1,765,046, 17.6.30. Appl., 19.12.28).—Animal fibres are subjected to the action of steam under pressure in the presence of a salt yielding an acid reaction on hydrolysis, e.g., magnesium chloride. F. R. ENNOS.

Treatment of liner [for interposition between layers of rubberised fabric]. L. B. SEBRELL, ASST. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,763,618, 10.6.30. Appl., 16.9.27).—The liner fabric is treated with nitrocellulose containing butyl phthalate. F. R. ENNOS.

Machine for dissolving cellulose xanthate and the like. R. THURM and E. SCHMERER, ASSTS. to BAKER PERKINS Co., INC. (U.S.P. 1,763,379, 10.6.30. Appl., 29.11.26).—Spaced above the bottom of a dissolving bowl is a cylindrical drum open at both ends and having a number of radial bars with cutting edges fixed near the lower end; a spindle arranged co-axially with the cylinder carries a number of rotatable impeller blades adjacent to the fixed radial bars. Lumps of cellulose xanthate are continuously drawn up through the cylinder by the rotating impeller and are sheared into small pieces by the movable and fixed cutting edges, so that fresh surfaces are being continually exposed to the rapidly circulating alkaline liquor. F. R. ENNOS.

Manufacture of acetylcellulose. H. A. GARDNER (U.S.P. 1,764,204, 17.6.30. Appl., 27.4.28).—After acetylation of cellulose in the usual way, the reaction mixture is poured into ethyl ether and the precipitated acetylcellulose is washed by refluxing with ether; the ether and acetylation mixture are recovered by fractional distillation of the mother-liquor. F. R. ENNOS.

Manufacture of esters of polysaccharide ethers and of plastic or other compositions comprising the same. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,897, 11.6.29).—A polysaccharide ether is esterified by heating it with a carboxylic acid above C₆, with or without a diluent and/or a catalyst. The ether-esters are soluble in single organic solvents, relatively stable to hydrolytic agents, and suitable for use in films, plastics, lacquers, etc. Examples are: starch butyl ether esterified with linoleic acid at 110—150°; cellulose ethyl ether, with coconut oil acids at 130—150° in presence of boric acid, or with benzoic or stearic acid at 150°; cellulose benzyl or butyl ether with linoleic acid and a little boric acid at 130—150°. C. HOLLINS.

Production of artificial silk or the like from viscose solution. Twisting and reeling of artificial silk. ZELLSTOFFFABR. WALDHOF, and A. BERNSTEIN (B.P. 335,605—6, 27.6.29).—(A) In a non-continuous process, filaments which have been coagulated from viscose solution in an acid bath are led directly into hot water and simultaneously sthenosised either by addition of formalin or by tension, after which they are immediately spooled. (B) After being drawn off from the twisting spool the filament is allowed to fall directly into a liquid, whence it is guided by a guide member immersed therein to the winding reel. F. R. ENNOS.

Spinning of artificial silk. H. WADE. From COMPTOIR DES TEXTILES ARTIFIC. (B.P. 335,613, 28.6.29).—The partly coagulated thread is drawn through a narrow tube along which a current of liquid, which may or may not have the same composition as the coagulating bath, is passed in the opposite direction; by varying the length of the tube and the speed of the liquid any suitable tension may be applied to the thread. F. R. ENNOS.

Manufacture of artificial filaments and other products from organic derivatives of cellulose. BRIT. CELANESE, LTD. (B.P. 313,885, 17.6.29. U.S., 16.6.28).—Organic derivatives of cellulose are dissolved in solvent mixtures consisting of acetone with 1—30% of a glycol or other polyhydric water-soluble alcohol, under such conditions that a substantial proportion of the latter is retained in the product; yarns made in this way are self-lubricated and are readily scoured and dyed. F. R. ENNOS.

Cellulose ether, or ester, compositions. CELLULOID CORP. (B.P. 308,658 and 335,582, 25.3.29. U.S., 23.3.28).—Solutions, plastic masses, etc. are prepared from (A) cellulose ethers or (B) cellulose esters by incorporating therein non-, partly, or wholly polymerised vinyl compounds (vinyl acetate) together with solvents or plasticisers (alcohol, benzene) for the cellulose compounds and/or the polymerised vinyl compounds; poly-

merisation of the vinyl compounds is subsequently completed where necessary. F. R. ENNOS.

Production of solutions and compositions of cellulose esters and ethers and products made therefrom. H. DREYFUS (B.P. 315,766, 16.7.29. Fr., 17.7.28).—Carboxylic esters or ethers of cellulose are dissolved in aqueous, organic, or mixed aqueous and organic solutions of urea, thiourea, guanidine, or their alkyl derivatives, for the production of dopes, varnishes, plastic masses, films, artificial silk, etc. F. R. ENNOS.

Cellulosic compositions. [Plasticisers for plastics, coating compositions, films, etc. from cellulose esters or ethers.] BRIT. CELANESE, LTD. (B.P. 313,535, 13.6.29. U.S., 13.6.28. Addn. to B.P. 313,134; B., 1930, 1061).—Aromatic compounds containing a halogenated side-chain, *e.g.*, dibromides of cinnamic acid, styrene, cinnamyl alcohol, and cinnamaldehyde, are added to cellulosic compositions as plasticisers and fireproofing agents, with or without other such agents. An example is: cellulose acetate (100 pts.), xylene-sulphonmethylamides (25 pts.), tolyl phosphate (7 pts.), cinnamic acid dibromide (12 pts.), and alcohol (60 pts.). C. HOLLINS.

Production and treatment of textile and other materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 334,853, 31.5.29).—Improved strength and elasticity and modifications in lustre result from the treatment of cellulose ether or ester (*e.g.*, acetate) materials, during or after spinning, with "organo-mineral" acids, *i.e.*, di- or poly-hydroxylic inorganic acids in which one or some of the hydroxyl groups have been replaced by organic radicals, excluding, however, sulphaaromatic-fatty acids. Examples are sulphaacetic acid, ethylenesulphonic acid, methanesulphonic acid, sulphonic acids generally, phosphinic acids, etc. The acids are used in 30–45% aqueous solution. C. HOLLINS.

Waterproofing compositions [for paper containers]. DUNLOP RUBBER CO., LTD., D. F. TWISS, and W. G. GORHAM (B.P. 335,559, 25.6.29).—A protective coating for paper, cardboard, etc. consists of an emulsion of rubber and a vegetable or mineral wax containing 20–95% of wax (calc. on the mixture); vulcanisation is unnecessary, since the presence of the wax prevents tackiness, and, further, the absence of zinc accelerators enables the composition to be used on food containers. A. J. HALL.

Machine for separating solids [and wool-fat] from liquids [wool-scouring wash]. J. and C. E. THOMSON, Assrs. to J. WINSLOE (U.S.P. 1,762,899, 10.6.30. Appl., 17.5.28. N.Z., 4.7.27).—See B.P. 304,718; B., 1930, 368.

Manufacture of artificial silk and the like. W. P. DREAPER (U.S.P. 1,780,346, 4.11.30. Appl., 3.1.27. U.K., 11.1.26).—See B.P. 273,354; B., 1927, 745.

Esterification of cellulose. F. BECKER, H. HEIMANN, and A. BAYERL, Assrs. to I. G. FARBEIND. A.-G. (U.S.P. 1,780,151, 4.11.30. Appl., 22.12.28. Ger., 24.12.27).—See B.P. 303,006; B., 1930, 504.

[Mechanism for] manufacture of waterproof fibrous products. N.V. DE BATAAFSCHE PETROLEUM

MAATS., Assees. of H. L. LEVIN (B.P. 319,212, 16.9.29. U.S., 17.9.28).

Collecting of artificial wool [from the spinning machine]. M. DASSONVILLE (B.P. 336,763, 26.9.29. Fr., 19.2.29).

Sodium sulphite or bisulphite (B.P. 317,040). **Alkalisng waste pulp-liquors** (B.P. 335,371). **Sulphite waste-liquors** (U.S.P. 1,764,600—1).—See VII. **Diaphragms for electrolytic cells** (B.P. 335,151).—See XI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Naphthenic sulphonic acids in the scouring of soiled fabrics and fibrous materials. G. PETROV (Masloboino Zhir.-Delo, 1929, No. 1, 34–42).—The oil-free sulphonic acids and their neutral salts have a detergent power greater than that of the soaps. The presence of sulphuric acid or sulphates has no effect on the detergent properties, which decreased with increase in the mol. wt. The application of the acids and their salts in the washing of fabrics and other materials is discussed. CHEMICAL ABSTRACTS.

Detergent value of soaps. PANTYUKOV.—See XII.

PATENTS.

Coloration of [cellulose ester and ether] textiles and other materials. BRIT. CELANESE, LTD. (B.P. 313,450—1, 11.6.29. U.S., 11.6.28. Addns. to B.P. 282,036; B., 1929, 169).—The method for applying to products containing cellulose ethers or esters fugitive colours from their solutions in organic solvents as described in the chief patent is suitable for permanently colouring (A) similar threads while they are travelling, and (B) fabrics and the like by printing, spraying, or stencilling methods. Polyhydric alcohols or their esters or ethers are particularly suitable as solvents, since they evaporate slowly; the ethylene, diethylene, and propylene glycols are among the numerous suitable solvents cited, and it is recommended that water should be added before use, equal in amount to that which would normally be absorbed by exposure to the atmosphere. A. J. HALL.

Production of coloured discharges. W. W. GROVES. From I. G. FARBEIND. A.-G. (B.P. 335,048, 7.9.29).—Goods impregnated with a 2:3-hydroxynaphthoic arylamide or other coupling component are printed with a paste containing a soluble leuco-ester of a vat dye, an insoluble chromate, and an alkali sulphite. On treatment with a diazo compound, without previous steaming, the vat dye shows as a coloured effect on coloured ground. Examples are: naphthol AS, printed with dibenzpyrenequinone leuco-ester, lead chromate, and potassium sulphite, and developed with diazotised 4-amino-4'-methoxydiphenylamine for yellow on a blue ground; naphthol AS/TR, printed with tetrabromoindigo leuco-ester, barium chromate, and potassium sulphite, and developed with diazotised 5-chloro-*o*-toluidine for blue on red. C. HOLLINS.

Production of discharge effects on textile materials. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 334,518, 29.4.29).—The action of discharge pastes,

particularly reducing pastes, and especially when used on acetate silk etc., is facilitated by first padding the dyed material with a non-volatile acidic substance, *e.g.*, oxalic acid, benzenesulphonic acid, citric acid.

C. HOLLINS.

Production of discharge effects on materials made of or containing cellulose esters or ether. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 334,543, 29.4.29. Addn. to B.P. 312,655; B., 1929, 678).—Instead of or in addition to chlorates, chromates are used as discharging agents. For coloured discharges a nitro-diarylamine, aminoanthraquinone, or acylaminoanthraquinone is added to the discharge paste. C. HOLLINS.

Treatment of cellulose ester materials. BRIT. CELANESE, LTD. (B.P. 313,971, 20.6.29. U.S., 20.6.28).—Materials containing cellulose esters are partly or completely saponified by subjecting them at 25–30° to the action of aqueous vapours of ammonia or volatile organic bases, *e.g.*, methylamine; the products show increased resistance to ironing and also to the delustring action of hot water, and have a higher moisture regain.

F. R. ENNOS.

Manufacture and treatment of textile or other materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 335,583, 16.3.29. Addn. to B.P. 332,231; B., 1930, 944).—The lustre-preserving treatments described in the prior patent are utilised in the production of printed lustre patterns on cellulose ester or ether materials (*e.g.*, cellulose acetate silk fabric is printed with a paste containing a solvent or gelling agent, dried, steamed with dry steam under pressure, and subjected to delustring by boiling in soap, whereby only the printed parts retain their original lustre), or in the manufacture of partly lustred woven and knitted fabrics (*e.g.*, fabric is woven from ordinary cellulose acetate yarn and yarn which has been treated with a gelling agent, and is then delustred).

A. J. HALL.

Treatment [delustring] of textile [artificial silk] fabrics and articles. BRIT. CELANESE, LTD., W. I. TAYLOR, and C. DREYFUS (B.P. 335,204, 15.6.29).—Methods described previously for delustring threads by means of abrasives (*cf.* B.P. 328,247; B., 1930, 763) are now applied to woven and knitted fabrics, whereby pattern lustre effects are obtained. A. J. HALL.

Rendering animal fibre materials immune from attack by moth and the like. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,886, 10.6.29).—The products obtainable by condensing a phenolsulphonic acid with an aralkyl compound, or a phenol with an aralkyl compound and subsequently sulphonating, are applied as mothproofing agents. Examples are products from: 4-chlorophenol-2-sulphonic acid and tetrachlorinated benzyl chloride; 2:4-dichlorophenol and 2:6-dichlorobenzyl chloride, sulphonated; β -naphthol and 2:4-dichlorotoluene- ω -sulphonic acid; β -naphthol and trichlorobenzyl chloride (3 mols.), sulphonated.

C. HOLLINS.

Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,762,233, 10.6.30. Appl., 14.7.28).—The felting properties of animal furs are

developed by a pretreatment with solutions of hydrogen peroxide and disodium hydrogen phosphate, either separately or together. Suitable solutions for the treatment of white coney and Australian rabbit skins are given.

A. J. HALL.

Manufacture and treatment of textile [crêpe] fabrics. BRIT. CELANESE, LTD., W. A. DICKIE, and R. W. MONCRIEFF (B.P. 335,180, 17.6.29).—Fabrics are woven from cellulose acetate yarns some of which have been already fully or partly shrunk by treatment with nitric acid (*d* 1.10–1.22) at 0–60° for a few minutes, and are then treated similarly in the woven state, whereby irregular shrinkage occurs and a crêpe or semi-crêpe appearance is produced (*cf.* B.P. 274,584; B., 1927, 747).

A. J. HALL.

Impregnation of textiles and other porous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,247, 20.6.29).—The materials are treated with solutions in organic solvents of water-insoluble cellulose derivatives (esters or ethers) and of bituminous substances, together with resins, plasticisers, colouring matter, or fillers, if desired, the quantity of solvent being sufficiently great to prevent closing of the pores.

F. R. ENNOS.

Improving the properties of artificial threads and films consisting of regenerated cellulose. W. MARSHALL (B.P. 335,176, 17.6.29).—The threads etc., and also textile materials coated or impregnated with regenerated cellulose, are treated with caustic soda solution (*d* not above 1.14, preferably 1.2–1.3) at 0–60° for a few minutes and then washed free from alkali with brine containing about 15% of sodium chloride (or other salts such as those mentioned in B.P. 323,307 (B., 1930, 281), whereby they gain improved softness, strength, elasticity, extensibility, and more uniform dyeing properties. Caustic potash is less effective than caustic soda (*cf.* B.P. 295,488; B., 1928, 745).

A. J. HALL.

Cleaning of rags by solvents. J. A. L. ROZIERES, and SOC. ANON. DES PROD. OLÉIQUES (B.P. 312,319, 17.5.29. Fr., 24.5.28).—Rags are continuously extracted in a digester by connecting in turn with a number of solvent holders containing solvent of varying grease content; after passing out of the digester the solvent is centrifuged to remove solid particles and returned to its appropriate holder. At the same time the solvent richest in grease flows continuously to a still, while the condensed solvent so obtained is returned to the holder used only for pure solvent, from which part is allowed to pass to the first container in order to maintain the grease content of the solvent contained therein at the desired value.

F. R. ENNOS.

Clearing cotton fibres out of digesters. BRIT. BEMBERG, LTD. (B.P. 335,457, 3.12.29. Ger., 15.12.28).—After running off the treating liquid, the fibres are thoroughly mixed with fresh liquid by the introduction of compressed air, which is then employed to expel the mixture of fibre and liquid.

F. R. ENNOS.

Mordanting and dyeing of materials made with or containing cellulose derivatives. H. DREYFUS (U.S.P. 1,779,464, 28.10.30. Appl., 22.11.27. U.K., 3.12.26).—See B.P. 287,204; B., 1928, 402.

Dyeing of cellulose derivatives. G. RIVAT, E. CADGÈNE, and C. DREYFUS, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,779,494, 28.10.30. Appl., 30.6.26).—See B.P. 273,692; B., 1928, 744.

Dyeing of cellulose esters and ethers. E. A. SPEIGHT, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,780,320, 4.11.30. Appl., 27.4.29. U.K., 30.5.28).—See B.P. 314,651; B., 1929, 716.

Ornamental fabric and its manufacture. C. DREYFUS and W. R. BLUME, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,780,645, 4.11.30. Appl., 26.6.28).—See B.P. 314,396; B., 1930, 1106.

Dyeing machinery. H. A. BLUNDELL, and BLUNDELL BROS. (LUTON), LTD. (B.P. 336,686, 23.7.29).

Dyeing machines for automatic fabric return. O. OBERMEYER (B.P. 336,724, 26.8.29).

Apparatus for liquid treatment of textile materials and the like. C. F. HAMMOND and W. SHACKLETON (B.P. 336,963, 22.7.29).

Drying apparatus [for clothes etc.]. C. B. COOKE (B.P. 336,679, 20.7. and 27.9.29).

Dyeing with vat dye enolic sulphuric esters (B.P. 334,921). Soluble compositions for dyeing or printing (B.P. 334,529).—See IV. Treatment of textile materials (B.P. 334,853).—See V. Sulphite waste-liquors (U.S.P. 1,764,600—1).—See VII. Fire-proofing of fabrics (B.P. 334,408).—See IX. Coloursing rubber oil substitute (B.P. 335,912).—See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Reaction of diphenylamine-sulphuric acid with nitrates in the presence of chlorides, with especial reference to its use in the determination of nitrates in arable soil. H. RIEHM (Z. anal. Chem., 1930, 81, 353—377).—In the reaction between nitric acid and diphenylamine in the presence of hydrochloric acid 1 mol. of nitric acid oxidises 1.5 mols. of the amine to the blue holoquinonoid salt. In the presence of excess amine two further reactions take place slowly: in the first place, the excess amine reduces the blue salt to a grey-green quinhydrone compound; and secondly, intensification of the blue colour occurs, possibly owing to the oxidation by atmospheric oxygen of the nitric oxide formed in the primary action. The stability of the blue colour is therefore at a maximum when equivalent quantities of nitrate and amine are present, and decreases with increasing excess of amine. A stable standard solution may be prepared by mixing equivalent quantities of diphenylamine and nitrate in the presence of sulphuric acid and diluting this solution as required with sulphuric acid of the same concentration. The reagent for a nitrate test is made by mixing 50 mg. of diphenylamine with 380 c.c. of water and sufficient sulphuric acid (which has previously been boiled with 5 g. of potassium chloride for 15 min.) to make 1 litre. For the analysis of a soil 5 g. are shaken with 50 c.c. of 2% potassium chloride solution for 30 min. and 2 c.c. of the filtered liquor are treated with 5 c.c. of the reagent, cooled, shaken, and set aside

for 2 hrs. The resulting colour is compared with that of solutions containing known amounts of nitrate in 2% potassium chloride after treatment with the reagent in the same way. Nitrites should be destroyed by addition of carbamide prior to making the test.

A. R. POWELL.

Deposition of vanadium pentoxide as catalyst on chamotte. I. E. ADADUROV and G. K. BORESKOV (J. Chem. Ind., Russia, 1929, 6, 208—209).—A solution of ammonium metavanadate, just acidified with hydrochloric acid, is boiled with chamotte. Precipitation is incomplete even after 1.5 hrs. CHEMICAL ABSTRACTS.

Deposition of silver metavanadate on chamotte. I. E. ADADUROV and G. K. BORESKOV (J. Chem. Ind., Russia, 1930, 6, 805—807).—Direct deposition is unsatisfactory; vanadium pentoxide is deposited first. CHEMICAL ABSTRACTS.

Determination of nitrous and nitric acids in concentrated sulphuric acid. H. A. J. PIETERS and M. J. MANNENS (Z. anal. Chem., 1930, 82, 218—224).—Nitrous acid is best determined colorimetrically with *m*-phenylenediamine and the total nitrogen by reducing the neutralised solution with Devarda's alloy and potassium hydroxide, and determining the resulting ammonia acidimetrically. A. R. POWELL.

Manufacture and use of calcium molybdate. H. C. MABEE (Canad. Dept. Mines, Ore Invest., 1928, 156—158).—Calcium molybdate for use in steel manufacture is readily prepared by roasting molybdenite at 600° until free from sulphur, and then heating the resulting molybdenum trioxide at 600° with finely powdered calcium carbonate. A. R. POWELL.

Reports of investigations: [Canadian] non-metallics laboratory. R. K. CARNOCHAN, C. S. PARSONS, and R. A. ROGERS (Canad. Dept. Mines, Ore Invest., 1928, 95—130).—Experimental work on the separation and purification of various non-metallic minerals, including asbestos, calcite, china clay, graphite, and garnet, is recorded. A. R. POWELL.

Precipitation of ferro-vanadium catalyst on chamotte. I. E. ADADUROV and G. K. BORESKOV (J. Chem. Ind., Russia, 1929, 6, 1365—1367).—A solution of ammonium metavanadate (0.1767 g. V_2O_5) is treated with ferric chloride (1.94 g.) and the precipitate is redissolved by addition of sulphuric acid (7.76 g.); the mixture is then heated with chamotte (10 g.), which, after being washed with cold water, retains 0.0635 g. V_2O_5 as $Fe_2O_3 \cdot 4V_2O_5 \cdot 4H_2O$. CHEMICAL ABSTRACTS.

Hydrogen for hydrogenation. A. MARKMAN (Masloboino-Zhir. Delo, 1929, No. 2, 17—20).—The use of electrolytic hydrogen is advocated.

CHEMICAL ABSTRACTS.

Determination of potash in dilute solutions. VON WRANGELL.—See XVI. **Sodium peroxide respirators.** STAMPE and HORN.—See XXIII.

See also A., Nov., 1337, Preparation and analysis of chromyl chloride (NÚÑEZ). 1369, Preparation of hydroxide jellies (PRAKASH and DHAR). 1380, Mixed catalysts (MITTASCH). Catalytic oxidation of ammonia (VON NAGEL). Mixed catalyst in ammonia synthesis (KEUNECKE and others). 1382, Electrolytic

preparation of silver oxide (NAYAR and MACMAHON). 1392, Determination of cyanides by copper sulphate (LORA Y TAMAYO). Determination of alkali hydroxides in the presence of alkali carbonates or of ammonia (SCHEWKET). 1393, Dermatological thallium salts (CLAVERA).

PATENTS.

Production of hydrogen cyanide. IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, J. McAULAY, W. B. FLETCHER, and H. A. T. MILLS (B.P. 335,585 and 335,947, [A, B] 27.3., 20.4., and 24.12.29, [A] 23.4.29).—(A) A mixture of a gaseous hydrocarbon, or gas containing a hydrocarbon, and excess of ammonia is passed through an unpacked reaction chamber, the inner-wall temperature of which is at least 1150° (preferably 1350—1450°). To prevent decomposition of the ammonia and hydrocarbon into their elements with consequent separation of carbon, the mixture should be brought to the reaction temperature as rapidly as possible and exposed for a short time only. The surface of the chamber should be free from materials liable to provoke decomposition of the gases, a suitable lining being glazed silica or sillimanite. (B) The gaseous hydrocarbon, or gas containing a hydrocarbon, is mixed, not with excess, but with the stoichiometric quantity of ammonia, a substantial amount of hydrogen being present as a diluent.

W. J. WRIGHT.

Manufacture of sodium sulphite or bisulphite solutions. ZELLSTOFFFABR. WALDHOF, and O. FAUST (B.P. 317,040, 5.7.29. Ger., 9.8.28).—Waste soda lyes, such as are obtained in artificial silk manufacture, are treated with sulphur dioxide or sulphurous acid, organic substances thus being precipitated and a clear solution of sodium sulphite or bisulphite obtained.

W. J. WRIGHT.

Production of sodium thiosulphate. H. HOWARD, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,760,137, 27.5.30. Appl., 5.12.25).—Sodium sulphide solution, e.g., waste sodium sulphide liquor, and sulphur dioxide, either as an aqueous solution or in the form of burner gases, are introduced into a solution of sodium sulphite at 60° at such rates that the sulphur dioxide content of the mixture is maintained in slight excess until the proportions of the reagents indicated by the equation: $2\text{Na}_2\text{S} + 3\text{SO}_2 + \text{Na}_2\text{SO}_3 = 3\text{Na}_2\text{S}_2\text{O}_3$ have been reached. Neither hydrogen sulphide nor free sulphur is thus appreciably liberated.

W. J. WRIGHT.

Alkalisng the waste liquors from the soda- or sulphate-pulp manufacture for the purpose of dry-distilling the same. E. L. RINMAN (B.P. 335,371, 24.9.29. Swed., 15.7.29).—After evaporation to *d* 1.25, the waste liquors are treated with oxide or hydroxide of barium or strontium, or a mixture of these, in an open vessel with stirring apparatus. The thick liquor obtained is evaporated to dryness and yields a pulverulent mass which does not sinter, this mass being then dry-distilled in a continuous furnace. Oxides or hydroxides of calcium, magnesium, aluminium, zinc, or iron may be added during alkalisng; if an inert substance, such as powdered carbon, be added, the subsequent dry distillation will be the more easily controlled.

W. J. WRIGHT.

Manufacture of products from sulphite waste liquors. (A) W. E. B. BAKER, (B) W. E. B. BAKER and F. G. ROBERTS, ASSRS. (B) to W. E. B. BAKER (U.S.P. 1,764,600—1, 17.6.30. Appl., [A] 7.4.23, [B] 26.5.23).—Sulphite waste liquor, after neutralising with lime to *p*_H 9, filtering, and concentrating to a 50% solid content, is (A) treated with zeolite, filtered from the calcium aluminium silicate formed, and acidified to the desired degree with acetic acid, yielding a solution of sodium ligninsulphonate suitable for treatment of leather and as a mordant; or (B) stirred with chromic acid solution until gelatinisation occurs, the product, after washing with water and dilute acid to remove sulphates and hydrated chromic oxide, consisting of lignin in the form of a reversible gel free from ligninsulphonic acid.

F. R. ENNOS.

Production of alkali sulphates. CHEMIEVERFAHREN GES.M.B.H. (B.P. 311,226, 22.4.29. Ger., 7.5.28. Addn. to B.P. 300,630; B., 1929, 718).—In a modification of the prior process, the crude potash salt, after roasting or grinding, is heated with mother-liquor from a previous operation, substantially free from ammonia, to hydrate the kieserite, and the cooled mixture is treated with ammonia, the temperature being kept constant. The potassium sulphate which settles out is treated with water to remove sodium sulphate, and the solution is returned to the process after hydration of the kieserite. The mother-liquor, containing ammonia and chlorides of ammonium, magnesium, and sodium, is treated with carbon dioxide, whereby magnesium ammonium carbonate is precipitated, and cooled to separate ammonium chloride, the final liquor being treated again with carbon dioxide to precipitate sodium bicarbonate. From the magnesium ammonium carbonate, magnesium carbonate may be obtained. If the magnesium ammonium carbonate is not removed, its mixture with ammonium chloride may be dried and calcined, so as to yield anhydrous magnesium chloride.

W. J. WRIGHT.

Production of sodium fluoride by the dry method. E. V. BRITZKE, W. I. BREMPPEL, and M. E. JAKUBOWITZ (B.P. 335,703, 29.8.29).—Hydrofluoric acid gas at about 70° is introduced into a tubular chamber in counter-current to calcined or caustic soda, which is conveyed through the tube by means of a central shaft with stirrers. The heat evolved is sufficient to evaporate the water formed in the reaction, so that dry, friable sodium fluoride is obtained, and by regulating the amounts of the components either the acid or neutral salt may be produced.

W. J. WRIGHT.

Manufacture of diammonium phosphate. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,008, 10.7.29).—Loss of ammonia, which results when a solution of diammonium phosphate is evaporated to dryness, is avoided by mixing the solution with solid diammonium phosphate and drying this mixture. In the preparation of diammonium phosphate by treating crude phosphate with sulphuric acid, the solution, after separation of calcium sulphate, is concentrated to *d* 1.4—1.5, cooled, and filtered. The phosphoric acid, free from calcium phosphate, thus obtained is neutralised with ammonia, the diammonium phosphate solution being treated as described.

W. J. WRIGHT.

Production of (A) alkali phosphates, (B) acid disodium pyrophosphate. METALLGES. A.-G. (B.P. 335,492 and 335,501, [A] 25.1.30, [B] 14.2.30. Ger., [A] 17.10.29, [B], 17.5.29).—(A) The production of alkali phosphates by fusing alkali sulphates with ferrophosphorus is effected without explosive reaction, such as may occur when a rotary furnace is employed, by heating the finely-ground components in an electric furnace. (B) A solution of phosphoric acid, mixed with sodium chloride, is evaporated *in vacuo* at 180–200°, higher temperatures being avoided to prevent the formation of insoluble metaphosphates. W. J. WRIGHT.

Production of alkaline-earth titanates. R. H. MONK and L. FIRING; the latter Assr. to J. IRWIN (U.S.P. 1,760,513, 27.5.30. Appl., 10.2.27).—An aqueous paste of freshly-precipitated titanium hydroxide is treated with an acetate of an alkali metal or ammonium to react with any sulphate ions, which are detrimental to the formation of titanates. After washing with water to remove the sulphate, an alkaline-earth carbonate, e.g., barium carbonate, is added in the form of paste to produce a compound such as BaTiO₃. Hydrochloric acid or barium chloride is added to accelerate the reaction and give the product a pure, white colour, and the mixture is finally heated to 750–880°.

W. J. WRIGHT.

Purification of zinc sulphate liquors. O. A. FISCHER, Assr. to R. H. CHANNING, JUN. (U.S.P. 1,761,782, 3.6.30. Appl., 10.2.27).—Iron salts are removed from zinc sulphate liquors by treating the latter with a mixture of minute bubbles of sulphur dioxide and air, produced by passing the mixed gases through a porous medium. In order to ensure complete oxidation of the iron salts, the sulphur dioxide should be caused to bubble through at least 2 ft. of the liquor. A precipitating agent is then added to the solution or, if desired, this may be added before the sulphur dioxide is introduced.

W. J. WRIGHT.

Production of aluminium salts. COLLOID-CHEM. FORSCHUNGS A.-G., Assees. of E. HERZFELD and H. WALKER (B.P. 336,181, 7.1.30. Switz., 7.1.29).—In the acid process for extracting aluminium, the difficulty in recovering iron-free aluminium salts from the mother-liquor is overcome by adding 96% alcohol. The almost pure aluminium sulphate, which is precipitated, is dissolved in water and reprecipitated with alcohol.

W. J. WRIGHT.

Mouldable product and its preparation. B. W. NORDLANDER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,761,740, 3.6.30. Appl., 14.5.29).—A thermoplastic material is obtained by fusing selenium sulphide with a suitable filler, such as asbestine, iron oxide, etc., at about 125° and then cooling; after pressing, the product may be hardened by "curing" at 80–90° for $\frac{1}{2}$ hr. An acidproof coating is produced on materials such as iron by suspending the selenium sulphide and a filler in a volatile liquid and spraying this on the iron. After evaporation of the liquid, the iron is heated at 105–115° and "cured."

W. J. WRIGHT.

Treatment of mixtures containing carbon dioxide and hydrogen for reducing or eliminating the hydrogen content, and the formation of carbon

monoxide therefrom. BRIT. CELANESE, LTD., W. BADER, and E. E. STIMSON (B.P. 335,632, 2.7.29).—The mixture of carbon dioxide and hydrogen is passed through a tube containing chromite, heated to 700–900°, by which means the hydrogen is largely eliminated; the treated mixture is then passed over heated carbon to yield carbon monoxide. W. J. WRIGHT.

Manufacture of mixtures of nitrogen and hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,524, 24.4.29).—The conversion of a solid, liquid, or gaseous carbonaceous mixture into a mixture of nitrogen and hydrogen by treatment with an electric arc together with air, hydrogen, or carbon dioxide has the disadvantage that the resultant mixture may contain undesirable amounts of methane or carbon dioxide. To avoid this, baffle plates are arranged at the end of the electric arc, thus promoting the intimate mixing of the gas by forming eddies, and preventing the formation of low-temperature zones, a temperature of at least 1400° being attained.

W. J. WRIGHT.

Manufacture of sulphur trioxide. CALCO CHEM. Co., Assees. of N. A. LAURY (B.P. 317,396, 9.7.29. U.S., 15.8.28).—A carrier for a vanadium catalyst used in the contact process is prepared from particles of a natural diatomaceous earth of a size larger than 6-mesh, these being fired at about 1000° and thereby acquiring great strength and becoming non-friable. W. J. WRIGHT.

Production of phosphorus oxychloride. J. S. DUNN, F. BRIERS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,065, 6.9.29).—Rock phosphate is treated with phosgene or with a mixture of carbon monoxide and chlorine at 350–600°, the calcium chloride formed being prevented from covering the phosphate, so that fresh surfaces are constantly exposed. This is effected by continuous grinding and stirring or by introducing coke, previously heated to 350°, or the process may be conducted in a rotary furnace in presence of pebbles. The mass may be intermittently washed with water, and the residual solid, after drying, again phosgenated.

W. J. WRIGHT.

Treatment of bromine and iodine. GOVETT, LTD., Assees. of E. GOVETT (B.P. 308,281, 20.3.29. U.S., 20.3.28).—A colloidal form of iodine ("iodine alpha") is prepared by causing iodine to react with an aqueous solution of tannic or gallic acid, and treating the colloidal hydrogen iodide produced with a nitrate or acetate of mercury or other suitable metal. The insoluble metallic iodide is decomposed with colloidal bromine, so as to liberate iodine, either free or combined with hydrogen, in solution, and this on evaporation yields iodine alpha. Colloidal bromine is preferably prepared by dissolving potassium bromate in tannic acid, removing the insoluble potassium tannate, and evaporating the hydrogen bromide solution to dryness. Alkali bromides and iodides are obtained in a colloidal form by treating the halogens with tannic or gallic acid, and causing the hydrogen halide to react with an alkali hydroxide; after removal of the potassium tannate, the hydrogen iodide is neutralised with sodium hydroxide. Both the colloidal halogens and the colloidal halides have therapeutic properties.

W. J. WRIGHT.

Stabilisation of hydrocyanic acid. H. LEHRECKE, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,780,198, 4.11.30. Appl., 23.6.26. Ger., 3.7.25).—See B.P. 254,747; B., 1927, 813.

Calcium chloride (U.S.P. 1,764,582).—See I. **Distillation products from vegetable substances** (B.P. 334,724). **Ammonia from coal gas etc.** (B.P. 334,619). **Sulphates and sulphur from gases** (B.P. 334,526). **Cyanogen compounds from gases** (U.S.P. 1,742,505).—See II. **Urea etc.** (B.P. 335,421 and 334,564).—See III. **Diaphragms for electrolytic cells** (B.P. 335,151). **Electrolysis of fused electrolytes** (B.P. 316,969). **Electrolytic production of oxygen and hydrogen** (B.P. 335,987).—See XI. **Fertiliser salts** (B.P. 335,175). **Fertiliser for seeds** (U.S.P. 1,762,294). **Weed-killer** (B.P. 335,203).—See XVI.

VIII.—GLASS; CERAMICS.

Devitrification of soda-lime-silica glasses. G. W. MOREY (J. Amer. Ceram. Soc., 1930, 13, 683—713).—A special study is made of that part of the ternary system $\text{Na}_2\text{O}, \text{SiO}_2\text{--CaO}, \text{SiO}_2\text{--SiO}_2$ which is of importance to the glass industry, viz., the range including 62—78% SiO_2 , 0—20% CaO , this region being represented by 87 glasses, instead of by 20 as in the previous work (cf. Morey and Bowen, B., 1925, 921). In a discussion of devitrification it is shown that the liquidus temperature is the only datum point which is solely a function of glass composition; at the liquidus the first trace of crystalline phase is in equilibrium with the glass; above it, the glass will dissolve any crystals which may have been formed, whilst crystals will tend to separate only below the liquidus. This and related phenomena are discussed at length. F. SALT.

Effect of alumina on the devitrification of a soda-lime-silica glass. G. W. MOREY (J. Amer. Ceram. Soc., 1930, 13, 718—724).—Starting with an initial glass of approximately the composition $\text{Na}_2\text{O}, \text{CaO}, 6\text{SiO}_2$, two series of experiments were carried out; in the first CaO was partly replaced by Al_2O_3 ; in the second alumina was added to the glass. In the first series 2% of alumina lowered the m.p. by about 80°, and the glass rapidly crossed the tridymite field and entered the field of $\text{Na}_2\text{O}, 3\text{CaO}, 6\text{SiO}_2$. On addition of further alumina, the liquid crossed this field and the CaO, SiO_2 field, until, on further replacement of CaO by Al_2O_3 , the mineral albite ($\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2$) finally became the primary phase, and the curve ended at the m.p. of albite, 1100°. In the second series of experiments tridymite was the initial phase; the glass containing 4% Al_2O_3 formed $\text{Na}_2\text{O}, 3\text{CaO}, 6\text{SiO}_2$ when crystallised at 1000°, but this decomposed into CaO, SiO_2 before the liquidus was reached. Wollastonite remained the primary phase as far as the curve was followed, to 9.1% Al_2O_3 and 1156°, at which point the temperature was rising rapidly. F. SALT.

Effect of magnesia on the devitrification of soda-lime-silica glass. G. W. MOREY (J. Amer. Ceram. Soc., 1930, 13, 714—717).—A series of experiments was carried out, beginning with a glass of the composition $1.15\text{Na}_2\text{O}, 0.84\text{CaO}, 6\text{SiO}_2$, in which tridymite separated as the primary phase. The addition of 2%

of magnesia lowered the m.p. considerably and the glass passed to the $\text{Na}_2\text{O}, 3\text{CaO}, 6\text{SiO}_2$ field, but with more than 3% of magnesia diopside became the primary phase and the m.p. rose rapidly. Complete replacement of CaO by MgO lowered the m.p., but tridymite remained the primary phase. F. SALT.

Dimensional changes caused in glass by heating cycles. A. Q. TOOL, D. B. LLOYD, and G. E. MERRITT (J. Amer. Ceram. Soc., 1930, 13, 632—654).—Nine lots of small samples of a medium flint glass were treated over the range 360—620°, the heating periods being varied from a few minutes at 620° to more than 1½ months at 360°. The dimensional changes produced by subjecting these glasses to heating cycles approaching the annealing range were measured, and the results are shown graphically. The total “permanent” change in length produced in this glass at 370—470° was found to be 17—18 μ per cm. R. J. CARTLIDGE.

Mechanical properties of some rolled and polished glass. E. M. GUYER (J. Amer. Ceram. Soc., 1930, 13, 624—631).—Young's modulus of elasticity, modulus of rupture, impact modulus of rupture, and wind-resistance were determined for various types of glass. The apparatus used in the four series of tests is described, and the results are tabulated. R. J. CARTLIDGE.

Use of polariscopes in the glass industry. F. W. PRESTON (J. Amer. Ceram. Soc., 1930, 13, 595—623).—The theory and construction of various types of polariscopes are discussed, and the application of such instruments in the detection of strain in glass is described. R. J. CARTLIDGE.

Design for enamel smelting furnace. R. D. COOKE (J. Amer. Ceram. Soc., 1930, 13, 655—657).—For corrosive enamels, which seriously reduce the life of the furnace lining, an open-hearth furnace is described, having the walls and the entire outside of the hearth exposed to the air. By using good-quality smelter brick in place of an insulating lining the life of the hearth was doubled and the cost reduced. R. J. CARTLIDGE.

Soluble salts in enamels. R. D. COOKE (J. Amer. Ceram. Soc., 1930, 13, 658—661).—Increasing amounts of borax, sodium carbonate, and sodium sulphate were added to portions of an enamel which had been washed free from soluble salts, and the effects noted. The cause of salts “setting-up” the enamel to proper consistency and the prevention or promotion of rusting of the steel during drying was also studied. It is assumed that these properties depend entirely on the nature and amounts of salts in solution in the enamel water. R. J. CARTLIDGE.

Determination of the mullite content of porcelain by chemical means and the relation between such mullite content and the properties of the porcelain. R. RIEKE and W. SCHADE (Ber. Deut. Keram. Ges., 1930, 11, 427—442).—A serviceable method of separating the mullite content from porcelain, giving comparable and sufficiently accurate values, is presented. The influence of the temperature and concentration of the hydrofluoric acid, and the effect of varying the time of the reaction were studied in further detail. The effect of the mineralogical composition and

the temperature and duration of firing on the mullite content was determined on a number of trial porcelains containing 40–60% of clay substance, 15–35% of quartz, and 15–35% of felspar. A study of the relation between mullite content and the physical properties of porcelain led to no definite result. Technical porcelains of different origins were tested for mullite content, from which conclusions were drawn regarding firing temperature and composition. The influence of the kaolin was determined on a number of porcelains of the same "rational" composition, but made with different kinds of kaolin. F. SALT.

Consistencies of raw terra cotta glazes. L. D. FETTEROLF (J. Amer. Ceram. Soc., 1930, 13, 675–682).—The glaze examined was composed of felspar 64.3%, flint 2.1%, whiting 8.9%, calcined zinc oxide 15.0%, and clay 9.7%. Tests were made on consistency changes of the glaze with ageing, and the effect of using an aged clay slip, a heat-treated glaze, and of the dissolution of salts from glaze materials on the causes of spontaneous thinning were noted. Additions of gum arabic in suitable amount served to stabilise glaze consistency satisfactorily. The results of adding acid, alkali, and water to such glazes are shown graphically.

R. J. CARTLIDGE.

Effect of temperature on cracking of plastic clay; temperature gradients inside the clay during steady heating in a saturated atmosphere and on drying at 100°. S. R. HIND and F. WHEELER (Trans. Ceram. Soc., 1930, 29, 314–325).—Test pieces of a standard purified Stourbridge clay containing 30.7–10.3% of moisture were subjected to steady heating in a saturated atmosphere, and the critical cracking temperatures were noted. Data obtained show that the critical temperature at which cracking commences is a function of the moisture content of the piece, and that with this clay no cracking occurs in a saturated atmosphere at any temperature up to the b.p. of water, when the moisture content of the piece is below 21.5% (on dry weight). R. J. CARTLIDGE.

Effects of firing temperature, kind of grog, and grading on the properties of firebrick material. A. J. DALE (Trans. Ceram. Soc., 1930, 29, 326–338).—Twenty-four sets of full-sized bricks were made of a single-bond clay admixed with special grogs, namely, "aloxite," "baddeleyite," silica glass, quartzite, and diaspore, which had previously been divided into coarse and fine grades. All the mixtures except diaspore contained 63% of grog and 37% of bond clay; with diaspore 25% of clay was used. Each set of bricks was divided into two parts, one portion going through the low-temperature building-brick fire, and the other to the firebrick kiln. The density, porosity, ordinary refractoriness, and refractoriness under a load of 50 lb./in.² of the mixtures were determined, and from data obtained it is concluded that when the amount of clay is not excessive, the value of a clay-bonded refractory is apparently governed by the nature of the grog, not of the bond-clay itself. R. J. CARTLIDGE.

[Separation of china clay and of garnet in] Canadian laboratory. CARNOCHAN and others.—See VII.

See also A., Nov., 1374, System K_2O - CaO - SiO_2 (MOREY and others).

PATENTS.

Tunnel kiln. A. McD. DUCKHAM (U.S.P. 1,780,119, 28.10.30. Appl., 16.7.28. U.K., 12.8.27).—See B.P. 285,323; B., 1928, 711.

Manufacture of refractory material. J. I. BRONN (U.S.P. 1,780,114, 28.10.30. Appl., 28.2.28. Ger., 15.7.26).—See B.P. 304,492; B., 1929, 247.

Glass-furnace forehearth. J. MONCRIEFF, LTD., and A. F. McNISH (B.P. 336,883, 6.1.30. U.S., 9.8.29).

Annealing furnaces [for glass]. W. A. MORTON (B.P. 336,596, 15.4.29).

Manufacture of cast glass plates and sheets. Y. BRANCART (B.P. 336,921 and Addn. B.P. 336,924, [A] 10.2.30, [B] 17.2.30. Belg., [A] 14.2.29, [B] 27.4.29).

[Frame for] manufacture of safety or unsplinterable glass. T. W. HOLT and J. F. W. STUART (B.P. 337,158, 2.10.29).

Manufacture of bonded abrasive articles. CARBORUNDUM Co., Assees. of H. C. MARTIN (B.P. 316,676, 31.7.29. U.S., 2.8.28).

Grading of abrasives for use in continuous glass-grinding machines, and apparatus therefor. PILKINGTON BROS., LTD., F. B. WALDRON, and J. H. GRIFFIN (B.P. 336,587–8, 12.7.29).

Tunnel kiln (U.S.P. 1,763,624).—See I. **Enamelling furnace** (B.P. 336,168). **Insulating material** (B.P. 315,265).—See XI.

IX.—BUILDING MATERIALS.

Application of the filtration method in volumetric analysis, especially in the analysis of Portland cement. H. T. BUCHERER and F. W. MEIER (Z. anal. Chem., 1930, 82, 1–44).—The method consists in adding the standard reagent to the test solution until no further precipitation can be observed, then filtering a small portion and testing one part of the filtrate with the reagent and another part with a standardised solution of the substance being determined. In this way a close approximation to the true result is obtained; a second trial is then made, adding a small deficiency of the precipitant without making a filtration and subsequently making tests after the addition of each succeeding drop until the filtrate ceases to give a precipitate. For the determination of calcium by the method, the neutral solution is treated with sodium acetate equivalent to 1–1½ times the amount of calcium present and titrated at 80° with oxalic acid. Magnesium may be determined with 8-hydroxyquinoline in a solution (100 c.c.) containing 20 c.c. of 2N-ammonium chloride and 10 c.c. of ammonia (d 0.88) at 80°; in this case the filtrate is coloured yellow when precipitation is complete. Both metals may be determined in the same solution, the calcium being first titrated with oxalic acid and then the magnesium with 8-hydroxyquinoline after addition of ammonia and ammonium chloride without filtering the calcium oxalate. Zinc and copper may be determined by titration of the ammoniacal solution with sodium sulphide at 20°; equally good results for zinc may be obtained

in acetic acid solution and for copper in sulphuric acid, and by working in these acid solutions copper and zinc may be determined successively in the same trial. Manganese may be titrated with sodium phosphate at 75° in a feebly ammoniacal 10% ammonium chloride solution, and iron and aluminium may be determined by precipitation of the cold solution containing 4 g. of sodium acetate per 100 c.c. with 8-hydroxyquinoline and titration of the excess of precipitant with bromine after filtration and acidification of the filtrate with hydrochloric acid. For the analysis of Portland cement, 1 g. is evaporated with 1:1 nitric acid to dryness and the residue is heated at 115°, cooled, and extracted with a 1:1 mixture of ether and absolute alcohol. The insoluble material is collected, washed with the extraction mixture, and digested with hydrochloric acid to obtain the silica in the usual way. The filtrate from the silica and the alcoholic solution are united and the iron and alumina removed with ammonia; calcium and magnesium are determined in an aliquot part of the filtrate by the methods described above, and the sum of the iron and alumina by the hydroxyquinoline titration. Iron alone is determined iodometrically in a portion of the solution obtained by dissolving the ammonia precipitate in hydrochloric acid.

A. R. POWELL.

Petrographic investigation of aluminous cements. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 352—358 B).—Fusion of a mixture containing 47% CaO, 44% Al₂O₃, and 9% SiO₂ gives a clear glass which devitrifies on heating at 1200° for 24 hrs. The devitrified material resembles porcelain in appearance and contains numerous spheruliths exhibiting needle-shaped double-refracting crystals radiating from the centre; under crossed nicols thin layers show either blue or yellow interference colours. The optical properties of devitrified calcium aluminates ranging from 3CaO,5Al₂O₃ to 3CaO,Al₂O₃ have been investigated: 3CaO,5Al₂O₃ forms prismatic doubly-refracting crystals, and CaO,Al₂O₃ a confused mass of twinned crystals exhibiting blue interference colours; 3CaO,Al₂O₃ forms isotropic octahedral crystals containing free calcium oxide; the stable modification of 5CaO,3Al₂O₃ forms anisotropic hexagonal crystals which exhibit only feeble double refraction, whereas the crystals of the labile modification resemble in appearance spheruliths. None of these substances forms true spheruliths such as are found in the cement. 2CaO,Al₂O₃ on fusion and devitrification gives a mixture of 3CaO,Al₂O₃ and 5CaO,3Al₂O₃, and a third substance of which the isotropic crystals radiate from nuclei; 3CaO,2Al₂O₃ crystallises in well-developed spheruliths which show the blue and yellow interference, and are apparently identical with those of the cement; the sintering temperature of this compound is about 1250° and the temperature of complete fusion 1300°.

H. F. GILLBE.

Injury to plaster due to osmosis. F. L. BRADY (Nature, 1930, 126, 684).—When lime plaster is applied to bricks containing magnesium sulphate, a semi-permeable membrane is precipitated in the brick and, as the wall dries out, magnesium sulphate concentrates behind the membrane, crystallises, and forces the plaster, together with a thin shell of brick, away from the wall.

Membranes of magnesium hydroxide capable of supporting osmotic pressures equal to 100 cm. of water have been precipitated in the walls of porous pots.

L. S. THEOBALD.

Asphalt tests. STRIETER.—See II.

PATENTS.

Treatment [fireproofing] of combustible substances [e.g., wood, fabrics]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,408, 28.9.29).—The materials are impregnated with a solution containing a combustion-retarding agent, e.g., ammonium phosphate, and a urea-formaldehyde condensation product. Other substances preventing crystallisation of the salt, e.g., starch or glue, may be added also, if desired.

A. B. MANNING.

Treatment of sawdust. E. ERLBACH (U.S.P. 1,764,249, 17.6.30. Appl., 12.8.26. Ger., 18.8.25).—Sawdust, made into a pulp with water containing caustic alkali, is pumped for about 2 hrs. through a pressure-proof system heated at about 300° at superatmospheric pressure, and the products of decomposition (acetic acid, methyl alcohol, acetone, and tar) are drawn off.

F. R. ENNOS.

Manufacture of [stringed] bricks. T. KOIDL and F. GORLICH (B.P. 337,231, 20.11.29. Austr., 20.11.28).

Moulding of artificial stone and imparting a finished surface thereto. F. T. WALKER (B.P. 337,074, 30.7.29).

[Compressed-air apparatus for] impregnation of timber. C. GOODALL (B.P. 337,133, 18.9.29).

Bituminous emulsions (B.P. 312,580). Emulsions with humic acid (B.P. 334,426).—See II. Casein-glue film (B.P. 314,344).—See XV. Immunising wood (B.P. 335,527).—See XVI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Reduction of iron ores with coal in coke ovens. E. V. BRITZKE and I. V. SHMANENKOV (Min. Suir., 1930, 5, 86—91).—Phosphorus, arsenic, vanadium, and titanium are practically completely removed. When more than 50% of ore is used the coke becomes pulverulent. Reduction is due to carbon, hydrogen, and methane. About 80—95% of the iron is reduced to the metal and 5—15% to ferrous oxide, whilst 1—5% remains unreduced.

CHEMICAL ABSTRACTS.

Reports of investigations: [Canadian] ore-dressing and metallurgical laboratories. [Beneficiation and low-temperature reduction of high-sulphur iron ore and the metallisation of the iron content of ilmenite.] R. J. TRAILL, W. R. McCLELLAND, and J. D. JOHNSTON (Canad. Dept. Mines, Ore Invest., 1928, 82—94).—The iron ores tested contained 53.35% Fe, 2.95% S, 9% SiO₂, and 5% CaO; roasting in a Herreshoff or rotary kiln furnace reduced the sulphur to a minimum of 0.8%, and subsequent reduction to sponge iron and magnetic separation gave a product containing a minimum of 0.6% S. Metallisation tests on an ilmenite containing 39% Fe, 31.75% TiO₂, 7.7% Al₂O₃, 7% SiO₂, and 2.6% MgO showed that 94% of the iron

could be reduced to metal at 990–1050° by means of coal in a rotary kiln type of furnace. Leaching of the product with 10% sulphuric acid gave a residue containing 75.8% TiO_2 , 6.27% Fe, and 0.8% C.

A. R. POWELL.

Loss in burning during steel heating. N. Y. TAITZ (J. Russ. Met. Soc., 1929, Mem., 288–326).—Data concerning surface oxidation during heating, rolling, and cooling are compared. A reducing atmosphere in the heating furnace does not appreciably decrease the loss. The metal should remain for a minimal time in the hottest zone; a magnesite bottom is recommended.

CHEMICAL ABSTRACTS.

Oxygen content of basic mild open-hearth steel before the addition of deoxidisers. A. K. SULIN (J. Russ. Met. Soc., 1929, Mem., 102–112).—Metal in a furnace heated by wood generator gas contained less oxygen than that in a furnace heated by crude oil. With a deeper hearth the oxygen content of the metal was lower. The oxygen content does not depend on the temperature, provided that the percentage of carbon is constant.

CHEMICAL ABSTRACTS.

Physical chemistry of steel manufacture. De-oxidation with silicon in the basic open-hearth process. C. H. HERTY, JUN., C. F. CHRISTOPHER, and R. W. STEWART (Min. Met. Invest. U.S. Bur. Mines etc. Co-op. Bull., 1930, No. 38, 1–172).—The amounts and types of silicates formed on deoxidation with silicon, the time required for their elimination, and their effect on the cleanliness of steel in the ladle have been investigated. There is formed at the slag-metal interface a layer of silicates which do not enter the slag unless a very long time is allowed for the re-boil. For the reaction $\text{Si} + 2\text{FeO} \rightleftharpoons \text{SiO}_2 + 2\text{Fe}$ the equilibrium constant $k = 1.65 \times 10^{-4}$. A number of general conclusions have been reached.

CHEMICAL ABSTRACTS.

Mechanical properties of Thomas steel and open-hearth steel. K. DAEVES (Stahl u. Eisen, 1930, 50, 1353–1356).—Average values of the mechanical properties (taken from a large number of tests) are compared for steels produced in the Thomas converter and in the Siemens-Martin furnace. For equal values of tensile strength Thomas steels have an elongation about 2% and a reduction in area about 4% greater than that of open-hearth steels; they have, however, a higher phosphorus and a lower carbon content. Thomas steel hardens more rapidly on drawing and cold-rolling and is therefore preferable for the manufacture of wires and drawn bars of all shapes, whereas open-hearth steel is to be preferred for sheet and deep drawing. The latter steel is less sensitive to ageing and its properties are rarely deleteriously affected by heat-treatment after cold work, whereas Thomas steel becomes brittle during normalising, and should be used, therefore, only when no subsequent heat treatment is to be given after cold work.

A. R. POWELL.

Prolonged loading tests on constructional steels at high temperatures. E. POHL, H. SCHOLZ, and H. JURETZKE (Arch. Eisenhüttenw., 1930–1, 4, 105–110; Stahl u. Eisen, 1930, 50, 1330–1331).—The minimum load required to cause creep at 300–350°

has been determined for a number of steels by subjecting them to a stress equal to about one third the yield-point load at the temperature of test and then gradually increasing this load until a slow, permanent, plastic flow is produced. Under these conditions a mild steel having a tensile strength of 35–44 kg./mm.² at 20° commenced to creep with a load of 15.2 kg./mm.² at 300°, 8.45 kg./mm.² at 400°, and < 3.2 kg./mm.² at 500°. The values for a 2% nickel steel (60 kg./mm.² at 20°) were > 20 kg./mm.² at 350°, 9.85 kg./mm.² at 450°, and 5.38 kg./mm.² at 500°. A molybdenum steel having the same tensile strength as the above carbon steel had a permanent strength of 11.2 kg./mm.² at 500°. By applying a load just below the permanent strength at the testing temperature for some time, the metal becomes appreciably stronger.

A. R. POWELL.

Effect of small quantities of copper on the resistance to corrosion of structural steel. O. BAUER, O. VOGEL, and C. HOLTHAUS (Mitt. Deut. Materialprüf., 1930, Spec. No. XI, 3–25; Chem. Zentr., 1930, i, 2963).—At the ordinary temperature the tendency to rust was only slightly decreased with increase in the copper content; at 50° no difference was usually observed. The solubility of steel in acid is markedly affected by the presence of 0.2–0.3% Cu: simultaneous presence of phosphorus diminishes resistance towards sulphuric acid.

A. A. ELDRIDGE.

Tensile properties and structure of drawn-steel wires in relation to the previous heat treatment. A. POMP and A. LINDENBERG (Stahl u. Eisen, 1930, 50, 1462–1467).—Steel wire with 0.6% C, 0.24% Si, 0.5% Mn, 0.019% P, and 0.022% S was heat-treated at various temperatures and quenched in air, in a lead bath, and in oil with a subsequent anneal, then drawn in 11 stages to a 92% reduction in area; at all stages of this procedure the tensile, bending, and fatigue strengths were determined and the structure was examined. The numerical data are reproduced graphically and briefly discussed. The results show that the effect of quenching temperature on the tensile strength is very small, provided that it is above 900°. The bending properties of air-cooled wire are best after a high-temperature anneal, and those of wire quenched in a lead bath when the latter is held at 490° and the metal is quenched from 935°. The resistance to fracture on repeated bending varies considerably with the nature of the heat treatment and with the degree of reduction in the subsequent drawing operation.

A. R. POWELL.

Presumed heterogeneity of martensite. E. MAURER and G. RIEDRICH (Arch. Eisenhüttenw., 1930–1, 4, 95–98; Stahl u. Eisen, 1930, 50, 1431).—The change in structure of hardened hyper- and hypo-eutectoidal steels during subsequent annealing to cause the disappearance of the martensite has been followed by micrographical examination after etching with alkaline sodium picrate. At the lower annealing temperatures, the precipitated cementite is in all cases regularly distributed throughout the specimen. At high temperatures the distribution of the cementite becomes irregular and only a few spheres of cementite are visible in the positions previously occupied by martensite needles; this phenomenon is due to the segregation of

the precipitated cementite along the edges of the needles forming a scum of gradually increasing thickness, which subsequently agglomerates into a few spheroidal aggregates of cementite. On etching with alcoholic nitric acid, hardened hypo-eutectoidal steels reveal a structure consisting of dark needles and flakes in a light ground-mass of fine needles; the dark constituent, which increases in quantity with decreasing carbon content, is darkened by etching with alkaline sodium picrate, whereas the light-coloured constituent remains unchanged in this reagent. The former, therefore, behaves similarly to the martensite needles in hyper-eutectoidal steels which have been annealed at 100–120°, and the latter similarly to unannealed martensite. Rapid quenching and a high manganese content prevent the formation of the dark constituent. In hardened steels with 0.2% C or less, the structure consists of ferrite and “annealed martensite” only; this accounts for the absence of a contraction in volume when the steel is annealed at 100°.

A. R. POWELL.

Iodometric determination of vanadium in special steels and in ferrovanadium. W. WERZ (Z. anal. Chem., 1930, 81, 448–450).—The steel (1–3 g.) is dissolved in 50 c.c. of phosphoric acid (d 1.7), 5 c.c. of nitric acid (d 1.2), and 175 c.c. of water, and the solution is boiled with 1 g. of ammonium persulphate for 15 min. After cooling to 70°, the permanganic acid formed is destroyed by adding 5–10 c.c. of 1% oxalic acid. The solution is then cooled to room temperature and treated with potassium iodide; the liberated iodine is titrated with 0.05*N*-sodium thiosulphate (1 c.c. \equiv 0.00255 g. V). Chromium does not interfere.

A. R. POWELL.

Determination of rust-resistance of special steels. V. DUFFEK (Korrosion u. Metallschutz, 1929, 5, Beih., 32–34; Chem. Zentr., 1930, i, 2308).—Measurements of the potential of nickel- and chromium-steels in *M*-ferrous sulphate solution gave results differing from those of the copper sulphate test. The latter gives results comparable with those of the former only when 0.1*M*-copper sulphate and 0.1*N*-sulphuric acid are used.

A. A. ELDRIDGE.

Protection against corrosion by treatment with phosphate. E. RACKWITZ (Korrosion u. Metallschutz, 1929, 5, 29–32; Chem. Zentr., 1930, i, 2307).—Iron alloyed with more than 6% of another metal cannot be parkerised. The process is described. The protective layer of insoluble phosphates is markedly adsorbent towards oils etc.

A. A. ELDRIDGE.

Hydrometallurgical treatment of high-grade iron-copper sulphide concentrates. R. J. TRAILL, W. R. MCCLELLAND, and J. D. JOHNSTON (Canad. Dept. Mines, Ore Invest., 1928, 132–153).—Ferric chloride leaching tests were made on a sulphide concentrate containing 26.5% Fe, 20% Cu, 9.1% Zn, 0.67% Pb, 30% S, 11.22 oz./ton Ag, and 0.8 oz./ton Au. Direct leaching gave almost as good extractions as leaching after a preliminary reducing roast with sulphur; over 90% of the zinc, copper, and silver were extracted and the residue contained practically all the gold in a form readily amenable to cyanidation. A good grade of copper precipitate was obtained, using scrap electrolytic iron as the precipitant. A preliminary leach of the

concentrate with saturated brine slightly acidified with hydrochloric acid removed over 96% of the lead with about 11% of the zinc, but no copper nor precious metals.

A. R. POWELL.

Treatment of mixed concentrates from base-metal sulphide ore. W. E. HARRIS (Canad. Dept. Mines, Ore Invest., 1928, 154–155).—In the treatment of roasted zinc-copper ores with sulphuric acid followed by electrolysis to remove the copper from the leach liquors, the copper content can be reduced to 0.8% with 6% of free acid before “burning” is observed in the deposit. Treatment of the insoluble residue by the Waelz process results in the elimination of 97–98% of the zinc in the form of an oxide containing over 56% Zn and only 0.5% Cu, leaving the greater part of the copper and all the gold and silver in the residue.

A. R. POWELL.

Reports of investigations: [Canadian] ore-dressing and metallurgical laboratories. [Flotation and cyanide tests.] C. S. PARSONS, A. K. ANDERSON, and J. S. GODARD (Canad. Dept. Mines, Ore Invest., 1928, 5–82).—A detailed account with experimental results is given of work done on the flotation of various simple and complex lead, copper, zinc, and molybdenite ores and on the cyanidation of five complex gold ores.

A. R. POWELL.

Effect of deoxidation and mould conditions on the tensile properties of carbon-steel castings. J. V. McCRAE and R. L. DOWDELL (Bur. Stand. J. Res., 1930, 5, 265–277).—Steel castings having elongation 24% and reduction of area 35% may be obtained by deoxidising with 1 lb. of aluminium per ton of steel shortly before casting; the tendency towards low ductility is avoided by using a steel containing 0.10–0.17% C, 0.60–0.70% Mn, and 0.30–0.40% Si. Moderate additions of vanadium and nickel, and heat treatment, have but little influence on the low ductility, and ferro-alloys of boron, zirconium, silicon, aluminium, or titanium are inferior to aluminium as deoxidants, and also reduce the ductility. Green-sand moulding is, in general, desirable, but tends to cause porosity, which, however, can be largely eliminated by control of the moisture content and permeability of the sand.

H. F. GILLBE.

Resistance of steels to abrasion by sand. S. J. ROSENBERG (Bur. Stand. J. Res., 1930, 5, 553–574).—The ball mill, the sand blast, and the Brinell machine have been compared as to their suitability for the testing of the resistance of metals to abrasion. The Brinell machine proved to be generally satisfactory. The resistance to wear of normalised, annealed, hardened, and tempered carbon steels increases with their hardness. Increasing the temperature of annealing resulted in a slight increase in resistance to wear, the increase being more marked in a low-carbon than in a high-carbon steel. Abrasion in this type of test is caused by the gradual breaking up of the individual grains, and not by the removal of separate grains in their entirety, and this action is accompanied by local strain-hardening.

W. E. DOWNEY.

Metallographic investigation of cast-iron pipe. V. N. SVECHNIKOV and K. F. STARODOUDOV (Messenger

ind. (russe) mét., 1928, No. 1, 94—108).—In defective pipes there were accumulations of fine flakes of graphite near the periphery, and fine-grained graphite was found between dendritic crystals of ferrite.

CHEMICAL ABSTRACTS.

Bearing bronzes with additions of zinc, phosphorus, nickel, and antimony. E. M. STAPLES, R. L. DOWDELL, and C. E. EGGESCHWILER (Bur. Stand. J. Res., 1930, 5, 349—364).—Tests of the resistance to impact, wear, and repeated pounding, and of the Brinell hardness have been made with copper-tin-lead bearing bronzes with and without zinc, phosphorus, nickel, and antimony. Alloys containing 4% Zn or 0.05% P are superior to the simple ternary alloys and to those containing nickel or antimony; 2% Ni reduces the wear-resistance, but gives increased resistance to pounding at the ordinary temperature, whilst 1% Sb reduces the impact-resistance without appreciably affecting the other properties. Three-dimensional models have been prepared showing the variation of the mechanical properties with the content of copper, tin, and lead.

H. F. GILLBE.

Occlusion of lead and copper in non-ferritic alloys by metastannic and metantimonic acids. S. DEL MUNDO (Philippine J. Sci., 1930, 43, 403—407).—The metastannic and metantimonic acids produced when white metal containing tin and antimony in a lead base is treated with nitric acid contain 1—3% of adsorbed lead. In the procedure recommended for the rapid routine determination of lead in such alloys, the metal (0.5 g.) is heated with 10 c.c. of concentrated sulphuric acid until reaction is complete, 50 c.c. of water are added, and the precipitate of lead sulphate, after being washed first with a 5% solution of sulphuric acid and then with water, is extracted with hot acid ammonium acetate and the lead determined as chromate in the usual way. This method is not suitable for alloys rich in tin, but the metastannic and metantimonic acids formed from these alloys usually contain only 0.1—0.5% of adsorbed lead and copper. R. CUTHILL.

Determination of phosphorus in aluminium. STEINHÄUSER (Z. anal. Chem., 1930, 81, 433—438).—Blackening of silver nitrate by the gases evolved when aluminium is dissolved in acid is not a sure indication of the presence of phosphorus, as silicon hydrides produce a similar effect. Objection is raised to all of the usual methods of determining phosphorus in aluminium, and the following method is recommended: 30—50 g. of metal are dissolved in hydrochloric acid in a vessel through which a current of hydrogen is passed and the gases are burned at a small jet beneath a funnel through which the products of combustion are drawn into 0.25% sodium hydroxide solution. When all the metal has dissolved, the funnel is rinsed with 50 c.c. of 3% hydrofluoric acid to remove the silica deposit and the acid and alkaline solutions are evaporated with a few drops of sulphuric acid to expel fluorine. The phosphorus is then precipitated as phosphomolybdate in the usual way. A. R. POWELL.

Solubility of copper in aluminium in the solid state. P. Y. SALDAU and N. ANISSIMOV (Zapiski Gornovo Inst., Moscow, 1929, 7, No. 3, 139—149).—

The limit of solubility at 15° is at 2.7% Cu. The hardness of the quenched metal increases from 2.7% Cu to a minimum at 6% Cu. CHEMICAL ABSTRACTS.

Corrosion of duralumin in relation to its composition. W. KROENIG (Korrosion u. Metallschutz, 1930, 6, 25—34; Chem. Zentr., 1930, i, 3235).—The binary aluminium-copper alloys showed moderate resistance to corrosion, which resistance was diminished by alternate immersion in sea-water and drying, when intercrystalline corrosion occurred. Magnesium abolishes these properties. Iron diminishes the resistance, whilst manganese (up to 0.6%) is advantageous.

A. A. ELDRIDGE.

Diffusion in cast bismuth-antimony alloys. G. MASING and H. OVERLACH (Wiss. Veröff. Siemens-Konz., 1930, 9, 331—338).—Homogenisation of such alloys on solidification causes an increase in the external measurements of the test piece. It is concluded that this phenomenon is due to diffusion in the solid state.

W. E. DOWNEY.

Behaviour of heat-stable alloys towards sulphur, and a new sulphur-resistant alloy. H. GRUBER (Heraeus Festschr., 1930, 45—51; Chem. Zentr., 1930, i, 2305).—Ordinary heat-resistant alloys are but slightly resistant towards hydrogen sulphide. Addition of chromium increases the resistance of iron towards sulphur; nickel is readily attacked. Addition of aluminium diminishes the corrosion. A resistant chromium-nickel alloy is specified.

A. A. ELDRIDGE.

Recovery of precious metals from residues. A. IEVINŠ (Latvij. Univ. Raksti, 1929, 1, 1—12).—The dry-fusion method for the recovery of precious metals gives better results than the chloride or cyanide methods when the total content of gold and silver is high, i.e., 1—3%. A single fusion may result in the separation of 99.92% of the whole. For complete separation the following factors must be taken into consideration: (a) The viscosity of the melt should be reduced by employing a flux containing calcium and magnesium carbonates. (b) The melt must be kept a sufficient length of time at 1350—1400°. A melt of composition 10SiO₂, 0.83Al₂O₃, 1.10FeO, 9.25CaO, 0.58MgO, and 1.6Na₂O gave a satisfactory yield. (c) The higher the percentage of precious metal in the charge the more complete is its separation, and not only the relative but also the absolute content of precious metal in the slag is smaller. (d) The richer the charge the smaller is the ratio (Au + Ag) : Pb necessary to obtain slags with equal content in noble metal. (e) By repeated fusion of the slag with 10% of litharge a further 20—40% of the precious metal contained in the slag can be separated.

M. S. BURR.

Improvement of the soldering of tramway cables. J. N. ALCÁZAR (Anal. Fis. Quím., 1930, 28, 1050—1054).—The hardness (Brinell) of a soldered cable joint is increased if, instead of being air-cooled, the joint be cooled in water at 25° (or, better still, at 3°): also the use of a copper-zinc-silver solder of lower m.p. than that of the usual copper-zinc solder is advantageous. The deformation on flexion is but little altered by this treatment.

H. F. GILLBE.

Electrostatics of flotation. H. B. BULL (Coll. Symp. Ann., 1929, 7, 115—118).—The *P.D.* established by galena falling through solutions of thorium, chromium, silver, lead, or cobalt nitrate of varying concentrations were determined. The "toxic" effect of thorium and chromium is not related to the charge on the galena, but that of silver, lead, and cobalt ions is attributed to an increase of that charge.

CHEMICAL ABSTRACTS.

Molybdenum and its applications. S. L. MALOWAN (Chem.-Ztg., 1930, 54, 893—894.)

Calcium molybdate. MABEE.—See VII. **Electrotyping solutions.** HALL and BLUM.—See XI.

See also A., Nov., 1354, **Change of resistance produced in metals by cold-drawing** (TAKAHASHI). 1359, **Cadmium-zinc alloys** (STOCKDALE). **Lead-antimony alloys** (SOLOMON and JONES). **System lead-tin** (HONDA and ABÉ). 1360, **Vanadium steels** (ÖYA). **Resistance and hardness of lead-thallium alloys** (TAMMANN and RÜDIGER). 1381, **Electrocrystallisation of copper** (KISTIakovSKI and others). 1382, **Preparation of pure electrolytic nickel** (FINK and ROHRMAN). **Electrodeposition of metals from liquid ammonia solutions of their salts** (AUDRIETH and YNTEMA). 1392, **Determination of silver by titration with thiocyanate** (HOLWECH). 1394, **Electrodetermination of nickel, cobalt, and zinc** (TOUGARINOV). 1395, **Electromagnetic separator for mineral powders** (HALLIMOND).

PATENTS.

Ore[roasting] furnace. C. T. KNIGHT (U.S.P. 1,764,313, 17.6.30. Appl., 29.10.28).—The furnace comprises a horizontal cylinder provided with an axial shaft carrying a spaced series of band wheels bound together with a number of spiral strips provided with a series of metal brushes at regular intervals; by rotating the axis of the cylinder the charge is caused to travel through the furnace and the walls are kept clean by the scouring action of the brushes. A. R. POWELL.

Furnaces and/or apparatus for annealing or heat-treating metal etc. A. SMALLWOOD and J. FALLON (B.P. 334,965, 25.4.29).—The furnace comprises a long, horizontal heating chamber containing a series of parallel tubes in which the goods are heated out of contact with the heating flames. Each tube is connected to a similar tube outside the furnace, which serves for cooling the charge, and finally means are provided for ejecting the charge into a quenching bath without its coming into contact with the air. An additional tube is provided within the heating chamber for preheating inert or reducing gases to be passed into the annealing tubes. A. R. POWELL.

Manufacture of wrought iron. A. M. BYERS Co., Assees. of A. H. BEALE and (A, C, D) J. ASTON (B.P. 315,826—9, 20.6.29. U.S., 19.7.28).—Molten steel is (A) passed through a slag bath, (B) poured over a slag bath, (C) poured into a bath of puddle slag, or (D) bessemerised to 0.1% C and poured through a ferrous silicate slag. In all cases the metal is worked up in the usual way to obtain an ingot of wrought iron. [Stat. ref. in (A—D).] A. R. POWELL.

Manufacture of malleable iron. BRIT. THOMSON-HOUSTON Co., LTD.; Assees. of I. R. VALENTINE (B.P. 315,724, 16.7.29. U.S., 16.7.28).—White cast iron is heated at 1000° for 4 hrs. to cause the maximum amount of carbon to enter into solid solution, cooled to below 700°, reheated at 800—850°, cooled abruptly to 700—750°, and maintained within this range for 4 hrs. with occasional abrupt changes of temperature of about 10°. This treatment causes complete graphitisation of the carbon. A. R. POWELL.

Stainless steel and the melting thereof. W. E. MARTIN and J. A. BERLYN (B.P. 336,024, 22.7.29).—The charge containing the requisite amount of nickel and chromium is heated in a crucible at such a rate that the temperature increases at the rate of 400° in 15 min. and the charge is poured at above 1800° after deoxidation with aluminium, with or without a small amount of chromium hydroxide to compensate for the loss of chromium during melting. A. R. POWELL.

Iron or steel alloys [manganese steel]. H. WADE. From TAYLOR-WHARTON IRON & STEEL Co. (B.P. 336,091, 4.10.29).—A steel containing 0.3—0.85% C, up to 4% Ni, < 0.4% Si, 10—15% Mn, and up to 8% Cr is claimed. After shaping, the article is annealed at 800—1150° and air-cooled. [Stat. ref.] A. R. POWELL.

Stable-surface alloy steel. R. P. DE VRIES, Assr. to LUDLUM STEEL Co. (U.S.P. 1,759,605—6, 20.5.30. Appl., [A] 20.1.26, [B] 22.1.26).—(A) The alloy consists of iron with 0.5—5% Cu, 0.5—6% Si, 0.1—6% Al, and 0.1—5% Ti, the sum of the silicon and aluminium being 2—6.1%. (B) The steel contains 1.5—6% Al, 0.5—6% Cu, and 0.5—1% C. These steels resist scaling up to 1000°. A. R. POWELL.

Heat-resistant metal [galvanised iron or steel] sheet. E. R. WEHR and C. C. MAHLIE, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,764,132, 17.6.30. Appl., 13.9.26).—The galvanised coating contains more than 2% Al (preferably about 8%); it remains untarnished when heated at 350° in air and is free from spangle and brittleness. A. R. POWELL.

Mineral flotation apparatus. R. LORD, Assr. to SOUTHWESTERN ENG. Co. (U.S.P. 1,761,136, 3.6.30. Appl., 1.9.26).—The apparatus comprises a hollow, frustoconical aerating rotor the smaller end of which dips into the pulp container; the upper end carries an apertured head connecting with the driving shaft. The aerator is provided with lower and upper slots and has, internally, a series of broken spiral riffles and, externally, vertical riffles. By rotating the apparatus at a high speed, air is drawn into the aerator through the head slots and thoroughly intermingled with the pulp by the action of the riffles. Ejection of froth from the pulp container is prevented by a series of vertical baffles regularly disposed around, but some distance away from, the aerator. A. R. POWELL.

Production of acid-proof etching grounds on printing plates. A. AXTHELM (B.P. 334,629, 13.6.29).—The plate is covered with powdered asphalt or resin and then placed in a chamber in which it is exposed to the vapour of alcohol or other solvent, which is distributed over its surface by means of a small fan in such a

way that a small amount of the solvent condenses on the surface of the plate, thus converting the powder thereon into a smooth, acid-resisting film which rapidly dries when the plate is removed from the chamber.

A. R. POWELL.

Production of finely-divided metals from metal carbonyls. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,007, 10.7.29).—A mixture of carbonyl vapour and inert gas is introduced into a vessel with heated walls and provided with a mechanism to impart a gentle whirling motion to the gases; simultaneously a stream of hot carbon monoxide is passed into the vessel so as to cause decomposition of the carbonyl out of contact with the walls of the vessel. A. R. POWELL.

Production of metals [chromium] and alloys. T. R. HAGLUND (U.S.P. 1,764,511, 17.6.30. Appl. 28.1.28. Swed., 1.2.27).—Briquettes of chromium oxide and carbon are smelted below a layer of slag in an electric arc furnace, the amount of carbon being so regulated that the slag contains at least 35% Cr_2O_3 and the current density being kept below 2.5 amp./cm.^2 of cross-section of electrode. After the charge is tranquil a further part of the chromic oxide is reduced from the slag by adding silicon or a silicon alloy.

A. R. POWELL.

Metallisation [with nickel] of electric non-conducting and/or inert base materials. J. G. A. RHODIN, and BRIT. METALLISING CO., LTD. (B.P. 334,510, 29.5. and 28.1.30).—The base material is heated at 350° and immersed in a solution of 10 vols. of nickel carbonyl in 90 vols. of trichloroethylene or tetrachloroethane, whereby it becomes coated with a film of metallic nickel containing carbon. The article is then heated to redness and subsequently electroplated with nickel, chromium, or other suitable metal.

A. R. POWELL.

Electrolytic system [for recovery of metals]. J. W. SCOTT, ASSR. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,764,650, 17.6.30. Appl., 23.12.26).—A number of pairs of tanks containing electrolyte and electrodes is electrically connected in series, and each pair is interconnected by a coupling, permitting electrolyte to pass from one tank to the other and constituting an auxiliary electrolyte cell, provided with cathode and anode, for recovery of metal within the coupling. J. S. G. THOMAS.

Electrolytic separation of copper. A. MOZER (B.P. 336,109, 18.10.29. Ger., 29.10.28).—A bath comprising a solution of cuprous iodide in potassium iodide is claimed; e.g., with a solution of 60 g. of cuprous iodide and 600 g. of potassium iodide in 1 litre of water a current density of 400 amp./m.^2 can be used at 0.3 volt. Addition of 5–10 g./litre of colloids, e.g., tannin or glue, improves the brightness and smoothness of the copper deposit.

A. R. POWELL.

Reduction of ores. F. L. DUFFIELD (U.S.P. 1,779,411, 28.10.30. Appl., 21.9.28. U.K., 10.10.27).—See B.P. 304,174; B., 1929, 287.

Recovery of metal values from their ores or from metalliferous materials. A. E. WHITE. From MEYER MINERAL SEPARATION CORP. (B.P. 336,770, 30.9.29).—See U.S.P. 1,730,584; B., 1930, 9.

Manufacture of metallurgical fuel. G. JAKOVA-MERTURI, ASSR. to CARBONISATION SOC. GÉN. D'EXPLOIT. DES CARBONES (U.S.P. 1,779,744, 28.10.30. Appl., 4.4.28. Fr., 11.4.27).—See B.P. 288,551; B., 1929, 915.

Steel alloy. L. M. BROWN (Re-issue 17,846, 28.10.30. of U.S.P. 1,643,054, 20.9.27).—See B., 1927, 847.

Extraction of tin from ores or materials containing tin. E. A. ASHCROFT (U.S.P. 1,780,106, 28.10.30. Appl., 5.5.28. U.K., 7.7.27).—See B.P. 295,805; B., 1928, 789.

[Anodes for recovery by] electrolysis of zinc from impure sulphate solutions. U. C. TAINTON (U.S.P. 1,759,493, 20.5.30. Appl., 1.4.26. Renewed 13.5.29).—See B.P. 280,103; B., 1928, 21.

Electrolytic manufacture of metal sheets and tubes. E. KELSEN (B.P. 314,444, 27.6.29. Austr., 27.6.28).

Centrifugal extractor (U.S.P. 1,764,214). Filtration apparatus (U.S.P. 1,762,738).—See I. Aluminium salts (B.P. 336,181).—See VII. Carburisation of metals (U.S.P. 1,763,248). Resistance furnaces (B.P. 336,078). Soldering of aluminium (U.S.P. 1,763,417). Arc-welding (B.P. 307,834). Electrolysis of fused electrolytes (B.P. 316,969).—See XI.

XI.—ELECTROTECHNICS.

Addition agents in copper electrotyping solutions. R. O. HALL and W. BLUM (Bur. Ständ. J. Res., 1930, 5, 767–773).—Phenolsulphonic acid is the best addition agent. Its use permits the electrotypes to be produced in much shorter time and to be harder than those obtained in ordinary baths. A suitable bath is described and the best conditions of temperature and current density are given. W. E. DOWNEY.

Treatment of sulphide-ore concentrates. HARRIS. —See X. Resistance of rubber and gutta-percha stored under water. CURTIS and SCOTT.—See XIV.

See also A., Nov., 1354, **Change in resistance produced in metals by cold-drawing** (TAKAHASHI). 1360, **Resistance and hardness of lead-thallium alloys** (TAMMANN and RÜDIGER). 1381, **Electrocrystallisation of copper** (KISTIAKOVSKI and others). 1382, **Pure electrolytic nickel** (FINK and ROHRMAN). **Preparation of silver oxide** (NAYAR and MACMAHON). **Electrodeposition of metals from liquid ammonia solutions of their salts** (AUDRIETH and YNTEMA). 1394, **Electrodetermination of nickel, cobalt, and zinc** (TOUGARINOV). 1395, **Electromagnetic separator for mineral powders** (HALLIMOND).

PATENTS.

Electric furnace method [for carburising metallic charges]. W. E. MOORE, ASSR. to PITTSBURGH RES. CORP. (U.S.P. 1,763,248, 10.6.30. Appl., 12.4.29).—Gas under pressure is forced up through the furnace bottom comprising a porous layer of carbonaceous material upon which the charge rests. (Cf. U.S.P. 1,674,982; B., 1928, 645.) J. S. G. THOMAS.

[Conveyor enamelling] electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of C. L. IPSEN

and J. L. McFARLAND (B.P. 336,168, 18.12.29. U.S., 18.12.28).—In an electric furnace comprising a regenerative chamber and a hair-pin-shaped heating chamber, the walls and floor of which are covered with resistors, means are provided for conveying suspended articles in parallel adjacent paths through the regenerator to and from the heating chamber. J. S. G. THOMAS.

Electric furnace. A. D. KEENE, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,763,239, 10.6.30. Appl., 29.4.24).—A resistor unit supporting device is described. J. S. G. THOMAS.

Electric resistance furnaces. W. J. MILLAR, and ELECTRIC FURNACE CO., LTD. (B.P. 336,097, 8.10.29).—A recess is formed in the furnace brickwork at each end of the resistors and holes are provided in a metallic casing, enclosing the brickwork, for the admission of air into contact with the ends of the resistors, convected air being discharged through a substantially vertical channel between the brickwork and casing. J. S. G. THOMAS.

Heating device for attaining high temperatures by means of electric heating resistances. F. NEUMANN (B.P. 312,932, 31.5.29).—Resistance wire, wound upon a refractory ceramic material rich in alumina, is enclosed within a casing of the same material. J. S. G. THOMAS.

[Cooling of] electrical resistance [annealing] furnaces. P. CARLBERG (B.P. 336,078, 23.9.29).—A cooling medium, *e.g.*, air and/or water, is passed through the tubular resistances of the furnace. J. S. G. THOMAS.

[Cooling of] induction furnace. G. B. TJOFLAT, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,763,200, 10.6.30. Appl., 13.9.28).—Induction coils of solid section are interposed between, and in heat-conducting relation to, the turns of an induction coil having a longitudinal passage way. J. S. G. THOMAS.

[Fan for] electric ovens. L. W. WILD, and WILDBARFIELD ELECTRIC FURNACES, LTD. (B.P. 334,621, 11.6.29).—Air within the oven is collected axially by a centrifugal fan and projected horizontally against the oven walls. J. S. G. THOMAS.

Promoting chemical reactions and physical processes by the use of a high-frequency rotary electric field. W. LÜDKE (B.P. 315,367, 12.7.29. Ger., 12.7.28).—Linear electromagnetic oscillations produced by condensers, inclined to one another, are superposed and combined to form more complicated, *e.g.*, circular or elliptical, oscillations. J. S. G. THOMAS.

Electric insulating material. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 315,354, 18.6.29. Ger., 12.7.28).—The use of a powdered piezo-electric material, *e.g.*, quartz, soaked in transformer oil is claimed. J. S. G. THOMAS.

Manufacture of [vitreous] heat-resisting insulating material. INTERNAT. GEN. ELECTRIC CO., INC., ASSECS. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 315,265, 26.6.29. Ger., 10.7.28).—Kaolin, talc, or heavy spar, with or without mica or asbestos, is used in place of the siliceous materials described in B.P. 152,780 (B., 1920, 320 A). [Stat. ref.] H. ROYAL-DAWSON.

Electrical insulating materials [containing rubber]. W. S. SMITH, II. J. GARNETT, J. N. DEAN, B. J. HARGOOD, and H. C. CHANNON (B.P. 334,587, 6.6.29. Addn. to B.P. 329,275; B., 1930, 723).—Dirt-free rubber is prepared by heating rubber under pressure at 150° in a solvent of b.p. about 120°, *e.g.*, light petroleum or benzene, whereby the viscosity of the solution is reduced sufficiently to enable non-rubber substances to be removed by filtration etc. The rubber is stabilised by addition of an antioxidant, *e.g.*, *p*-aminophenol, and, if desired, mixed with gutta-percha and/or balata. Alternatively, the rubber may be hardened by mixing with bitumen of low ash and free carbon contents and of high m.p. J. S. G. THOMAS.

Manufacture of resistor material. J. SLEPIAN, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,763,268, 10.6.30. Appl., 15.10.23).—Plates composed of a hardened mixture of lampblack, carborundum, and kaolin are saturated with an oxidising agent, *e.g.*, chromic acid, potassium nitrate, potassium permanganate, and heated at about 300°, whereby the carbonaceous material is oxidised. J. S. G. THOMAS.

Electrode for electric welding or soldering [of aluminium or its alloys]. E. J. CLARKE, ASSR. to ALLOY WELDING PROCESSES, LTD. (U.S.P. 1,763,417, 10.6.30. Appl., 15.2.28. U.K., 29.3.27).—An aluminium core is coated with a flux composed of 30–60 pts. of potassium fluoride, 30–60 pts. of potassium chloride, and 3–20 pts. of sawdust, of sufficient thickness to confine the arc to the crater formed. J. S. G. THOMAS.

Increasing the electrical conductivity of atmospheres surrounding electric arcs used for heating or welding purposes. I. G. FARBENIND. A.-G. (B.P. 307,834, 11.3.29. Ger., 14.3.28).—An aqueous solution or paste containing salts of potassium or alkali metals of higher at. wt., together with oxygen compounds of titanium, uranium, molybdenum, tungsten, aluminium, or iron is introduced into the region surrounding the arc, *e.g.*, by means of a hollow electrode. [Stat. ref.] J. S. G. THOMAS.

System of electrical precipitation [for gases]. C. S. WEYANDT, ASSR. to SYNTRON CO. (U.S.P. 1,760,461, 27.5.30. Appl., 21.11.23).—A vibrating device, controlled by an electric valve, for periodically vibrating the vertically suspended conductor of the precipitator is claimed. J. S. G. THOMAS.

[Electrodes for] electrostatic precipitator. R. B. RATHBUN, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,763,010, 10.6.30. Appl., 12.8.26).—Pairs of co-axial electrodes, comprising a wire surrounded by a cylindrical open-mesh electrode, are arranged in a conduit through which gas flows substantially at right angles to the axes of the electrodes. J. S. G. THOMAS.

Heat-treatment of loaded [electrical] conductors. G. W. ELMEN, ASSR. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,763,884, 17.6.30. Appl., 29.5.26).—A loaded conductor, heated at 900°, is cooled at the rate of about 50° per min. so as to increase the permeability of the loading material (nickel-iron alloy). J. S. G. THOMAS.

Electron-emission material. H. C. RENTSCHLER, J. W. MARDEN, and C. T. ULREY, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,440, 27.5.30. Appl., 23.12.22).—A refractory cathode composed of 93% W and 7% U is claimed. J. S. G. THOMAS.

Manufacture of [activated] electron-emitting [cathode] devices etc. C. T. ULREY, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,454, 27.5.30. Appl., 24.7.22).—The refractory cathode is arranged in an evacuated envelope with another electrode composed in part of thorium which is heated by electron bombardment and sputtered on to the refractory cathode. J. S. G. THOMAS.

Electric gaseous-discharge device. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. G. FOUND (B.P. 314,811, 1.7.29. U.S., 2.7.28).—Broad thermionic electrodes, each comprising a hollow nickel body coated with, *e.g.*, barium carbonate mixed with a cellulose compound binder and then baked, and a tungsten or molybdenum heater, are arranged at opposite ends of an envelope containing gas, *e.g.*, neon, helium, or mercury vapour, at a pressure of 1–5 mm. Hg. J. S. G. THOMAS.

[Gaseous-discharge current] rectifier. H. C. RENTSCHLER and W. W. MERRYMON, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,525, 27.5.30. Appl., 15.12.26).—A substantially pure and gas-free carbon anode of relatively small area and a relatively large cathode coated with misch-metal or metal of the cerium group of rare-earth metals are arranged within an envelope containing a monatomic gas, *e.g.*, argon, neon, or helium. J. S. G. THOMAS.

Electro-discharge devices. Electrode. H. C. RENTSCHLER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,524 and 1,760,526, 27.5.30. Appl., [A] 2.10.25, [B] 16.2.29).—(A) A relatively large cathode and a relatively small anode, formed at least in part of misch-metal or a metal of the cerium group of the rare-earth metals, are arranged within an envelope containing a monatomic gas, whereby the starting and operating potentials are reduced. (B) A cathode composed at least in part of alkaline-earth oxide(s) and a co-operating electrode, composed of an alloy of chromium and nickel and, if desired, iron, coated with chromium oxide but free from iron and nickel oxides, are arranged in an envelope. J. S. G. THOMAS.

Coated cathodes for electron-discharge devices. WESTINGHOUSE LAMP Co., Assees. of (A) E. G. WIDELL, (B) M. N. FREUDENBURGH (B.P. 315,800 and 316,104, [A] 18.7.29, [B] 22.7.29. U.S., [A] 18.7.28, [B] 20.7.28).—Cathodes are coated with material prepared by (A) dissolving a cellulose ester, *e.g.*, cellulose acetate or nitrate, in a solvent, *e.g.*, alcohol-ether, amyl acetate, to which about 0.5% of a plasticiser, *e.g.*, ethyl phthalate, is added; or (B) suspending electron-emission material, *e.g.*, a mixture of pure alkaline-earth carbonates and a binder, *e.g.*, nitrocellulose, in an organic liquid which is completely vaporisable *in vacuo* and is inert towards the electron-emission material, and in which the binder is soluble, *e.g.*, ethyl carbonate and/or oxalate, together with, if desired, anhydrous alcohols of lower b.p. J. S. G. THOMAS.

Electric incandescence lamps. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 335,903, 1.7.29).—A singly- or doubly-helical tungsten filament is arranged in an envelope containing mercury or other substance the vapour of which is inert to tungsten, the distance between the filament and the envelope wall being not more than 0.4 of the corresponding distance in the ordinary tungsten-filament gas-filled lamp of the same wattage with spherical bulb. [Stat. ref.] J. S. G. THOMAS.

Photo-electric cells. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of H. F. MESICK (B.P. 319,734, 4.9.29. U.S., 27.9.28. Addn. to B.P. 303,476; B., 1930, 108).—By arranging within the envelope of the cell a metal member coated with a colloidal suspension of graphite in water, the process of heating *in vacuo* for the removal of excess alkali metal, described previously, is rendered unnecessary. J. S. G. THOMAS.

Photo-electric cells [of condenser type]. ARCTURUS RADIO TUBE Co., Assees. of S. RUBEN (B.P. 318,641, 7.9.29. U.S., 8.9.28).—Adjacent surfaces of co-operating electrodes, *e.g.*, of copper, coated with light-sensitive material, *e.g.*, cuprous oxide, are separated by a translucent dielectric material, preferably of high sp. inductive capacity, *e.g.*, glycerin, castor oil, or ethyl alcohol. J. S. G. THOMAS.

Light-sensitive discharge apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. R. KOLLER (B.P. 314,096, 22.6.29. U.S., 22.6.28).—A photo-electric device comprising a highly evacuated, elongated envelope containing a cathode and anode, and a light-sensitive discharge controller composed of a band of monatomic photo-sensitive material, *e.g.*, caesium or rubidium, deposited on the inner wall of the envelope remote from the cathode, is claimed. J. S. G. THOMAS.

Weston standard electric cell. W. W. TRIGGS. From WESTON ELECTRICAL INSTRUMENT CORP. (B.P. 335,452, 28.11.29).—An apertured disc covered with fine-mesh cloth prevents access of the mercurous sulphate, used as depolariser, to the neighbourhood of the cell terminal. J. S. G. THOMAS.

Dry cell. W. D. STALEY, Assr. to BURGESS BATTERY Co. (U.S.P. 1,760,090, 27.5.30. Appl., 6.7.28).—A dry-cell cathode core is coated first with agar-agar and then with a mixture composed of ungelatinised potato starch, 10–40% of ammonium chloride, and 1–4% of a binder of gelatinised starch. The bottom of the cell is insulated by means of wax. J. S. G. THOMAS.

Production of plates for electric storage batteries and accumulators. G. F. JUDD and J. CARLIER (B.P. 344,711, 9.9.29).—A mixture containing flowers of sulphur 1 pt., caustic soda 1 pt., and water 8 pts. is heated, allowed to cool, and then mixed with lead oxides to form a paste which is pressed into a frame and baked hard. After cooling, the plate is coated with lead. J. S. G. THOMAS.

[Electrolyte for] electrical storage batteries or accumulators. T. STANDLEY (B.P. 336,113, 23.10.29).—Warm solutions containing, respectively, 2 pts. of sulphuric acid and 2 pts. of sodium silicate each in

5 pts. of water, are mixed at about the same temperature. J. S. G. THOMAS.

[Grids, frames, etc. for] electric accumulators.

I. G. FARBERIND A.-G. (B.P. 334,628, 12.6.29. Addn. to B.P. 311,401; B., 1930, 824).—The grids etc. are swaged or stamped from a hard-lead band, containing about 3% Sb, worked through an extrusion press, and, if desired, further treated by hammering or pressing.

J. S. G. THOMAS.

Manufacture of [diaphragms for] electrolytic cells. J. Y. JOHNSON. From I. G. FARBERIND A.-G. (B.P. 335,151, 22.3.29).—A woven fabric containing wires and spun threads, preferably of asbestos, is claimed.

J. S. G. THOMAS.

Electrolysis of fused electrolytes. HIRSCH, KUPFER- U. MESSING-WERKE A.-G. (B.P. 316,969, 10.7.29. Ger., 7.8.28).—The electrolyte is heated by eddy currents induced in electrically conducting, heat-transferring material, e.g., the electrodes, the electrolyte container, or loose material arranged in the electrolyte. The method is applicable to the electrolytic production of aluminium, potassium, sodium, etc.

J. S. G. THOMAS.

Electrolytic apparatus [for production of oxygen and hydrogen]. A. E. KNOWLES (B.P. 335,987, 6.7.29. Addn. to B.P. 320,388; B., 1930, 21).—An automatic feed-water and gas-washing arrangement is fitted to the cells described previously.

J. S. G. THOMAS.

Production of magnetic bodies [cores etc.].

F. J. GIVEN, R. M. C. GREENIDGE, and J. R. WEEKS, JUN., ASSTS. to BELL TELEPHONE LABS., INC. (U.S.P. 1,759,612, 20.5.30. Appl., 6.6.29).—The magnetic stability of finely-divided nickel-iron alloys is controlled by suitable pre-annealing heat-treatment of the particles, the time and temperature of treatment being determined by the relative proportions of the constituents present in the alloy.

J. S. G. THOMAS.

Measuring the intensity of light. F. W. GILLARD (B.P. 335,179, 17.6.29).—A graduated light filter is used to maintain substantially constant the intensity of light falling upon a photo-electric cell connected with or inserted in an electric circuit including a valve amplifier.

J. S. G. THOMAS.

Optical pyrometer. A. BESTELMEYER, ASST. to HARTMANN & BRAUN A.-G. (U.S.P. 1,762,380, 10.6.30. Appl., 16.7.26).—The apparatus comprises an ammeter reading the current (but graduated in degrees of temperature) taken by a lamp which is fed by small dry battery or transformer through an adjustable resistance; a telescope passing through the ammeter case and having slits for viewing the lamp and radiant body simultaneously; a removable red screen to allow the test to be made with rays of one wave-length only; a removable smoked screen for use at high temperatures, the ammeter being doubly graduated.

B. M. VENABLES.

Heat-treatment of magnetic materials. L. W. MCKEEHAN, ASST. to BELL TELEPHONE LABS., INC. (U.S.P. 1,762,730, 10.6.30. Appl., 30.10.26).—See B.P. 303,928; B., 1929, 217.

[Positive-column gaseous] discharge tubes. A. E. WHITE. From CLAUDE NEON LIGHTS, INC. (B.P. 337,002, 18.7.29).

[Gas-filled] electric-discharge devices. H. J. SPANNER and U. DOERING (B.P. 310,895, 2.5.29. Ger., 2.5.28).

[Electromagnetic] testing of magnetisable bodies. E. POTTER. From C. KINSLEY (B.P. 336,987, 19.6.29).

[Enclosed-type] hydrometers [for accumulators]. J. H. COLLIE (B.P. 335,698, 29.8.29).

Drying apparatus (B.P. 335,942). **High-vacuum pumps** (B.P. 336,001).—See I. **Carbon electrodes** (B.P. 312,940). **Control of water-gas sets** (U.S.P. 1,758,891).—See II. **Nitrogen-hydrogen mixtures** (B.P. 335,524).—See VII. **Chromium and alloys** (U.S.P. 1,764,511). **Metallisation of non-conducting materials** (B.P. 334,510). **Electrolytic system for metals** (U.S.P. 1,764,650). **Metal sheets and tubes** (B.P. 314,444). **Copper** (B.P. 336,109).—See X. **White lead** (B.P. 314,987).—See XIII.

XII.—FATS; OILS; WAXES.

Determination of the detergent value of soaps. N. PANTYUKHOV (Masloboino-Zhir. Delo, 1929, No. 2, 20—25).—The relative interfacial tension between benzene and 0.2% solutions of commercial soaps was determined by Traube's drop method. The highest value was given by neutral soaps containing 75% of fatty acid, whilst alkaline soaps (alkali 0.21%, fatty acid 46.7%) gave the lowest values.

CHEMICAL ABSTRACTS.

Olive oil. II. Acidity. F. TRAETTA-MOSCA and M. VENEZIA (Annali Chim. Appl., 1930, 20, 452—456).—The development of acidity depends on the temperature treatment and other conditions to which the olives are subjected.

T. H. POPE.

Detection of adulteration of "Abrasinöl" (wood oil from Indo-China) with castor and arachis oils. E. STOCK (Farben-Ztg., 1930, 36, 173).—Examination of laboratory-prepared mixtures of "Abrasinöl" (a recently introduced varnish raw material) with various proportions of castor and arachis oils shows that whilst acid value and saponif. value determinations will not reveal adulteration, the refractive index of Abrasinöl (1.512 at 19°) falls with increasing proportion of adulterant. Alcohol extraction serves to indicate which of the above two adulterants has been used.

S. S. WOOLF.

Brazil-nut oil. H. A. SCHUETTE, R. W. THOMAS, and M. DUTHEY (J. Amer. Chem. Soc., 1930, 52, 4114—4117).—The oil expressed from the nuts and that extracted from the residue by light petroleum have the following constants (the figures in parentheses are for the extracted residual oil): d_{20}^{25} 0.9150 (0.9143), n_D^{20} 1.4678 (1.4683), iodine value (Wijs) 99.92 (95.21), saponif. value 194 (198), ester value 193.9 (193.8), Reichert-Meissl value 0 (0.31), Polenske value 0 (0.32), free fatty acids (as % oleic) 0.006 (0.02), acetyl value 12.3 (12.3), unsaponifiable matter 0.64% (0.68%).

The residual oil contains myristin (1.79%), palmitin (13.55%), stearin (2.58%), olein (55.64%), and linolein (21.65%).
H. BURTON.

Oil in the juice of berries. V. RUCHKIN (Masloboino-Zhir. Delo, 1929, No. 2, 47—48).—Oils of the bark (3.06%), berries (2.00%), and seeds (12.13%), respectively, of *Hippophae rhamnifolia*, L. (Siberia), afford the following values: acid 9.60, 7.35, 4.42; saponif. 189.2, 190.0, 192.5; ester 179.6, 182.7, 188.1; iodine (Hübl) —, 76.5, 138.2; iodine (Margosches) 56.3, 76.8, 138.4; Reichert-Meißl —, 0.79, 0.58; acetyl (Normann) —, 16.4, 10.4; unsaponif. —, 3.66, 1.78; hydroxy-acids —, 0.79, 0.61; hexabromides —, 0, 19.58; *d* —, 0.92435, 0.92783; f.p. 12.0°, 6.4°, —20.0°; *n* 1.4639, 1.4642, 1.4739; f.p. of fatty acids 39.5°, 34°, 1.3°; mol. wt. of fatty acids —, 276.5, 278.9; iodine 61.2, 78.2, 140.3; linolenic acid 0, 0, 14.63; linoleic acid 0, 10.5, 12.31; isolinolenic acid 0, 0, 20.69; oleic acid 62.6, 63.4, 41.47; palmitic and stearic acids 37.4, 10.4, 10.9%.

CHEMICAL ABSTRACTS.

Determination of oil in vegetable materials. B. MASLENIKOV (Masloboino-Zhir. Delo, 1930, No. 2, 28—30).—The finely-powdered material is mixed with a soluble salt, and a smooth paste is prepared by the addition of water, affording on drying a stable, porous mass. The mixture is repeatedly granulated during the extraction.

CHEMICAL ABSTRACTS.

Coriander as a source of fatty oil. J. ORLOV (Masloboino-Zhir. Delo, 1929, No. 3, 26—28).—The greenish fatty oil (17—21%) has iodine value 89.7, saponif. value 214.6, and acid value 4.3.

CHEMICAL ABSTRACTS.

Oil from Kuban *Perilla ocumoides* seeds. M. ZHDAN-PUSHKIN (Masloboino-Zhir. Delo, 1929, No. 2, 44—47).—The seeds contain moisture 6.30, protein 23.12, oil 45.07, nitrogen-free extractive matter 10.28, crude fibre 10.28, ash 4.64, essential oil 0.29%. The essential oil, *d* 0.9304, has saponif. value 24.8, acid value 2.6, ester value 22.2; it polymerises, with thickening, when heated. The fatty oil has acid value 1.0, saponif. value 190.06, ester value 189.06, Reichert-Meißl value 1.43, Polenske value 1.62, acetyl value 0.71, iodine value 203.05, hexabromide value 63.65, unsaponifiable matter 0.3%, Hehner value 95.8. The fatty acids were examined.

CHEMICAL ABSTRACTS.

Solid vegetable oils. S. IVANOV (Masloboino-Zhir. Delo, 1929, No. 1, 32—34).—The seeds of *Simmondsia californica* (Mexico) contain 6.85% of moisture and 44.49% of a solid oil having acidity 5.4, saponif. value 165.7, iodine value 79.3—80.2, *d*₂₅ 0.8990. Oil of the seeds of *Pentaclethra macrophylla* (W. Africa) has *d*₁₅ 0.916, m.p. 20.1°, acid value 6.02, saponif. value 189, iodine value 76.57, titre 51.5. Oleic acid and acids similar to linolenic acid are present. Oil (36%) of the seeds of *Butyrospermum parkii* (Sudan) has *d*₁₅¹⁰⁰ 0.860, m.p. 27.2°, acid value 106, saponif. value 244.7, iodine value 52—61, and contains stearic (34—37%) and oleic (60%) acids.

CHEMICAL ABSTRACTS.

Chemistry of the sulphation of oils. Report of a Committee of the Society of Leather Trades'

Chemists. D. BURTON and F. G. A. ENNA (J. Soc. Leather Trades' Chem., 1930, 14, 459—477).—The possible reactions which may occur when an oil is sulphonated are discussed. Since commercial sulphonated oils may contain neutral oil, free fatty acids, sulphuric acid compounds, various polymerides, hydroxy-compounds, oxidised products, inorganic salts, soaps, and water, the insoluble bromide value is found to yield more information about the behaviour of marine animal oils on sulphonation than does the iodine value. A high figure indicates the presence of sufficient acids of the clupanodonic type to render sulphonation difficult. Three cod oils of normal iodine value were found by the present authors to be too violent on sulphonation. By determination of the acetyl value of the sulphonated oil the nature of the sulphonation, whether at the double linking or hydroxyl group, can be ascertained.

D. WOODROFFE.

Vegetable oils as lubricants. CHAMPSAUR.—See II. "Water of vegetation" of olives. TRAETTA-MOSCA and VENEZIA.—See XIX.

See also A., Nov., 1368, **Separation of sodium soaps of higher fatty acids** (OSTWALD and ERBRING). 1483, **Oil from seeds of *Cassia Tora*** (JOIS and MANJUNATH). **Oil from *Cocos nucifera*** (CHANDRASENA). 1483, **Calculation of lipins as fat** (FINCKE).

PATENTS.

Manufacture of soaps. A. IMHAUSEN (B.P. 335,954, 6.5.29).—Soap of high lathering power is made by mixing coconut (or palm-kernel) oil, oil paste soap, or coconut oil and the equivalent alkaline lye (after saponification), into a (liquid) boiled and grained soap, after separation of the latter from the foots.

E. LEWKOWITSCH.

Colouring rubber oil substitute. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,912, 29.5.29).—Emulsions or suspensions of rubber oil substitute (factice from linseed or rape oils etc.) are mixed with aqueous pastes of inorganic or organic, water-insoluble dyes, pigments, lakes, leuco-vat dyes, etc.; or such colouring matters are precipitated in the emulsions, which are afterwards broken by the addition of acid agents, salts, etc.

E. LEWKOWITSCH.

Preparation of water-soluble product derived from the fatty acids occurring in wool fat. A. THAUSS and G. MAUTHE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,252, 4.11.30. Appl., 16.12.27. Ger., 17.12.26).—See B.P. 307,776; B., 1929, 442.

Preparation of a product obtainable by treating wool fat with a sulphonating agent. G. MAUTHE and A. THAUSS, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,027, 28.10.30. Appl., 24.2.28. Ger., 1.3.27).—See B.P. 236,252; B., 1929, 608.

Oxidation of fats, [oils], waxes, and resins. W. PUNGS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,780,632, 4.11.30. Appl., 19.10.25. Ger., 22.10.24).—See B.P. 258,099; B., 1926, 987.

Stable sulphonic acids etc. (B.P. 313,453).—See III. **Waterproofed paper** (B.P. 335,559).—See V. **Artificial masses** (B.P. 334,572).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

[Paint] **siccative.** A. ZINOVIEV (Masloboino-Zhir. Delo, 1929, No. 2, 39—43; No. 3, 51—56).—Lead manganese linoleate is preferred; its preparation is described. **CHEMICAL ABSTRACTS.**

Preparation of varnish oil by dehydration. A. ZINOVIEV (Masloboino-Zhir. Delo, 1929, No. 1, 12—16).—The colloidal substances remain in solution at all temperatures if the oil is previously dehydrated, preferably by passing a current of dry carbon dioxide through it at 140—150°. **CHEMICAL ABSTRACTS.**

Synthetic camphor etc. SCHWYZER.—See III.

PATENTS.

Preparation of oil paints. L. BLUMER (L. BLUMER CHEM. FABR.) (B.P. 314,992, 28.6.29. Ger., 7.7.28).—Phthalic acid, salts thereof, or salts of similar polybasic carboxylic acids are introduced as "swelling bodies" into oil paints. The partly gelatinised product does not sink into porous surfaces and several coats may be applied without intervening drying periods.

S. S. WOOLF.

Manufacture of [weather-resistant] paints. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,626, 2.5.29).—Precipitated zinc oxide dried at not much above 500° is introduced into paints in quantity at least sufficient to convert into soaps all free or combined fatty acids present in the vehicle, the remaining quantity of pigment being of the usual character. **S. S. WOOLF.**

Production of bituminous paint or varnish. CRAIGBANK CHEM. CO., LTD., and J. H. TAYLOR (B.P. 336,117, 26.10.29).—Tannic acid (5—15 vol.-%) is compounded with the bituminous paint or varnish, or its constituents, to ensure drying with a plastic skin and to give the product anti-corrosive and anti-fouling properties.

H. ROYAL-DAWSON.

Electrolytic production of white lead. R. J. FROST (B.P. 314,987, 24.6.29. Austral., 6.7.28).—Metallic lead electrodes immersed in a dilute aqueous solution of commercial sodium bicarbonate are subjected to the action of a direct current flowing in alternate directions for 15 min. in each direction, the electrolyte being regenerated by the addition of carbon dioxide. The flotation of the white lead thus formed and its removal to hydro-extractors and filters are assisted by circulation of the electrolyte and by the evolution of gases from the electrodes. **S. S. WOOLF.**

Rosin soap lakes of azo compounds. (Sir) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 334,874, 8.3.29).—A heavy-metal salt is added to a mixture of acid azo dye and rosin soap in aqueous medium, whereby the metal lake and resinate are co-precipitated. The azo dye may be substantially insoluble in water, and the rosin soap added prior to or during coupling of the azo-dye components. The resinate lakes are insoluble in oil and in water, and are suitable for printing inks. Examples are: β -naphthylamine-1-sulphonic acid \rightarrow β -naphthol, rosin soap, barium or calcium chloride; *p*-toluidine-*o*-sulphonic acid \rightarrow 2:3-hydroxynaphthoic acid, rosin soap, calcium chloride; β -naphthylamine-1-sulphonic acid \rightarrow 2:3-hydroxynaphthoic *m*-nitroanilide, rosin soap, barium

chloride; α -naphthylamine \rightarrow Laurent acid, rosin soap, calcium chloride. **C. HOLLINS.**

Manufacture of artificial masses as bases for coatings, impregnating agents, binders, etc. H. HÖNEL (B.P. 334,572, 8.5.29).—An ester, particularly a polyhydric alcohol ester of a mono- or poly-basic acid, is heated, in absence of solvent or dispersing agent, with a low-molecular product obtained by alkaline condensation of formaldehyde with a phenol in which at least one of the positions 2, 4, and 6 is substituted: the low-molecular product should be one which is capable of thermo-hardening. Amongst the examples given are: beeswax with the liquid product from *p*-tert.-amylphenol and formaldehyde; castor oil with *p*-cresol-formaldehyde; "honey oil" (moderately boiled wood oil and linseed oil) with thymol-formaldehyde; glyceryl succinate-lactate with *p*-chlorophenol-formaldehyde; glyceryl citrate-ricinoleate-salicylate with *p*-cresol-formaldehyde; glyceryl phthalate-stearate with the formaldehyde condensation product from the reaction product of *o*-cresol and methyl ethyl ketone; etc. **C. HOLLINS.**

Solution of cellulose derivatives [lacquer]. A. KNORR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,780,883, 4.11.30. Appl., 16.3.28. Ger., 23.12.24).—See B.P. 275,653; B., 1927, 905.

Applying nitrocellulose varnishes. H. F. SCHULZ (U.S.P. 1,780,844, 4.11.30. Appl., 30.11.26. Ger., 4.12.25).—See B.P. 262,440; B., 1927, 916.

Manufacture of polybasic acid-polyhydric alcohol resins. H. WADE. From BAKELITE CORP (B.P. 336,645, 10.7.29).—See U.S.P. 1,739,771; B., 1930, 677.

Manufacture of inlaid linoleum. ARMSTRONG CORK CO., Assees. of J. C. MCCARTHY (B.P. 317,003, 15.7.29. U.S., 8.8.28).

(A) **Decoration, (B) manufacture, of surface coverings [inlaid linoleum].** ARMSTRONG CORK CO., Assees. of [A] H. W. PRENTIS, [B] S. H. HARTMAN and C. F. HUMPHREYS (B.P. 316,549 and 316,970, 15.7.29. U.S., [A] 15.7.29, [B] 7.8.28).

Floor-covering materials [linoleum] and their decoration. ARMSTRONG CORK CO., Assees. of J. C. MCCARTHY (B.P. 319,655, 20.7.29. U.S., 25.9.28).

Calcination of pigments (B.P. 335,659).—See I. **Distillation of resinous woods** (U.S.P. 1,757,144).—See II. **Azo lakes** (B.P. 334,754).—See IV. **Poly-saccharide ether compositions** (B.P. 334,897). **Cellulose ether or ester compositions** (B.P. 308,658 and 335,582). **Products from cellulose esters or ethers** (B.P. 315,766). **Cellulosic compositions** (B.P. 313,535).—See V. **Titanates** (U.S.P. 1,760,513). **Mouldable product** (U.S.P. 1,761,740).—See VII. **Coated cathodes** (B.P. 315,800).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Change of electrical properties of rubber and gutta-percha during storage under water. H. L. CURTIS and A. H. SCOTT (Bur. Stand. J. Res., 1930, 5, 539—552).—The effect of storage under water for periods up to seven years on the resistivity, dielectric constant, and power factor of samples of rubber and

gutta-percha has been investigated. The resistance-time curves showed a break or marked change in direction several months before actual failure of the rubber occurred. In the case of power factor and *D.C.* dielectric constant curves, breaks occurred at a later time. The decrease in resistivity is ascribed to the formation of fine holes through the material. Microscopical examination showed fern-like figures projecting into the rubber. The catalytic action of copper salts from electrodes inside the tube specimens accelerated the ageing of the rubber and probably changed its character.

W. E. DOWNEY.

PATENTS.

Rubber compositions [anti-agers] and their manufacture. H. A. MORTON (B.P. 314,756, 1.7.29. U.S., 30.6.28).—Ethylenediamines in which one or more of the 8 hydrogen atoms is replaced by a hydrocarbon residue, especially an aryl group, excluding *N*-naphthyl derivatives, are incorporated with a rubber mix as anti-agers. The *N*-aryl groups may be monosubstituted, e.g., by hydroxyl, amino-, chloro-, or nitro-groups. Amongst the anti-agers mentioned are: *NN'*-di-phenylethylenediamine, *NN'*-diphenyl-*NN'*-diethylethylenediamine, *NN'*-diphenyl- $\alpha\beta$ -propylenediamine, *NN'*-diphenyl- $\beta\gamma$ -butylenediamine, *NN'*-diphenyl- $\beta\gamma$ -diaminoisopentane [$\beta\gamma$ -dianilinoisopentane], and *NN'*-diphenyl- $\alpha\alpha\beta\beta$ -tetramethylethylenediamine. The substituted ethylenediamines have practically no accelerating effect.

C. HOLLINS.

Manufacture of rubber vulcanisation accelerators. W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,779,715, 28.10.30. Appl., 2.3.27).—See B.P. 286,749; B., 1928, 377.

Rubberised fabric (U.S.P. 1,763,618). Water-proofed paper (B.P. 335,559).—See V. **Insulating material (B.P. 334,587).**—See XI. **Colouring rubber-oil substitute (B.P. 335,912).**—See XII.

XV.—LEATHER; GLUE.

Standardisation of hide powder. IV. Interpretation of the results of the relative specific surface test. H. G. BENNETT (J. Soc. Leather Trades' Chem., 1930, 14, 481—485; cf. B., 1927, 534).—There is a definite relationship between the relative specific surface results and the soluble matter in a hide powder. The coefficient of correlation for the successful powders (B3—B6) was -0.86 , and for another series of powders was -0.95 . As the soluble matters decrease and the purity of the powders increases the degree of correlation will decrease. When the soluble matter is reduced to zero the correlation coefficient will be zero also. The coefficient if regularly determined will indicate how near this ideal of zero water-soluble matter has been approached.

D. WOODROFFE.

Influence of temperature in single-bath chrome-tanning. C. OTIN and G. ALEXA (J. Soc. Leather Trades' Chem., 1930, 14, 450—459).—Samples of hide powder were treated at 25°, 32°, and 42°, respectively, with one-bath chrome-tanning liquors having basicities varying from 0 to 53%, and after 48 hrs. the powder was washed and analysed. The amount of chromic oxide fixed by the powder was found to be a function of

the basicity (the higher the basicity the greater was the quantity fixed), and to increase with rise in temperature. The effect of temperature was most marked for the liquor of 32% basicity; when such was used 42% and 91% more chromic oxide was fixed at 32° and 42°, respectively, than at 25°. The amount of SO_4 fixed by the powder decreased as the basicity of the solutions increased. It was shown by time tests that the amounts of chromic oxide and sulphuric acid fixed by the powder were a function of the time at all temperatures, and the greatest increase in the fixation of chromium was caused by increasing the temperature of the tanning liquors in the early stages of tanning. The basicity of the liquors was diminished by a rise in temperature. The effect of temperature on chrome-tanning liquors is attributed to the expulsion of carbon dioxide, increased hydrolysis and polymerisation, and greater degree of "olation" with increased colloidal character.

D. WOODROFFE.

Wilson-Kern method [of tannin analysis] in laboratory practice. A. T. HOUGH and R. DRU (J. Soc. Leather Trades' Chem., 1930, 14, 478—481).—The tanned powder is transferred to a 500-c.c. filter bell 10 cm. in diam., the mouth of which is covered with the usual filter cloth; the filter bell is immersed to a depth of 3 cm. in a 500-c.c. porcelain basin (carrying a stirrer) full of distilled water and rests on a Y-shaped stand. Means are provided for the inflow of distilled water and for the restricted exit of the washings. The filter bell is gently agitated during the first hour of washing and then set aside overnight. To complete the process 6—10 litres of water and 12—20 hrs.' washing are necessary.

D. WOODROFFE.

See also A., Nov., 1458, **Change in gelatin on storage** (THIMANN and PAGE).

PATENTS.

Production of a lather-forming depilating preparation. E. HADANK (B.P. 317,707, 19.8.29. Ger., 18.8.28).—Alkali or alkaline-earth sulphides are mixed with a peroxide, an inert diluent, and a mucilage.

D. WOODROFFE.

Unhairing agent [for hides]. A. C. ORTHMANN, Assr. to PFISTER & VOGEL LEATHER CO. (U.S.P. 1,763,319, 10.6.30. Appl., 5.3.28).—Beet-sugar residues (8—20 pts.) are mixed with caustic soda (3—3.5 pts.), lime, or other alkaline medium, and water (150 pts.), and the hides are immersed therein for 5 days.

D. WOODROFFE.

Tanning animal hides with iron salts. K. STÜRMER (U.S.P. 1,763,596, 10.6.30. Appl., 12.2.29. Ger., 18.2.28).—Hides are treated with solutions of ferrous salts, which may or may not have been pre-treated with nitrogen monoxide. Pure nitrogen monoxide is then introduced into the solutions, or the hides are subjected to the action of air or oxygen under pressure.

D. WOODROFFE.

Acceleration and improvement of tanning processes. F. POSPIECH, Assr. to CHEM. FABR. POTT & CO. (U.S.P. 1,763,363, 10.6.30. Appl., 26.8.27. Ger., 21.8.26).—Condensation products of naphthalenesulphonic acids and aliphatic alcohols above C_2 , e.g.,

naphthalenesulphonic acid and isopropyl alcohol, are added to the tan liquors. D. WOODROFFE.

Manufacture of a casein-glue film. P. M. F. BEHNKE, N.V. HANDELMAATS. "CUBA," and N.V. HOUTIND. "PICUS" (B.P. 314,344, 21.6.29. Ger., 25.6.28).—Heated liquid casein glue or a hot solution of casein glue is brought on to a cold or cooled metal surface, dried if necessary, removed, and used in the preparation of ply-wood. D. WOODROFFE.

Felt (U.S.P. 1,765,046).—See V. **Preparation of fur** (U.S.P. 1,762,233).—See VI. **Sulphite waste-liquors** (U.S.P. 1,764,600—1).—See VII. **Immunising glue** (B.P. 335,527).—See XVI.

XVI.—AGRICULTURE.

Magnesium and calcium in zeolitic soils. J. F. BREAZEALE (Arizona Agric. Exp. Sta., Tech. Bull., 1929, No. 26).—Alkali soils were percolated with various simple and mixed salt solutions and the nature of the exchanged bases was examined. The presence of a common ion in the percolating solution depressed the ionisation of the soil zeolites and retarded the exchange of bases. Percolation of a calcareous magnesium-zeolite soil with equivalent solutions of either calcium or magnesium salts led to the same final equilibrium in the percolate. Replacement of bases by solutions of calcium or magnesium salts was similar in both calcareous magnesium- and calcium-zeolite soils. Calcium carbonate in zeolitic soils took part in base-exchange reactions as readily as the more soluble calcium salts, but magnesium carbonate in the form of magnesite or dolomite was practically inactive in this respect, except in the presence of carbon dioxide. Treatment of soil with dilute hydrochloric acid resulted in some decomposition of the zeolite as well as replacement of base by H⁺. This action was reversed by subsequent addition of solutions containing exchangeable base, the re-formation of zeolite being largely controlled by the reaction of the solution used. A. G. POLLARD.

I. Effect [on crops] of yard manure and "huminit" in comparison with mineral fertilisers. II. Composition of some green fodders and green manuring plants. III. Effect of chromium on plant growth. IV. Effect of arsenic on plant growth. E. HASELHOFF, F. HAUN, and W. ELBERT (Landw. Versuchs-Stat., 1930, 110, 247—268, 268—283, 283—286, 287—289).—I. "Huminit" used in conjunction with artificial fertilisers tends to increase crop yields, but is inferior in this respect to farmyard manure.

II. Analyses are recorded of the food values of the tops and the content of fertiliser constituents in both tops and roots of a number of crops in varying stages of growth.

III. In sand cultures and in soils chromium compounds depressed the growth of barley and mustard. Chromium hydroxide was less injurious than chromium trioxide, and the effects of both were more pronounced in a sandy soil than in loam. Small quantities of chromium were absorbed by the plants.

IV. Addition of calcium arsenate to soil (up to 0.003% As) prevented the growth of barley in a sandy soil, but was without influence on the crop yield on a loam.

A. G. POLLARD.

Interrelation of nutrients and soil reaction on growth and inoculation of lucerne. M. C. SEWELL and P. L. GAINES (Soil Sci., 1930, 30, 297—305).—In sand cultures lucerne grew well in media of p_H 4.5 if well supplied with nutrients, including calcium. Nodulation was scanty in media of p_H 6.0, but well-developed at p_H 7.5. In soils the supply of phosphate to the plant may be more important than the reaction in controlling the amount of nodulation. A. G. POLLARD.

Determination of potash in very dilute solutions and in soil liquors. M. VON WRANGELL (Z. anal. Chem., 1930, 82, 224—226).—The aqueous extract of the soil is evaporated to dryness, the residue heated to expel ammonium salts and dissolved in 2 c.c. of 3% acetic acid, and 1 c.c. of the solution treated with 1 c.c. of sodium cobaltinitrite solution (cf. Kramer and Tisdall, A., 1921, ii, 412). Next day the precipitate is collected, washed by centrifuging, and dissolved in 5 c.c. of 0.1N-sodium hydroxide; the solution is diluted, treated with 1 c.c. of a 0.15% solution of indole in 10% alcohol and with 1 c.c. of 1:1 sulphuric acid, and diluted to 100 c.c. The resulting colour is compared with that produced by a standard sodium nitrite solution under the same conditions. A. R. POWELL.

Use of preservatives to prevent loss of nitrogen from cow excreta during the day of collection. R. B. FRENCH (J. Agric. Res., 1930, 41, 503—506).—Cow faeces and urine placed together, but unmixed, lose 1—2.6% of their total nitrogen, and, when mixed, 6—30% during 18 hrs. These losses may be prevented by the addition of 2 c.c. of N-copper sulphate, -zinc sulphate, or -formaldehyde per 100 g. of mixture. E. HOLMES.

See also A., Nov., 1482, **Germination of seeds** (SASAKI). **Energy-storage of plants and their carbon and nitrogen contents** (HÖNL).

PATENTS.

Manufacture of fertiliser salts capable of being stored. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,175, 15.6.29).—The caking on storage of granular, non-hygroscopic fertilisers is prevented by coating them with 1—5% of water-insoluble non-volatile oils. The oils may contain solid, water-repellent substances such as waxes, paraffins, or fats. W. J. WRIGHT.

Fertilising solution for seed treatment. W. F. GERICKE (U.S.P. 1,762,294, 10.6.30. Appl., 11.8.25).—Seeds are immersed for 24 hrs. in an aqueous solution containing 4 mols. of monopotassium phosphate and 1 mol. of caustic potash per litre, and, while still wet, are coated with plaster of Paris. A. G. POLLARD.

Disinfection of seeds. W. P. RALEIGH, ASSR. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,764,888, 17.6.30. Appl., 26.4.28).—Seeds are dusted with an insoluble additive compound of a mercuric salt (e.g., mercuric chloride) and hexamethylenetetramine mixed with an inert, finely-divided carrier such as graphite. A. G. POLLARD.

Fungicide. E. B. ALVORD, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,762,709, 10.6.30. Appl., 7.9.26).—The use of colloidal copper sulphide, either dry or in a

liquid vehicle containing a dispersing agent, is claimed. The product does not "burn" delicate foliage when used as a fungicide.

A. G. POLLARD.

Non-alkaline chlorate weed-killer. R. N. CHIPMAN (B.P. 335,203, 14.6.29. U.S., 22.12.28).—The herbicidal efficiency of chlorate weed-killers is increased by addition of acid substances in sufficient amount to bring the p_H below 7, whilst avoiding liberation of chlorine. Thus by adding 1, 2, and 5% of zinc chloride, the p_H was lowered to 7.1, 6.6, and 6.4, respectively; by the same percentages of copper chloride, to 5.2, 5.1, and 5.1; and by boric acid, to 7.0, 6.6, and 6.2.

W. J. WRIGHT.

Nicotine carbolates (U.S.P. 1,762,471).—See II.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Manufacture of adhesives. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,876, 1.5.29).—Adhesives made from gum arabic, dextrin, etc. are protected from bacteria, moulds, etc. by incorporation of hydroxylated di- or tri-arylmethanes containing nuclear halogen, e.g., the compounds of B.P. 316,900 and 330,893—4 (B., 1929, 888; 1930, 896), such as 5:5'-dichloro-2:2'-dihydroxydiphenylmethane, 3:3':5:5': tetrachloro-2:2'-dihydroxydiphenylmethane, 4:5'-dichloro-2-hydroxydiphenylmethane, 3':3'':5':5'': tetrabromo-4':4'-dihydroxy-1:1-diphenylcyclohexane, 4-chloro-2:6-bis-(5-chloro-2-hydroxybenzyl)phenol, etc.

C. HOLLINS.

Conveyor-dryers (B.P. 336,009).—See I. Unhairing hides (U.S.P. 1,763,319).—See XV.

XVIII.—FERMENTATION INDUSTRIES.

Pectin from lemon residues. LEONE.—See XIX.

See also A., Nov., 1409, **Volumetric determination of acetone** (MEYER and MATHEY). 1431, **Sterols from yeast** (WIELAND and GOUGH). 1473, **Catalase of milk and its determination** (ZAYKOVSKY and ALENIEV). 1476, **Uricase from ox-kidney: its preparation and properties** (TRUSZKOWSKI).

XIX.—FOODS.

Direct iodising of milk is possible. E. D. DEVEREUX (Mich. Agric. Exp. Sta. Quart. Bull., 1929, 193—194).—Colloidal iodine may be added to milk (up to 100 p.p.m.) without alteration of colour or taste, even after ageing or souring.

A. G. POLLARD.

Determination of lactic acid in milk and milk products. L. H. LAMPITT and M. BOGOD (Compt. rend. 9me. Congrès de Chim. Ind., 1930, 6 pp.).—The method is based on the oxidation of lactic acid with potassium permanganate, the resultant acetaldehyde being distilled into a known volume of bisulphite solution, and the excess titrated with iodine. Full experimental details are given. Experiments showed that losses occur during the distillation unless the receiver is fitted with a U-trap containing 1 c.c. of 0.01N-iodine diluted with 10 c.c. of water. The optimum concentration for the permanganate solution was found to be 0.004N, the addition of 100 c.c. occupying 1 hr. The distillate

(80—100 c.c.) and contents of the U-tube are titrated with 0.01N-iodine, allowance being made for that contained in the trap. The iodine equivalent of the bisulphite solution having been determined, the difference between the titrations gives the acetaldehyde value (1 c.c. of 0.01N-iodine \equiv 0.45 mg. of lactic acid). When tested with zinc lactate and milk powder containing known amounts of zinc lactate it was found that a correction factor of 10/9 must be applied to obtain the true results. It has thus been shown that the lactic acid content of normal milk is lower than the acidity as indicated by titration, but if alkali has been added during manufacture the difference diminishes and may even change its sign.

H. J. DOWDEN.

Refractometer in milk analysis. G. D. ELSDON and J. R. STUBBS (Analyst, 1930, 55, 618—625).—The copper sulphate method (B., 1927, 375) of producing milk serum for determining the refraction gives good and concordant results if care be taken to prevent evaporation. Sera may also be prepared by the use of 7% phosphotungstic acid solution containing 2.5 c.c. of hydrochloric acid per 100 c.c., but this reagent also precipitates the albumin, and the refractions obtained are, on the average, 3.25 lower than those obtained with copper sulphate. The sour-serum method is satisfactory, provided that the milk is kept at a uniform temperature; 21° is suggested as a standard. In general, the refraction of milk decreases slightly with increasing age whatever method is used in the test.

A. R. POWELL.

Variation in the analytical data of butter as a result of alteration and ageing. F. ARNAUDO and A. SOLDI (Annali Chim. Appl., 1930, 20, 405—409).—Results obtained with a number of samples show that ageing and alteration of butter under ordinary conditions are accompanied by diminution of the Reichert-Meissl and Zeiss refractometer values, by slight variations in either direction of the Polenske value, and by marked increase in the total free acid. As the last-named increases in amount from 8 to 80 (c.c. of N-alkali per 100 g.), the refractive index falls progressively by 0.2—2.2 and the Reichert-Meissl value by 0.4—7.2. In those exceptional cases when the samples have an odour of butyric esters, the decrease in the Reichert-Meissl value may amount to 16.8 and that in the Zeiss value to 4.2, the total acidity being 138—145.

T. H. POPE.

Composition of some rabbit carcasses. W. K. WILSON (J. Min. Agric., 1930, 36, 1203—1206).—The chemical composition of rabbit flesh (Angora) closely resembles that of chicken, the average protein content differing by less than 1%. The composition of the flesh from male and female rabbits was noticeably different, especially in the fat content, females containing 4—6% more fat than the males.

B. W. TOWN.

Substance contained in the "water of vegetation" of olives. F. TRAETTA-MOSCA and M. VENEZIA (Annali Chim. Appl., 1930, 20, 449—451).—Application of high pressure to comminuted olives yields, together with the oil, water of vegetation, which contains a methylpentitol, $C_6H_{14}O_5 \cdot H_2O$, m.p. 164°, this giving a penta-acetyl derivative, m.p. 121°. On oxidation with

bromine the polyhydric alcohol forms a compound which gives a *phenylhydrazone*, $C_6H_3O_4(N_2HPh)_2$, m.p. 195°.

T. H. POPE.

Extraction of pectins from lemon residues (pastaccio). P. LEONE (Annali Chim. Appl., 1930, 20, 433—444).—Owing to the threat directed against the citrus industry by the competition of fermentation citric acid, the economical production of pectin from pastaccio becomes of importance. The most satisfactory method of precipitating solid pectin is by means of alcohol, and to replace the inevitable losses of alcohol the author proposes to produce alcohol by fermenting the carbohydrates of the lemon residues. Pastaccio contains 80% of water, the dry matter having the percentage composition: ash 4—4.2, fats, resins, and waxes 24—35, cellulose 19—22, lignin 8.4—9.2, groups forming furfuraldehyde 8—9.5, reducing sugars 1.2—1.8, hemicellulose, gum, etc. 9.2—15.7, pectins 15—19, nitrogenous substances not determined. The treatment suggested is briefly as follows: the pastaccio is heated with twice its weight of 0.05% hydrochloric acid at 103—105° and filtered, the filtrate being concentrated to one half of its volume in a triple-effect apparatus and fermented, best with a selected yeast. The fermented liquid is cleared by centrifuging and is then treated with an equal volume of 96% alcohol to precipitate the pectins, which are filtered off. The dilute alcoholic filtrate is concentrated in a rectifying column, the amount retained by the pectin being recovered by means of a vacuum desiccator followed by a column. Appreciable loss of pectin occurs only if the fermented liquid is kept for some time.

T. H. POPE.

Sulphuring of apricots. A. V. LYON (J. Coun. Sci. Ind. Res. Australia, 1930, 3, 161—166).—The sulphuring is best performed in hoods with small vents to ensure the slow burning of the sulphur (the open system). With the vents shut (the closed system) burning ceases before an adequate supply of sulphur dioxide is produced. The period of sulphuring was 4—5 hrs., and the mean sulphur dioxide content was 11 grains per lb. Properly sulphured apricots possess the bright colour desired by the trade; a dark or dull colour indicates under-sulphuring.

B. W. TOWN.

Methylene-blue in tinned peas. D. HENVILLE (Analyst, 1930, 55, 629).—A sample of tinned peas became green when the liquid was poured away and the peas were exposed to the air. A similar effect was obtained by boiling raw peas with 0.01—0.02 grain of methylene-blue per lb. and subsequently exposing them to the air.

A. R. POWELL.

Phytin from refuse of mustard manufacture. N. BELIAIEV (Masloboino-Zhir. Delo, 1929, No. 3, 28—30).—The ground press-cake is washed with water and treated with dilute hydrochloric acid; the extract is heated to coagulate the proteins and the phytin is precipitated by neutralising the filtered solution with 10% ammonia solution. The extraction and precipitation are repeated twice; the yields were 5.73 and 3.87% from the press-cake and bran, respectively.

CHEMICAL ABSTRACTS.

PATENTS.

Heating of apparatus for condensing milk and the like. N. J. NIELSEN (B.P. 335,964, 5.6.29).—The use of hot water instead of steam as the heating medium for the evaporation of milk etc. is found to reduce the formation of crusts on the tubes.

B. M. VENABLES.

Production of egg-yolk substitutes. B. REWALD (U.S.P. 1,762,077, 3.6.30. Appl., 12.3.29. Ger., 7.9.27).—A substitute for egg yolk in bakery goods consists of an emulsion of butter or margarine with 5—20% of soya-bean lecithin.

E. B. HUGHES.

Machines for kneading butter, margarine, and other foodstuffs. N.V. GRASSO'S MACHINEFABRIEKEN, and H. A. M. GRASSO (B.P. 336,837, 13.11.29).

Manufacture of [withered] tea. J. A. MAIN (B.P. 336,619, 14.6. and 7.11.29 and 3.3.30).

Grinding mills (B.P. 336,005).—See I. **Water-proofed paper** (B.P. 335,559).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Reactions of antifebrin and phenacetin. L. EKKERT (Pharm. Zentr., 1930, 71, 626—628).—Full details are given of the hydrolysis of the drugs to aniline and *p*-phenetidine, respectively, and the identification of the latter by well-known colour reactions.

H. E. F. NOTTON.

Acidimetric determination of theobromine in diuretin, calcium-diuretin, and in other theobromine preparations and mixtures. H. BOIE (Pharm. Ztg., 1930, 75, 968—969).—The sample of diuretin (0.5 g.) dissolved in 100 c.c. of water is made weakly acid by adding 15 c.c. of 0.1N-sulphuric acid, and to the mixture, after boiling to expel carbon dioxide and cooling, are added 1.5 c.c. of phenol-red solution (0.1 g. of phenol-red, 5.7 c.c. of 0.05N-caustic soda in 500 c.c. of water) and 2—3 c.c. of 0.1N-caustic soda (slight excess). The solution is then titrated until distinctly acid with 0.1N-sulphuric acid and by deducting from the total acid used the volume of alkali added the alkalinity of the sample is found (*a*). To the solution are now added 20 c.c. of 0.1N-silver nitrate, and the nitric acid liberated by the formation of the theobromine-silver complex is titrated to completion with 0.1N-caustic soda (*b*) (1 c.c. of 0.1N-caustic soda \equiv 0.01801 g. of theobromine). In diuretin, if equimolecular proportions of caustic soda and theobromine be present, the values of *a* and *b* are identical. The method is applicable to other theobromine preparations, provided a sufficient excess of silver nitrate solution be added to remove any iodides or thiocyanates. The results are in good agreement with those calculated from the nitrogen content and those determined iodometrically. The values obtained by the official method are about 15% too low.

H. J. DOWDEN.

Determination of colchicine in the seeds of *Colchicum autumnale*, L. P. R. COLL and P. J. PREIONI (Rev. farm., 1930, 72, 187—189).—The seeds (20 g.) are digested with water (200 c.c.) at 55—60° for 3 hrs. After 6 hrs. 140 c.c. are decanted, shaken with

lead acetate solution, and filtered. After addition of disodium hydrogen phosphate and shaking the mixture is filtered, 30 g. of sodium chloride then being dissolved in 110 c.c. The liquid is shaken with 50 c.c. of chloroform, 40 c.c. being evaporated and the residue weighed. Heraul's method is not trustworthy.

CHEMICAL ABSTRACTS.

Oil from *Perilla ocumoides* seeds. ZHDAN-PUSHKIN.—See XII.

See also A., Nov., 1393, **Dermatological thallium salts** (CLAVERA). 1418, **Local anæsthetics from amino-alcohols** (FOURNEAU and others). 1421, **Organo-antimony compounds** (DYKE and JONES). 1430, **Synthesis of an isomeride and a homologue of ephedrine** (FOURNEAU and others). 1431, **Cannabinol, the active principle of hashish** (BERGEL and others). 1432, **Organo-antimony compounds** (NIYOGI). 1451, **Synthesis of pharmacological thiazole amines** (HINEGARDNER and JOHNSON). 1454, **Alkaloid of *Skimmia repens*** (ASAHINA and others). **Skimmianine** (ASAHINA and INUBUSE). 1455, **Strychnos alkaloids** (LEUCHS and WEGENER). 1456, **Strychnine; sensitivity of chemical and physiological tests** (WARD and MUNCH). **Sulphonated aromatic arsenic compounds** (BARBER). 1458, **Mercurated phthaleins** (GREENBAUM). 1460, **Colour reaction of ephedrine** (SIVADJIAN). **Reactions of thiophen (EKKERT).** **Micro-detection of barbituric acid derivatives** (VAN ITALLIE and STEENHAUER). 1461, **Reactions of homatropine and novatropine** (EKKERT). **Titrimetric determination of primary arsinic acids** (KING and RUTHERFORD). **Toxicological determination of morphine** (PEREIRA). 1471, **Physiological assay of belladonna extracts etc.** (JENDRASSIK and WILL). **Methods of ergot assay** (SMITH and STOHLMANN). 1480, **Preparation of crystalline follicular hormone** (BUTENANDT). 1484, **Degradation of nicotine in tobacco** (FAITLOWITZ).

PATENTS.

Manufacture of 1-phenyl-2-methyl-3:4-trimethylene-5-pyrazolone. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P. 122,466, 12.5.26. Cf. B.P. 260,577; B., 1927, 869).—Ethyl cyclopentanonecarboxylate, b.p. 108°/12 mm., is condensed with α -phenyl- β -methylhydrazine in presence of sodium ethoxide at 140°.

C. HOLLINS.

Manufacture of compounds of 1-aryl-2-alkyl (or -2-aralkyl)-3:4-trimethylene-5-pyrazolones with dialkyl- and aralkyl-barbituric acids. C. MANNICH (G.P. 454,697, 17.1.26).—Analgesics are obtained by mixing the named ingredients in molecular proportions, or by mixing their solutions (using excess of the more soluble) and allowing to crystallise. Products from 1-phenyl-2-methyl-3:4-trimethylene-5-pyrazolone (cf. B.P. 260,577; B., 1927, 869) with allylisopropylbarbituric acid (m.p. 141°), with phenylethylbarbituric acid (m.p. 125°); from 1-*p*-tolyl-2-ethyl-3:4-trimethylene-5-pyrazolone with allylisopropylbarbituric acid (m.p. 127°); from 1-phenyl-2-benzyl-3:4-trimethylene-5-pyrazolone with phenylethylbarbituric acid (m.p. 64°), are described.

C. HOLLINS.

Preparation of remedies for cardiac diseases.

W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 335,202, 10.6.29).—Fresh or dried muscles of warm-blooded animals are extracted at the natural p_H of the material or in presence of added alkali, with a water-soluble organic solvent in presence of more than 10% of water. The solvent-free extract is freed from lipins and albumin by treatment with a water-insoluble organic solvent and the final aqueous extract is saponified, and the resulting solution, if necessary, freed from by-products by treatment with a water-insoluble organic solvent or by evaporation and treatment of the residue with a water-soluble solvent. The initial extraction of the muscles may be carried out with a water-insoluble solvent, in presence of water if dried muscles are used, and the residue extracted with a water-soluble solvent in presence of water; after removal of the solids the aqueous extract is treated as above. E. H. SHARPLES.

Manufacture of preparations possessing intense antirachitic properties. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 335,277, 25.6.29).—Ergosterol, either solid or dissolved in an organic solvent such as ethyl alcohol, acetone, or ethyl acetate, is irradiated by means of ultra-violet rays, irradiation preferably being stopped before the maximum of antirachitic activity is reached. Unchanged ergosterol is then separated from the irradiated product by crystallisation from the solvent, this separation being continued until the product has $[\alpha]_{D}^{25}$ greater than +25° in alcohol. It is better to irradiate the hot solution. Substances having activities greater than 50 million units are claimed. E. H. SHARPLES.

Betaine thiocyanate. R. BERENDES and L. SCHÜTZ, Assrs. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,780,860, 4.11.30. Appl., 18.6.28. Ger., 28.6.27).—See B.P. 316,693; B., 1929, 835.

[Production of] alkaline-earth salts of arsenobenzene derivatives. R. WERNER and A. ROTTMANN, Assrs. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,779,649, 28.10.30. Appl., 21.9.29. Ger., 5.9.28).—See B.P. 318,556; B., 1930, 1046.

Basic imino-ethers (B.P. 308,218).—See III. **Bromine and iodine** (B.P. 308,281).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Action of ammonium salts on photographic developers. M. MIYATA and Y. SASAKI (Rep. Imp. Ind. Res. Inst., Osaka, 1930, 10, No. 17).—The activation is physical in character. CHEMICAL ABSTRACTS.

See also A., Nov., 1329, **Actinic power of magnesium light** (ARENS and EGGERT). 1384, **Relationship between particle size and sensitivity of photographic emulsions towards X-rays** (EGGERT). **Influence of the developer on the properties of a photographic plate** (PERRAKIS). 1385, **Action of mercuric chloride on the photographic plate** (REYCHLER). **Herschel effect** (NARBUTT).

PATENTS.

Development of photographic reversal films. E. MANKENBERG, Asst. to AGFA ANSCO CORP. (U.S.P.

1,780,025, 28.10.30. Appl., 15.3.28. Ger., 25.3.27).—See B.P. 287,542; B., 1929, 378.

[Tri-pack for] colour photography. T. T. BAKER, A. B. KLEIN, and COLOUR SNAPSHOTS (1928), LTD. (B.P. 337,057, 27.7.29).

[Die-cast cellulosic] photographic film spools. I. G. FARBENIND. A.-G. (B.P. 316,302, 10.7.29. Ger., 27.7.28).

Making of X-ray photographs. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 336,918, 7.2.30. Holl., 20.2.28).

[Production of prints in natural colour in] colour photography. T. T. BAKER (B.P. 337,040—1, 25.7.29).

Measuring the intensity of light (B.P. 335,179).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Sensitiveness of gelatinised [smokeless] powders. M. TARLÉ (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 398—400).—The influence of the temperature of drying on the sensitiveness to shock and the ignition temperature of strip and flaked powders was investigated, the fall-hammer results being quoted as percentages of those obtained with undried powder. Loss in weight on drying at various temperatures for various periods was also determined. Up to 100° sensitiveness varied approximately with loss in weight; it may be inferred that at low temperatures, at which the stability is only slowly affected, a physical change occurs in the powder. At temperatures above 100° decomposition is a factor, since heating for 2 hrs. at 120° causes a loss in weight of only 2%, but gives a fall-hammer test of 81—82%. Between 60 and 80° a definite surface alteration takes place in gelatinised powders, and a study of this alteration, as well as of the relation between loss in weight and sensitiveness, may be the means of determining the age of a powder and its suitability for use. No appreciable change in the temperature of ignition was observed with various temperatures or times of heating. This does not necessarily imply that the powder underwent no change, since small changes in the physical structure do not affect the test.

W. J. WRIGHT.

Properties of nitroglycerin isomerides. A. A. DSERSCHKOVITSCH and K. K. ANDREEV (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 353—356, 400—403).—By Hess' method the liquid labile and stable isomerides of nitroglycerin gave total compressions of 26.5 and 27.5 mm., respectively. The solid stable form gave an average compression of the lower cylinder of 6.3, and the solid labile form of 4.8 mm.; in the former case the upper cylinder was completely demolished. Tested by Kast's method, liquid nitroglycerin gave an average compression of 1.84, the solid stable isomeride gave 3.07, and the solid labile one 0.70 mm. The velocities of detonation, determined by Dautriche's method with a No. 8 detonator, were 1165 m./sec. for nitroglycerin and 9150 m./sec. for the solid stable isomeride. The labile isomeride failed to detonate completely with a No. 8 detonator, but when initiated by a 20-g. tetryl primer it gave the value 9100 m./sec. Liquid nitroglycerin initiated by 10—15 g. of the stable isomeride also attained a velocity of 9100 m./sec. The labile isomeride gave the highest lead block results, 518—562 c.c.; the stable form gave 392—397, and liquid nitroglycerin 486—518 c.c.

Investigations were made of the linear velocity of crystallisation; for the labile form it is only half that of the stable form. The velocity of isomerisation was also determined. The chemical nature of the two isomerides is discussed, and an explanation advanced of the explosion abnormalities of liquid nitroglycerin and nitroglycerin explosives.

W. J. WRIGHT.

See also A., Nov., 1379, Detonation of solid explosives (GARNER).

XXIII.—SANITATION; WATER PURIFICATION.

Effectiveness of sodium peroxide respirators. G. STAMPE and E. HORN (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 419—422; cf. B., 1929, 798).—The evolution of oxygen from hydrated "pyroxylyt" (water 6%) is continuous; in 200 days 1 kg. evolved 3800 c.c. of oxygen, and there was no evidence of cessation at the end of that period. It is considered that during hydration superficial formation of sodium peroxide dihydrate occurs, and this readily decomposes, the liberated hydrogen serving to hydrate other molecules of the peroxide. The decomposition of sodium peroxide cartridges, when used in respirators, has been investigated, and is discussed.

W. J. WRIGHT.

Determination of small quantities of active chlorine in purified water. S. ANSELMi and A. CALò (Annali Chim. Appl., 1930, 20, 410—424).—Examination of the various methods proposed for the determination of small proportions of active chlorine in water purified by means of hypochlorites, chloramine, or gaseous chlorine shows that the most satisfactory is the o-tolidine colorimetric method (cf. Ellms and Hauser, B., 1914, 804), which is sensitive to 0.02 mg. Cl per litre and may be used for amounts up to 0.2 mg. per litre. The coloration is best compared with a scale obtained by mixing copper sulphate and potassium dichromate solutions in various proportions. For higher concentrations of chlorine, the potassium iodide-starch paste method is recommended, the iodine liberated being titrated with 0.01N-sodium thiosulphate; the proportion of chlorine found should be increased by 0.1 mg. per litre, which represents the limit of sensitivity of the reaction.

T. H. POPE.

ERRATUM. B., 1930, 796.—In the abstract of the paper on removal and determination of nitrates in sewage effluents and waters (by J. W. H. JOHNSON), in line 13 and subsequent lines the words "by suggesting . . . distillation" should be replaced by the following: "by suggesting that the samples were not boiled down as indicated, but distilled. The examination of these distillates would then disclose only half the original amount of nitrates, the other half having been found to disappear on distillation; this would fully account for the Commission's results of only 50% nitrate removal by boiling."

[Fatal] poisoning by amyl acetate. CRECELIUS (Klin. Woch., 1930, 9, 452—454; Chem. Zentr., 1930,

PATENTS.

Method of filtering (U.S.P. 1,762,560). Removing solids from strainers (B.P. 335,767). Treatment of boiler water (U.S.P. 1,762,748).—See I.